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Foreword

The papers in this Record, dealing with various facets of asphalts and asphalt additives, should be of interest to state and local construction, design, materials, and research engineers as well as contractors and material producers.

Rogge et al. present an overview of the Oregon Department of Transportation's use of emulsified asphalts for cold in-place recycling (CIR) of asphalt pavements. They report three significant findings from their study: careful project selection is important to the success of a CIR project, maintenance of proper proportions of emulsion and mix water is the biggest challenge in the field, and excess moisture from construction must be allowed to leave the mat before sealing. Vivier and Brule report on the use of gap-graded cold asphalt concrete in France made practical by polymer-modified asphalt emulsions and synthetic fibers. The binder used is specially formulated to be slow setting. The addition of fibers increases the viscosity of the emulsion and later the shear strength and wear resistance of the cold gapgraded asphalt concrete. Badaruddin and McDaniel report on an investigation to determine the feasibility of using CIR in Indiana. Performing their study on a low-volume flexible pavement, they report that after 5 years in service, a recycled portion of the pavement is performing better than a conventionally treated portion. O'Leary and Williams discuss the New Mexico State Highway and Transportation Department's in situ cold recycling program. They report successful pavement performance with the selection of high float styrenebutadiene-styrene polymer-modified emulsions to overcome rutting, reflective cracking, and moisture damage previously experienced with conventional rejuvenators. Pickering et al. present the laboratory evaluation of three different antistripping additives—lime added to the aggregates and two new-generation liquid additives mixed with the asphalt cements. The laboratory evaluation found the liquid additives not to be effective antistripping additives.

Bishara and McReynolds correlate performance-related characteristics of 20 asphalt cements with their physicochemical parameters using Corbett's fractions and high performance gel permeation chromatography. Lau et al. discuss the use of the pressure oxygen vessel for approximating road aging of asphalts. Shiau et al. investigated the aging characteristics of 20 asphalts commonly used by Florida and its neighboring states by utilizing the thin film oven test (TFOT) and rolling thin film oven test (RTFOT) at three temperatures (285°F, 325°F, and 365°F). They report that it appears feasible to use TFOT and RTFOT at the higher temperature to simulate the aging process on asphalt binders used in Florida. Button reports on a survey of modified asphalt test pavements using various asphalt additives to evaluate performance during construction and service. Kandhal and Khatri conducted a study to relate asphalt absorption to the physical properties of the mineral aggregate and asphalt cement binder. They found that generally the asphalt absorption decreased with an increase in the viscosity of the asphalt cement and that the most important pore size range in aggregates affecting the asphalt absorption appears to be 0.05 to $0.1~\mu m$.

Brown et al. discuss a study to evaluate the potential benefits of styrene-butadiene rubber (SBR) latex in hot mix asphalt mixtures. They report that no significant long-term benefits to date can be attributed to the use of SBR latex in dense graded asphalt mixtures. Collins and Bouldin discuss the aging stability of conventional and polymer-modified asphalts. They report that improved thermal and oxidative stability can be achieved by modifying asphalts with saturated polymers.

Use of Asphalt Emulsions for In-Place Recycling: Oregon Experience

D. F. Rogge, R. G. Hicks, T. V. Scholz, and Dale Allen

An overview of the use of emulsified asphalts for cold in-place recycling (CIR) of asphalt pavements by the Oregon Department of Transportation (ODOT) is presented. Project selection, design, construction, and inspection considerations are presented. ODOT has successfully used both CMS-2S (now called CMS-2RA) and high-float emulsions for recycling. Typical projects have been recycled to depths of 2 to 4 in. followed by either a chip seal or an open-graded emulsion mix. A brief overview of the CIR process is followed by a discussion of project selectionwhere to use and where not to use CIR. Procedures for estimating required water and emulsion additions to recycled asphalt pavement are presented. Procedures for field adjustments of emulsion and water content and for proper compaction of CIR pavements are presented. Finally, a discussion of problems experienced and appropriate preventive and remedial measures is presented. Significant findings include the following: (a) careful project selection is important to CIR project success; (b) the maintenance of proper proportions of emulsion and mix water is the biggest challenge in the field (the estimation procedure presented provides a good starting point and the discussion of construction control provides criteria for adjustments to meet changing field conditions); and (c) excess moisture from construction must be allowed to leave the mat before sealing, which should take place before winter conditions.

The national trend away from new construction to preservation of the highway system is requiring highway agencies to seek alternative approaches to rehabilitating distressed pavements. One promising and cost-effective approach is cold in-place recycling (CIR). Although CIR of asphalt pavements has been used in the United States in some form since the 1920s, several methods have evolved since 1980. During this period, spurred by the development of milling and reclaiming equipment, CIR has evolved into one of the fastest-growing pavement rehabilitation techniques.

BACKGROUND

Region 4 of the Oregon Department of Transportation (ODOT) constructed its first CIR project in 1984. Spurred by the initial success of the project, use of CIR in Region 4 has continued to grow, and other ODOT regions have investigated and constructed projects implementing CIR techniques. Figure 1 shows the growth of the use of CIR in ODOT Region 4. The sharp decrease from 1986 to 1987 was due to budgetary constraints.

Although exact figures are not available for 1989 to 1991, it is known that CIR construction has continued to grow beyond the 1988 levels shown.

The term cold recycling is frequently misunderstood because it has been used to describe different processes used with substantially different design concepts and results. These processes include in-place recycling of bound asphalt concrete (AC) material only, as well as full-depth reclamation where unbound granular base material is incorporated with the addition of emulsified asphalt. ODOT has used recycling of AC material only. Consequently, CIR is discussed in this paper as the cold in-place recycling of AC materials only. Full-depth reclamation is not addressed.

When CIR is performed on a uniform pavement designed and built to specifications, it is expected that the recycled pavement can handle low to medium traffic as a wearing course (with chip seal) and medium traffic volumes as a base course. When CIR is performed on a pavement with significant maintenance patches over a uniform pavement or a pavement with minimal design used in the original construction, it is likely that the recycled pavement can be used as a wearing course only for low-volume roads.

CIR treatments produce significant cost savings (see Table 1) compared with conventional hot mix overlays (I). In addition, there are savings in energy, a conservation of materials, a reduced impact on the environment, and production rates as high as 6-lane mi per shift. Another significant advantage of CIR is the ability to limit the correction to the distressed lane.

ODOT has used the resources of Oregon State University (OSU) for developing CIR procedures and gathering and analyzing performance data. These research efforts are documented in detail elsewhere (1-4) and summarized by Scholz et al. (5). This paper is drawn primarily from Rogge et al. (2).

OBJECTIVES

The overall objective of this paper is to provide the user/contractor with a better understanding of the project selection, design, construction, and quality control consideration required to ensure a successful cold in-place recycled project. Specifically, the objectives are to provide the following:

- 1. Guidelines for project selection, sampling, and mix design;
- 2. Information on equipment, procedures, and specification for CIR;

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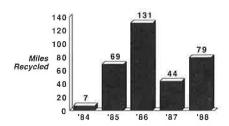


FIGURE 1 Growth in the use of CIR in ODOT Region 4.

- 3. Quality assurance guidelines for the engineer, contractor, and inspector; and
 - 4. Guidelines for postconstruction evaluation.

This paper is based on CIR design and construction as practiced by the Oregon Department of Transportation (ODOT) in 1990 using CMS-2S or HFE-150 asphalt emulsion and milling to depths of 2 to 4 in. with the majority of projects milling to 2 in. It is not intended for use on projects involving full-depth reclamation.

CIR THEORIES

Two theories have been proposed when designing cold recycled asphalt pavement (3). Briefly, the theories are as follows:

- 1. Treat the millings as a black aggregate with some hardened asphalt coating, and design an asphalt content to coat the milled particles. The assumption is that the millings will act as an aggregate.
- 2. Evaluate the physical (and possibly the chemical) characteristics of the asphalt in the old pavement and add a rejuvenating or softening agent that will restore the asphalt to its original condition. The assumption is that 100 percent softening occurs and a "new asphalt" is created.

In recent years, California, Oregon, Nevada, and New Mexico have concluded that a combination of the two theories most likely occurs. This "effective asphalt theory" is shown in Figure 2.

On the basis of this theory, a percentage of the old asphalt softens and combines with the added emulsion to produce an asphalt content in the mixture known as the effective asphalt. The percentage of asphalt that is softened is directly related to the softness of the old asphalt, the recycled asphalt pavement (RAP) gradation, and the percentage of asphalt in the old mix. Because these values can be readily measured, they have been incorporated into a procedure to estimate an initial emulsion content, which is described later in this paper.

TABLE 1 COST COMPARISON OF CIR VERSUS 2-in. HOT-MIX OVERLAY BASED ON ODOT REGION 4 DATA AND 1990 COSTS

	First Cost	Life Cycle Cost (Equivalent Annual Cost)
2 Inch Hot-Mix Overlay	\$5.25/sy	\$13,000-\$14,000/mi
2 Inch CIR with Chip Seal	\$2.10/sy	\$5,000-\$8,000/mi

Effective Asphalt = % Emulsion + % of Softened Asphalt Where:

- % emulsion is the amount of emulsion added; and
- % of softened asphalt is directly related to:
 - · Viscosity and/or penetration of old asphalt
 - Gradation of RAP
 - Percent asphalt in old pavement

FIGURE 2 Design theory for CIR pavements.

PRECONSTRUCTION STEPS

Project Selection and Testing Plan

Project selection is an important factor in determining the success of a CIR project. Not all projects are appropriate for CIR. Improper project selection can result in project failure. Figure 3 shows types of projects for which CIR is and is not recommended. Once it has been determined that CIR is feasible, preconstruction activities include field sampling, laboratory testing, and mix design.

Field Sampling

After a project has been identified as a recycle candidate, the first step in the preliminary engineering phase is to perform a paper search on the history of that highway. The information to be collected is the type of asphalt used in the pavement, thickness of the pavement, and termini of previous jobs (Figure 4). The project is then divided into preliminary mix design areas (e.g., shown in A, B, and C). Within each area, milling samples should be obtained using a small 16-in. mill. The sample frequency in each design area would be a minimum of three samples plus three backup samples on each section. The sample locations would be selected visually by identifying representative locations within the design area. Milling depth should correspond to the proposed recycle depth. Samples should be collected with the mill in forward motion. If visible maintenance patches or other intermittent treatments occur within the section, a sample would be taken from that section noting on the sample that it came from a patched area. Samples are kept separate and submitted to the laboratory for testing.

Laboratory Tests on RAP

Consistent with the previously described effective asphalt theory, the following tests should be performed on the RAP obtained from the field sampling:

- 1. Gradation of the RAP millings (16-in. mill),
- 2. Extracted asphalt content,
- 3. Penetration of extracted asphalt at 77°F, and
- 4. Absolute viscosity of extracted asphalt at 140°F.

These values are then used to estimate the optimum emulsion content, described in the next section.

a) CIR Not Recommended
Pavements with obvious subgrade problems
Work area cannot accommodate traffic volume
Asphalt is stripping from aggregate*
Mixes exhibiting rutting due to unstable, fat mixtures
Cold and damp conditions, including heavily shaded areas
Late fall or early winter treatment
Asphalt pavement is less than 1,5 inch thick
Intent is to widen roadway by reducing thickness and incorporating unbound shoulder rock
b) CIR Recommended
Cracked and broken pavements
Pavements raveled due to age
Rough pavements
As leveling and base for overlays
ADT 5000 or less unless multilane
Where selective rehabilitation is needed (e.g., in truck lane of 4-lane roadway)
Native aggregate poor or in short supply

^{*}Emulsions contain effective antistrip agents. While it is not recommended at this time that CIR be used to correct pavements with stripping problems, CIR may prove to be an effective treatment. Use of time in CIR is also being evaluated.

FIGURE 3 Considerations for project selection.

Mix Design Considerations

Several procedures are available to determine the optimum emulsion and water content for CIR mixtures. The one described is based on the work performed in Oregon.

Estimation of Emulsion Content

The procedure to select the amount of emulsion to be added to a recycled mixture is essentially an estimation process. It begins with a base emulsion content to which adjustments are made on the basis of the results of laboratory tests conducted

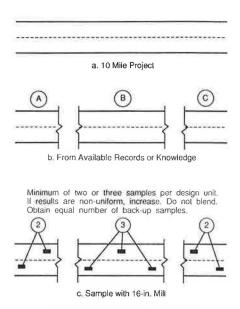


FIGURE 4 Suggested field sampling.

on a sample taken, using a 16-in. mill, from the pavement to be recycled. It has been found through experience with the CMS-2S and HFE-150 emulsions that a base emulsion content of 1.2 percent is a good starting point (5). Adjustments are then made to the base content according to the softness of the extracted asphalt, gradation of millings as produced by the 16-in. mill, and the percentage of asphalt recovered from the sample.

The penetration (ASTM D5) or the absolute viscosity (ASTM D2171) laboratory test results are used to determine the softness of the extracted asphalt, and the RAP gradation is determined for only three screens—½ in., ¼ in., and #10. The percentage of recovered asphalt is determined by the Abson method (ASTM D1856). From the laboratory test results, the added emulsion content (based on dry weight of millings) can be determined through the use of Figure 5 and the following equation:

$$EC_{EST} = 1.2 + A_{G} + A_{A/C} + A_{P/V}$$

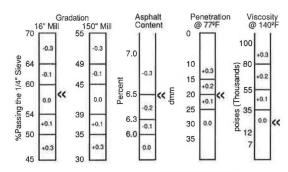


FIGURE 5 Emulsion content adjustments for gradation, asphalt content, and asphalt softness.

where

 EC_{EST} = estimated emulsion content (percent),

1.2 = base emulsion content (percent),

 $A_{\rm G}$ = adjustment for gradation (percent),

 $A_{A/C}$ = adjustment for residual asphalt content (per-

cent), and

 $A_{P/V}$ = adjustment for penetration or viscosity (percent).

For borderline cases (those that fall on a boundary) in Figure 5, the adjustment resulting in a lower estimated emulsion content should be used. Also, where there is a discrepancy between the adjustments for penetration and absolute viscosity, the adjustment resulting in a lower estimated emulsion content should be used. For example, given 58 percent passing the ½-in. screen on the 16-in. mill, 6.5 percent residual asphalt, a penetration of 20 dmm, and a viscosity of 19,000 poises, the adjustments are as follows (for borderline cases, use adjustment producing lower emulsion content):

- 1. 0.0 percent for gradation,
- 2. -0.3 percent for asphalt content, and
- 3. 0.0 percent for penetration/viscosity.

The estimated emulsion content is 1.2 percent + 0.0 percent - 0.3 percent + 0.0 percent = 0.9 percent.

The advantage of this approach is that it provides a rapid and simple method to determine the estimated emulsion content. The laboratory tests used are widely accepted. The results appear to produce the optimum emulsion content within a fraction of 1 percent. For most recycle projects where preservation or restoration of an existing pavement is the primary objective, the estimated emulsion content is adequate for the final recommended design.

Estimation of Amount of Water Required

To ensure adequate dispersion of the emulsion and complete coating of the RAP, addition of water to the mix is usually required. A saturated surface damp condition is desired. To determine the total liquids content, the modified Oregon State Highway Division test method OSHD TM-126 is used (3, Vol. 2). The test determines how much total liquids a particular recycle mix can tolerate. Thus, once the estimated emulsion content is determined, the modified OSHD TM-126 test is conducted on the mix to determine the total liquids content.

Briefly, the total liquids test is conducted as follows (see OSHD-TM-126 for complete details):

- 1. Samples are prepared at the final estimated design emulsion content and at incremental water contents (e.g., 0.5, 1.0, and 1.5 percent). Each sample weight is recorded.
- 2. Each sample is placed and rodded in a split mold in two lifts.
- 3. Each sample is gradually compressed to a total load of 25 kips: 1 min to achieve 20 kips plus ½ min to achieve the additional 5 kips. The 25-kip load is held for 1 min.
- 4. The specimen weights are then determined. The difference between the initial sample weight and the weight of the compacted specimen is the liquid loss.

The total liquids content that results in a liquid loss of 0.035 to 0.14 oz (1 to 4 g) is used as the desired total liquids content. From this, the water content can be calculated (total liquids content minus estimated emulsion content).

Seal Versus Overlay

The decision to seal or overlay must be predicated on the structural design assumptions of the roadway. Minimal surface sealing such as a fog seal would be used on a pavement that is expected to perform satisfactorily for a short time or as part of a stage-type reconstruction. The type and amount of traffic to use the recycled surface also influences whether a seal is necessary and the type of seal. Fog seals, chip seals, double chip seals, hot mix overlays, and open-graded emulsion mix overlays have been used successfully. All agencies now recommend that all cold recycled mixes have, as a minimum, a chip seal placed on their surface.

CONSTRUCTION OPERATIONS

Proper project selection, preconstruction investigation, and estimation of emulsion and water contents are fundamental to the success of a CIR project. The project can still fail, however, if proper construction procedures are not followed.

CIR consists of milling the existing asphalt pavement to a specified depth, mixing the RAP with water and asphalt emulsion, depositing the mixture in a windrow, and paving and compacting with conventional paving equipment. Figure 6

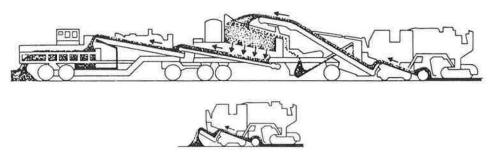


FIGURE 6 Equipment normally used in CIR treatments: top, full train with mill, screen deck, crusher, belt scale, and pug (laydown with a standard paving machine); bottom, single unit train with asphalt emulsion sprayed into mill chamber.

shows the two types of CIR equipment that have been used successfully in Oregon. After excess water has been allowed to evaporate from the CIR mat (usually 3 to 14 days), the mat is recompacted and either sealed with a chip seal or overlaid before the arrival of autumn weather conditions. A more detailed discussion of construction operations appears later under the heading "Controlling Construction Operation To Achieve Optimum Results."

QUALITY ASSURANCE GUIDELINES

Even if one develops the best mix design and construction specifications, the project may fail if the engineer and contractor are not fully informed of the limitations of CIR mixtures. This section discusses some ways of minimizing problems with cold recycling that should be considered on all projects.

Engineer Orientation

When using cold recycle for the first time, it is important to alert the project engineer about what is expected from the new procedures. In cold recycling there are objectives or expectations different from those associated with hot mix. Some of the areas that should be considered in use of cold recycling are as follows:

- 1. Is the CIR to be a stabilized base with a surface course?
- 2. Will the CIR be used instead of a leveling course?
- 3. Will the CIR be used, with a chip seal, as a wearing course?

The project engineer should be made aware of the importance of the procedures listed in this paper and what the results mean to the completion of a successful project. These procedures should also be stressed to the contractors at the prebid conference and to the contractor at the preconstruction conference.

A postconstruction debriefing with the project engineer provides a valuable source of information for modification of future contracts. Also, using a project engineer experienced in CIR to train other engineers helps to minimize problems on future CIR projects.

Contractor Qualifications

Although most agencies do not have specific provisions for contractor prequalification, the following contractor qualifications are desirable:

- 1. Knowledge of paving materials;
- Experience in all types of bituminous paving, but particularly cold mixes;
- 3. Ability to coordinate, manage, and supervise a multifaceted, high-production project; and
 - 4. Prior experience with cold recycling.

The contract specifications may preclude some of the contractors from bidding because of the lack of adequate equip-

ment. Contractors that are not properly equipped will not be able to provide an acceptable product.

Field Testing and Quality Control

CIR may still be considered more of an art than a science. Experience is still the best teacher in determining what to do. In addition to required testing and measurements, continual visual examination during and after construction is required for successful CIR projects.

Special provision should be made to ensure adequate proportioning, mixing, curing, and compaction of the recycled asphalt pavement. Several factors will influence the behavior and performance, including

- Size gradation,
- Pavement and air temperature as well as solar heating,
- · Amount of emulsion and water added,
- Temperature of emulsion and mixing water,
- · Curing time, and
- Compaction.

Control of these factors through regular testing and evaluation reduces the potential for problems. Observations have shown the following factors to be important:

- 1. Using too much asphalt emulsion or recycling agent will result in an unstable mix that is subject to rutting and shoving. Too little asphalt emulsion may cause the mixture to ravel. Minor raveling is acceptable.
- 2. Excessive mix water may cause the asphalt to flush to the surface and will retard curing. Too little mix water results in mix segregation, raveling under traffic, or high void contents.
- 3. Coarse gradation of the processed RAP may cause problems with laydown, segregation, dragging, and excess voids. Fine gradation tends to reduce the mix's tolerance for water and emulsion deviations.
- 4. Water required for mixing is generally in excess of that required for compaction. Curing or removal of excess water, or both, will generally be required before adequate compaction can be achieved.

Depending on the weather, curing may take place in a matter of a few hours, or it may be a few days or longer. To minimize problems associated with curing of the emulsion, ODOT has introduced the practice of heating the mix water and the emulsion to the 120°F to 140°F range. Although this process does not significantly increase the mixture temperature because of the relatively small proportion of liquids in the mix, it is the opinion of field personnel experienced in CIR construction that the practice reduces curing problems in cool or damp conditions. One experienced inspector believes that heating the water reduces the need to increase the emulsion content when conditions are cool. The practice of heating the water may cause problems of breaking and curing too fast, however, where windrow temperatures would be in excess of 120°F.

Controlling Construction Operations To Achieve Optimum Results

Table 2 summarized ODOT's recommendations for field quality control. Most of the items presented in Table 2 are straightforward and not subject to error. The challenges of field control are in two major areas: verification and adjustment of emulsion and water content, and proper compaction and traffic control procedures.

"If Things Aren't Going Well, Key on the Water or Emulsion"

These are the words of an ODOT project manager with extensive experience in CIR. They stress the criticality and uncertainty of maintaining proper mix proportions in the variable conditions of the field.

The central laboratory should have provided estimated emulsion requirements for areas where milling samples were taken. Observation of the entire project referenced to milling locations should indicate whether patching has been done subsequent to the millings, or whether the pavement is more variable than the design would indicate. Either of these occurrences can serve as a warning to expect changes in emulsion content or other problems. If the milling locations are representative of the pavements to be recycled, design emulsion contents should be reliable.

If observation of the project indicates poor drainage or heavily shaded areas, or both, expect RAP to have higher moisture content than the milling samples tested in the lab, and therefore anticipate that the amount of water to be added will be less than that estimated in design. Heavily shaded areas will also serve as a warning that emulsion curing will be slow and that 24-hr traffic control should be anticipated.

The normal procedure is to start construction with the water and emulsion contents designed for the pavement. Experienced CIR inspectors and construction supervisors then verify or adjust the quantities of liquids added through several procedures. They conclude that the mix is too wet or too dry by holding and squeezing the windrowed material in their hands, standing on the windrow, and noting the way they sink, observing the nurse tank track imprint in the edge of the windrow, or observing the way the windrow "slumps" as it is deposited. What they are looking for is thorough coating of the RAP material and workability. After initial rolling, the color of the mat should be brown rather than shiny black. On the basis of these observations, liquid addition may be increased, decreased, or remain the same. Obviously, these procedures are subjective, highly dependent on the experience of the inspectors and construction supervisors. Objective, quantifiable procedures are needed.

The use of the Kelly ball (ASTM C360) to replace these subjective tests has been investigated, but with inconclusive results. It is known that constructible pavements resulted when Kelly ball displacements 30 sec after placement on a leveled section of windrow ranged between 1.75 and 2.5 in. (mean of 2 in. for readings taken) and when the ball sank an additional 0.1875 in. during the next 30 sec. It is not known whether mixtures with improper emulsion and water contents would have similar displacements and rate of displacement.

Checking gradation is an objective method of adjusting emulsion content. On the basis of the percentage of RAP passing the ¼-in. sieve, variations from the design basis may be noted and adjustments made in accordance with Figure 6. Significant variations from the gradation upon which the design is based will be visually apparent to the experienced observer.

The total liquids test, OSHD-TM 126 (2), provides a rational way of checking emulsion and water contents, but since the test takes 45 min, the information is not available for immediate adjustment. Liquids loss from this test should target a range of 0.035 to 0.14 oz (1 to 4 g) with losses of up to 0.35 oz (10 g) being acceptable. If loss is greater, cut back on water added. If loss is less, add additional water. The liquid lost should only be slightly discolored. Dark liquid lost in-

TABLE 2 RECOMMENDED FIELD QUALITY CONTROL PROGRAMS

Tests	Frequency	Purpose
RAP gradation	1/2 mile	Identify changes in pavement material
Emulsion content and water content	Continuous meter reading	Verify design content
Emulsion content and water content	Daily tank sticking	Verify meter reading
Total liquids	1/2 mile	To adjust water
Liquid loss color	1/2 mile	To adjust emulsion content
Emulsion and mix water temperature (when heating is required)	1/2 mile	Assure proper temperature
Mix temperature	1/2 mile	Verify minimum laydown temperature
Emulsion quality	Every 50 tons	Check product consistency
Depth and width	Random	Establish pay items
Smoothness	Random	
	Optional Tests	70-
Extracted gradation	Random	Information only (after recycle)
Extracted asphalt content	Random	Information only (after recycle)
Viscosity/penetration	Random	Information only (after recycle)

dicates too much emulsion; clear liquid lost indicates possibly insufficient emulsion. When in doubt, err on the side of too little emulsion.

The paving ahead of the mill should be examined for the presence of "fat" spots or unstable mixes. The emulsion content should be dropped 0.2 percent in areas that appear slightly fat and 0.4 percent in areas that are obviously unstable and rutted. These adjustments are made only if field samples are not taken at the exact locations of the distress.

Adjustments to emulsion content should also be made on the basis of the appearance of the mat after initial compaction, assuming that the pavement is reasonably uniform. Additional emulsion should be added (up to +0.2 percent) if the mat remains brown or is prone to raveling. At the other extreme, the emulsion content should be reduced 0.2 percent if the mat is very black and shiny and no raveling is apparent, if pushing or rutting occurs under traffic, or if bleeding or flushing occurs. The recycled pavement should have a brown appearance for at least $24 \, \mathrm{hr}$.

In general, when there is too much water in the mixture, the material looks wet and cannot be compacted. It "shoves" and has the appearance of an overrolled hot mix. If there is too little water, the mixture looks dry, the RAP material will not show uniform coating, and the compacted mat will ravel.

When making adjustments to what has been a satisfactory operation, the amount of total liquids is generally kept at the same level. In other words, if emulsion content is reduced 0.2 percent, water addition is increased by the same amount; if emulsion content is increased, water addition is decreased accordingly.

Balling of fines may occur. The problem usually results from inadequate mixing, excessive emulsion, or excessive fines.

A problem that may occur that is related more to the paver than to the liquids content is segregation of the mat. Segregation at the center of the panel is usual, resulting in a very open appearance. The paver may require modification to minimize this segregation.

Compaction, Traffic Control, and Rerolling

Specifications will provide guidelines for compaction. However, it is important to remember that the purpose of initial rolling is simply to get the material set. It is not desired to seal the surface initially. Moisture must get out so that the material may cure. It is important not to overroll, which may be manifested by the flushing of emulsion or fines, or both, to the surface. The paving machine should stay close to the recycling train or single unit train, but the breakdown roller should stay well back, particularly when working in cold temperatures or compacting fine materials.

Generally, recycling operations should not be undertaken or continue unless weather conditions and forecasts indicate that at least 3 hr of 90° windrow temperatures are likely. If this guideline is followed, traffic may be allowed (with speed limitations) on the mat at the conclusion of rolling operations. If shaded areas or weather conditions inhibit curing, 24-hr traffic control may be required. If it is anticipated that areas will not set up and will ravel, a very light application of tack coat may be made as a preventive measure. This application consists of either the emulsion being used for recycling or an

SS1 diluted with an equal quantity of water and applied at a rate of 0.08 gal/sy, followed by applications of choke sand. As long as this application allows moisture to escape from the mat, it is preferred to increasing the emulsion added to the RAP, since excess emulsion may lead to instability at a later date.

About 1 week after traffic has been allowed on the mat, the mat is once again rolled to remove any wheeltrack rutting and to seal the surface. Rerolling should not be allowed until excess moisture has evaporated from the mat. One way to determine that adequate drying has taken place is to leave total liquids briquettes along the roadway to experience curing conditions identical to those experienced by the mat. When the briquettes no longer lose weight, moisture content has reached equilibrium.

Sealing

A well-constructed CIR mat will rut if sealed too soon or crack if not sealed before winter conditions. A minimum moisture content before sealing should be specified, and some type of seal should always be installed before the onset of freezethaw conditions.

Lime

CIR projects may require the addition of lime to the RAP. Preliminary investigation indicates that the addition of lime greatly accelerates the curing process (6). To date, the most efficient method for introduction of lime is to spread granulated lime uniformly ahead of the milling machine, introduce water to slake the lime, roll the granules to allow dispersion of the lime into the mix, and use the milling machine to disperse the lime into the mix. From a process control point of view, the following are critical:

- 1. The proper amount of lime to achieve the desired concentration in the mixture and the proper breakdown of the lime granules in order that the lime be completely dispersed into the mix in a fashion that will allow coating of RAP/aggregate particles should be noted.
- 2. Mix and milling head water addition should be reduced to compensate for the addition of water to slake the lime.

Postconstruction Evaluation

Work performed during the period 1984 to 1991 by ODOT indicates that the most common problems encountered include rutting, raveling, cracking, and local failures. Causes of and solutions to these problems are summarized in Table 3.

Rutting is considered to be the most serious problem. However, it can be corrected by re-recycling as follows:

- 1. In cases with excess emulsion, re-recycle the distressed area and increase the depth of cut to pick up additional material
- 2. In cases of flushing, re-recycle (without going any deeper and without adding recycling agent or emulsion) and delay the initial compaction period.

Туре	Causes	Solutions	Comments
Rutting	Too much emulsion Sealing surface during laydown Early application of chip seal	Proper mix design Limited vibratory compaction and prohibit pneumatic rollers until mix is stable Allow cure period to reduce moisture to 1.0-1.5% (usually 2 weeks of good weather)	Occurs within the first few days after construction or during hot weather the season following recycling
Raveling	Cool weather/shaded areas Slow setting asphalt emulsion Inadequate traffic control Insufficient asphalt emulsion	Preheat mixing water Fog seal areas experiencing raveling Use pilot cars	Occurs within first few hours after opening surface to high speed traffic
Cracking	Open-graded nature of mix Freeze-thaw action if not sealed Insufficient structural section Improper asphalt emulsion Rolling too soon	low to medium trafficked roads thaw action if not sealed being structural section ber asphalt emulsion low to medium trafficked roads Use an open-graded emulsion mix or hot mix on heavily travelled roads Allow drying prior to compaction	
Local Failures	Inadequate base Wet subgrades	Identify these areas prior to recycling Dig out, rebase, and patch prior to recycling	These frequently occur on low volume highways with little or no base rock. Since CIR has little strength during the first 24 hrs, truck traffic will break up new CIR surfaces.

TABLE 3 PAVEMENT PROBLEMS IN CIR MIXES—CAUSES AND SOLUTIONS

CONCLUSIONS

Conclusions to be drawn from this paper include the following:

- 1. Project selection is an important factor for successful CIR. Trying to recycle pavements with soft base or wet subgrade, inadequate width, inadequate depth of AC pavement, or extremely variable RAP properties, for example, is futile. See Figure 3 for a list of projects to avoid.
- 2. Maintenance of proper proportions of emulsion and mix water during recycling is the biggest challenge in the field. Design of proper emulsion and water contents and procedures for verifying and adjusting them in the field are critical.
- 3. Initial compaction (rolling) of the recycled mat is intended to set rather than seal the surface. Trapping excess moisture in the mat is detrimental to performance.
- 4. Placing a chip seal on the CIR mat before winter conditions is essential to prevent moisture and freeze-thaw damage to a pavement with high voids content (10 to 15 percent). However, the pavement must not be sealed too soon, trapping excess moisture in the mat. Specifications should preclude sealing the CIR mat if moisture exceeds 1.5 percent.

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REFERENCES

- D. F. Rogge, T. V. Scholz, and R. G. Hicks. In-Depth Study of Cold In-Place Recycled Pavement Performance, Vol. 1—Final Report. FHWA-OR-RD-91-02A, Dec. 1990.
- D. F. Rogge, R. G. Hicks, and T. V. Scholz. In-Depth Study of Cold In-Place Recycled Pavement Performance, Vol. 2—Construction and Inspection Manual. FHWA-OR-RD-91-02B, Dec. 1990.
- 3. R. G. Hicks et al. Development of Improved Mix Design and Construction Procedures for Cold In-Place Recycled Pavements. OR/RD-87-06, Vols. 1 and 2, Oregon Department of Transportation, Feb. 1987.
- T. V. Scholz, R. G. Hicks, and D. Allen. Development of Improved Mix Design and Construction Procedures for Cold In-Place Recycled Pavements. OR/RD-89-01, Vols. 3 and 4, Oregon Department of Transportation, June 1988.
- T. V. Scholz, R. G. Hicks, D. F. Rogge, and D. Allen. Use of Cold In-Place Recycling on Low-Volume Roads. In *Transportation Research Record* 1291, Vol. 2, TRB, National Research Council, Washington, D. C., 1991, pp. 239–252.
- H. Zhou and S. E. Nodes. Study of Lime vs. No Lime in Cold In-Place Recycled Asphalt Concrete Pavements. OR-RD-92-02, Oregon Department of Transportation, Sept. 1991.

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Gap-Graded Cold Asphalt Concrete: Benefits of Polymer-Modified Asphalt Cement and Fibers

Maurice Vivier and Bernard Brule

Currently in France, the cold asphalt concrete market is growing, with microsurfacing increasingly replacing and giving better results than chip seal surface dressings. As part of the promotion of cold asphalt concrete in France, one contractor recently developed the first gap-graded cold mix. The use of polymer-modified asphalt emulsions and synthetic fibers made this practical. The binder is a cationic, ethylene vinyl acetate modified asphalt emulsion specially formulated to have controlled slow-setting characteristics. Both long- and short-term benefits are derived from the addition of fibers. The fibers initially increase the viscosity of the emulsion, so that gap-graded formulations can be placed without segregation. After curing, the fiber network reinforces the cold gap-graded asphalt concrete, increasing the shear strength and wear resistance. To date more than 500,000 m² of gap-graded fibrated asphalt concrete have been successfully laid.

Slurry seal technology was imported from the United States to France more than 25 years ago. Initial success led to the laying of 5 million m² of slurry seal in 1965. Unfortunately, the technique was abandoned a few years later, when skid resistance problems occurred.

In the early 1980s, with the advent of improved mixing and laying equipment, there was a renewed interest in cold asphalt concrete. Conventional slurry seals were normally designed with natural sand (about 50 percent) and small aggregates (less than 3 mm). The new specially adapted spreading machines allowed the replacement of natural sand with crushed sand from hard rock, and the use of 0/4, 0/6, or 0/10 graded aggregates.

Cold asphalt concrete may be defined as an asphalt emulsion (with or without polymer modification) mixed with continuous or gap-graded 0/4, 0/6, or 1/10 aggregate containing 100 percent crushed sand. Its market is currently growing as it replaces chip seal surface dressings. This improved technique has resulted in fewer broken windshields, a longer construction season, smoother riding surfaces, noise reduction, and very few failures.

To promote cold asphalt concrete in France and improve road safety through coarser and more durable pavement surfaces, a contractor recently developed the first gap-graded cold asphalt concrete. An integral part of this system is the use of polymer-modified asphalt emulsion in conjunction with synthetic fibers.

GAP-GRADED FIBRATED COLD ASPHALT CONCRETE

In this process gap-graded aggregate containing synthetic fibers is mixed with a polymer-modified asphalt emulsion.

Aggregates

The aggregates consist of crushed hard rock meeting French specifications for asphalt concrete wearing courses. They are gap-graded, usually from 0/6 to 0/10 mm. Two typical grading curves are shown in Figure 1 (0/10 and 0/6 mm).

Fibers

Extra-fine synthetic organic fibers are used. These relatively long (typically 4 mm by 1.6 decitex) polyacrylonitrile fibers are surface treated to facilitate dispersion in aqueous media.

The fiber content of the asphalt concrete is very low (0.1 to 0.2 percent based on dry aggregate weight). However, because the fibers are extremely fine, there are a large number of fibers per unit area, yielding a very dense fiber network. For example, a 0/10 fibrated gap-graded mix with 0.15 percent fiber (3 decitex, 6 mm in length) spread at a rate of about 20 kg/m² will be reinforced by 15 million fibers/m². The fibers form a discontinuous chain, close to 100 km in length.

Binder

The binder is a polymer-modified asphalt cationic emulsion specially formulated to have controlled slow setting characteristics. Cationic emulsions perform well in French climatic conditions and allow the use of many aggregates, including unaltered extrusive igneous materials (granite, diorite, andesite, and basalt) and metamorphic rocks (gneiss, quartzite, and amphibolite).

The modification by ethylene vinyl acetate (EVA) copolymer simultaneously improves four essential properties of the binder: cohesion, temperature susceptibility, rheological behavior, and adhesion. The Vialit ram pendulum test demonstrates cohesion improvement by measuring the energy absorbed by the fracture of a binder film under a given impact. The binder to be tested is used to adhere a grooved steel cube to a grooved steel stand. A ram pendulum is released to knock

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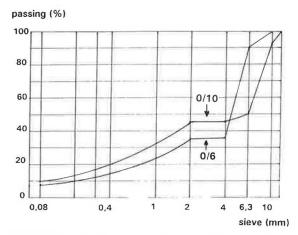


FIGURE 1 Grading curves of gap-graded aggregates used for fibrated cold asphalt concrete.

the cube off the stand, causing a cohesive failure of the binder. The cohesion is quantified by the difference between the energy needed to dislodge the cube and the energy needed to dislodge a blank—the cube without the binder. The test is run at a series of temperatures (-30° C to $+60^{\circ}$ C), and a cohesion versus temperature curve is generated. Figure 2 shows the curves of neat and EVA-modified asphalt cement. It shows that addition of 3 percent polymer increases the maximum cohesion at a given temperature as well as broadens the range of temperatures of high cohesive strength.

MIXING AND LAYING PROCEDURES

The mix is designed using a cohesiometer (modified manual cohesion tester—ISSA Technical Bulletin 139 and ASTM D 3910-80A) and a wet track abrasion tester (WTAT; ISSA Technical Bulletin 100 and ASTM 3910-80A). The cohesiometer determines the rate of set as well as the rate of development of wet cohesive strength of cold mixes, and the wet track abrasion tester measures resistance to wear.

The equipment used for mixing and laying the fibrated gapgraded cold asphalt concrete is identical to that commonly used for microsurfacing. The fibers are added volumetrically in proportion to the aggregates. Because the fibers are very fine, extremely small quantities (not exceeding a few grams per square meter) are involved. To ensure reliable and accurate addition, a special device to distribute the fibers into the aggregate was developed. An important advantage of this patented device is the dry addition of the fiber, which is more convenient than wet processes. The dry addition allows better control of the mixture moisture content, a critical laying parameter.

Typical application rates of the wet material are 12 to 15 kg/m² for 0/6 mm gap-graded (2/4) material and 20 to 23 kg/m² for 0/10 mm gap-graded (4/6) material. For example, a 0/6 mm gap-graded (35 percent of 0/2 and 65 percent of 4/6) cold asphalt concrete was laid at a rate of 13 kg/m² on a high-traffic highway in western France 2 years ago with excellent results. It should be noted that 0/10 cold asphalt concrete is used in rare cases where very high skid resistance is needed.

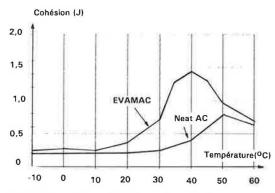


FIGURE 2 Effect of EVA on the cohesiveness curve of an asphalt cement.

Generally, a 0/6 cold asphalt cement surface is preferred even for roads carrying heavy traffic.

BENEFITS OF FIBER ADDITION

Benefits During and Immediately After Construction

The addition of fibers greatly increases the apparent viscosity of the asphalt emulsion and significantly modifies its rheological behavior, resulting in a thixotropic mixture. A Contraves TV viscometer with MS2 spindle was used to study the effect of fibers on the emulsion viscosity. This viscometer is capable of measuring 40 to 700 mPa.s. Figure 3 shows the increase in viscosity of the asphalt emulsion. Here the addition of 0.2 percent fiber increases the apparent viscosity of the emulsion by a factor of ten.

The higher emulsion viscosity and the modification of its rheological behavior (especially the very high viscosity at low shear) cause a concomitant change in the rheology of the mix, providing it with exceptional homogeneity. The French standard method slump test (NF 18 452) measures the time that a given amount of material needs to fill a given volume. Figure 4 shows the effect of fibers on the flow time versus moisture content. For a given water content, the flow time of the fibrated mix is about 3 sec longer than that of the mix without fibers.

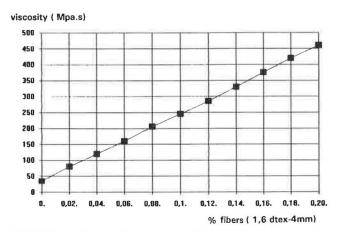


FIGURE 3 Effect of fibers on emulsion viscosity.

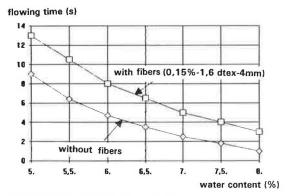


FIGURE 4 Modification of mix rheology by fibers (NF 18 452 slump test).

Because the consistency of the mix is substantially improved to a thixotropic pastelike material, gap-graded formulations can be used without risk of segregation. For the same reason, a gap-graded fibrated mix can be applied in very wide passes with results uniform across the whole pavement. In addition, there is no loss of aggregate due to traffic after reopening of the road. The fibers increase the shear strength of the mix even before the setting process is complete.

Long-Term Benefits

After curing, the cold gap-graded asphalt concrete is reinforced by the fiber network, which substantially increases the shear strength. Figure 5 is a diagram of the device used to test the shear strength. At 30°C, a beam of cured cold asphalt concrete (20 by 5 by 1 cm), fixed for 10 cm at one end, is uniformly loaded at 2.4 g/cm². The deformation at the end of the beam is measured as a function of time. Figure 6 shows that the shear resistance of the fiber-reinforced cold mix is roughly two times that of the control cold mix without fibers.

Similarly, the wear resistance of the fibrated mix in the presence of water is noticeably improved over that of the nonfibrated mix. In Figure 6 the wear resistances of two 0/10 mm cold mixes, one of which contains 0.15 percent fibers (3.3 decitex; 8 mm in length), were measured by the wet track abrasion test. This test measures the mass loss of immersed samples subjected to friction by a rubber pad. To accelerate the wear, the samples were conditioned before the test for 16 hr at a temperature of 18°C and 60 percent humidity (conditions different from those of American specifications).

Figure 7 clearly shows that the resistance to wear of the fibrated cold mix is much better than that of the unmodified mixture.

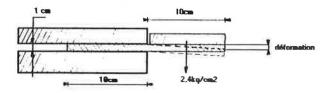


FIGURE 5 Device used to measure the shear strength of cold asphalt mixes.

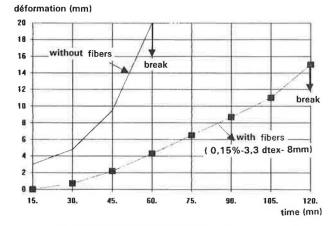


FIGURE 6 Creep of a beam of cold asphalt concrete with and without fibers.

The gap-graded aggregate ensures a granular surface texture, reducing glare and providing better drainage when it rains and, most important, imparting good skid resistance even for high-speed traffic. The skid resistance was measured with the R4E skidding resistance trailer built by the network of French Public Works Laboratories (LPC). This device measures the longitudinal coefficient of friction (LCF) of pavements and runways. A passenger car-type wheel with constant vertical load is towed at constant speed by a vehicle that also sprays the pavement with water. The wheel is locked for lengths of about 20 m, and the mean torque of the resistant rotational forces is measured. The test may be run at speeds from 40 to 140 km/hr, and measurements can be made in traffic. Figure 8 shows that gap grading ensures good skid resistance. LCF values greater than 0.45 at 80 km/hr measured on 0/10 gap-graded cold mixes bear evidence to safe surface courses even for high-speed driving.

CONCLUSIONS

The fibrated gap-graded cold asphalt concrete with polymermodified asphalt emulsion is suitable for any type of traffic

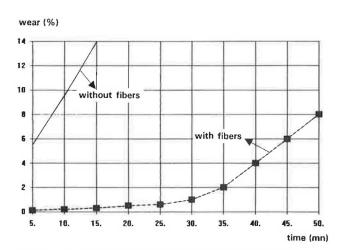


FIGURE 7 Wet track abrasion test results for two cold mixes.

skid resistance

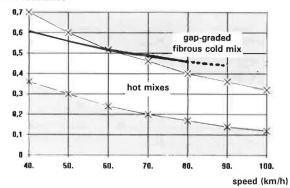


FIGURE 8 Skid resistance of gap-graded cold asphalt mix compared with conventional hot mix (white area corresponds to the LCF envelope of French roads).

(including high-volume roads) for maintenance of all types of pavements, as well as for new construction.

A maximum aggregate size of 6, 10, or, in rare cases, 14 mm, is selected depending on the degree of surface texture desired, the traffic type or level, and the admissible noise level under the given traffic conditions. A 0/10 grading will thus be selected for very heavy or rapid traffic in the open country, whereas the less noisy 0/6 material will be used in urban or heavily populated areas.

To date more than 500,000 m² of gap-graded fibrated asphalt concrete have been laid in various locations, some of

TABLE 1 LPC R4E SKIDDING RESISTANCE TRAILER LCF RESULTS FOR A COLD ASPHALT CONCRETE AFTER 5 YEARS

Speed (kph)	LCF	
40	0.47	
60	0.44	
80	0,36	

which are heavily trafficked sections of major highways with constantly accelerating accident rates (called black points in France). The first jobs, dating back to 1986, are still performing well, particularly in reducing the number of accidents and maintaining consistent surface texture.

For example, a control section has been monitored on a very high-traffic road (more than 1,000 heavy trucks per day). After 5 years the skid resistance measured with the LPC R4E skidding resistance trailer remains very good, as indicated in Table 1.

The cost depends greatly on the area and type of laying machine used. In France, a 0/6 mm cold asphalt mix surface (10 000 m²/day, 15 kg/m²) costs roughly 15 FF/m² (less than \$3.00/m²). The additional cost of the fibers is estimated to be about 4 percent.

The use of gap-graded cold mix should therefore continue to develop, further contributing to the revival of cold asphalt concrete in France.

Publication of this paper sponsored by Committee on Characteristics of Bituminous Materials.

Cold In-Place Recycling for Rehabilitation and Widening of Low-Volume Flexible Pavements in Indiana

SHAKOR R. BADARUDDIN AND REBECCA S. McDaniel

A rural road in Indiana was selected for rehabilitation and widening in 1986. The project was carried out as a study to investigate the feasibility of using cold in-place recycling in the state of Indiana. The highway selected was a 20-ft-wide low-volume road. One-half of the 9.85-mi project was cold in-place recycled and widened to 24 ft, and the other half was widened in the conventional way by adding 2-ft-wide strips on each side of the existing roadway. The entire pavement was then overlaid with hot mix bituminous mixture. An evaluation after 5 years in service indicates that the recycled half of the highway is performing better than the conventionally treated half. Field investigations indicate less distress and an absence of widening cracks on the recycled pavement. Laboratory tests on the field cores also indicate a generally better pavement condition in the recycled half. The other half of the pavement is showing serious distress in the form of widening cracks reflected upward and some alligator cracking. This half of the pavement will need rehabilitation much earlier than the recycled part.

A cold in-place recycling project was carried out in 1986 on SR-38 in Indiana as part of a widening and resurfacing procedure. The project involved recycling the base layer while widening it from the existing width of 20 ft to 24 ft and topping the surface with hot mix asphalt. An adjacent section of the same highway was widened and resurfaced the conventional way. Two-ft-wide strips of bituminous mix were added on each side of the pavement before resurfacing the entire surface with hot mix asphalt. This paper reports on the performance of the two pavement sections after 5 years in service.

The recycled pavement has performed well and is in better condition than the highway section that received the conventional treatment. The project indicates that cold in-place recycling is a feasible alternative for rehabilitation of low- to medium-volume roads.

PROJECT DESCRIPTION

A 9.74-mi section of SR-38 in Indiana was selected for rehabilitation using cold in-place recycling in 1986. The Research Division was called in to study the pavement after the contract had been let. The section was due for rehabilitation work, which was to include widening and resurfacing the entire length from the existing 20 ft to 24 ft. SR-38 was a two-lane, low-volume rural highway with about 1,500 vehicles per

day in 1981, which has since increased to about 1,900 vehicles per day. The location of the site is shown in Figure 1 (I).

The section was divided into halves of 4.87 mi each; the western half received the standard widening treatment, which included adding 2-ft-wide strips of bituminous mixture on each side of the existing 20-ft-wide roadway. The strips were laid in a prepared trench and then compacted flush with the existing pavement edge. The eastern half of the pavement section was milled to a depth of about 6 in. and recycled in place on the road surface as a 24-ft-wide base layer. The entire pavement was then resurfaced with hot mix bituminous mixtures. The cross sections of both treatments are shown in Figure 2. An initial report about the project was first published in 1988 (1), from which further details can be obtained.

The comparisons made and conclusions drawn here refer to the composite pavement sections rather than individual layers because of the differences in cross sections. It was believed that this comparison would be useful in helping the department choose between the two rehabilitation strategies. The total depths of the two pavement sections are approximately the same, although they are composed of different materials. The deflection data, discussed later, indicated that the pavements were roughly equivalent in structural strength at the time of construction.

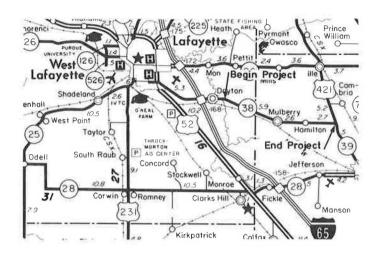
The purpose of this study was to evaluate the efficiency of cold in-place recycling and the structural strength and performance of the resulting pavement structure compared with the conventional widening and resurfacing treatment used in Indiana.

EVALUATION TECHNIQUE

The evaluation was carried out in two stages. The first was the nondestructive testing in the field. The field tests formed the basis for selecting the good and bad sites and characterizing the performance of the pavement. The second stage was the destructive testing of the field cores, which was carried out entirely in the laboratory to determine the characteristics of the materials that made up the pavements.

A technique had to be selected to obtain the optimum information to perform an effective evaluation. The choice was between randomly testing the entire pavement in the study and taking samples from representative locations of the pavement. It was decided to select a good and bad pavement section from each half of the project and conduct the eval-

Indiana Department of Transportation, 1205 Montgomery Street, P. O. Box 2279, West Lafayette, Ind. 47906.



Total Project Length 9.76 Miles

RESURFACE Western 4.88 Miles

RECYCLE Eastern 4.88 Miles

Area Enlarged



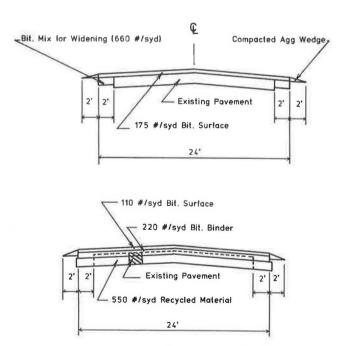


FIGURE 2 Typical cross sections: top, resurfaced pavement; bottom, recycled pavement.

uation on them only. In this way it was thought that the entire pavement would be represented within the best- and worst-performing pavement sections that were selected, and thus a more meaningful comparison could be made between the recycled and nonrecycled halves of the project. The question that remained was what criteria to use to select these sections.

Since the whole pavement was Dynaflect tested every year as part of the monitoring activity by INDOT, these data were readily available to be used as a criteria for identifying the best- and worst-performing sections. Dynaflect tests are non-destructive and reveal the structural integrity of the pavement system and its support layers. The deflection basin W1 (2) was used as a basis in this selection because that reflects the pavement support conditions relevant to this study.

A summary of the 10 best and worst Dynaflect W1 values for tests carried out in 1990 is presented in Table 1. The table indicates that there are good as well as bad representative sections in both halves of the pavement in the study. Thus the likelihood of having biased or skewed the data by selecting exceptionally good or bad sections in that particular half is minimized. The entire Dynaflect data set was tested for normality using the chi squared test, and the coefficients of kurtosis and skewness obtained indicated a normal distribution.

TABLE 1 SUMMARY OF 10 BEST AND WORST DYNAFLECT SECTIONS

	R	ECYC	CLED SECTIO	NS	F	ESUR	FACED SEC	TION
	E-BOUND STATIONS	W1	W-BOUND STATIONS	W1	E-BOUND STATIONS	W1	W-BOUN STATIONS	
	177	154	182	139	95	153	62	186
	186	139	157	132	100	145	63	167
	173	136	167	131	62	134	92	149
	182	134	164	128	61	134	100	145
	164	132	181	122	92	129	94	145
	168	128	154	120	93	128	61	143
	184	124	160	118	69	123	95	139
	143	124	178	118	111	121	99	134
	136	123	138	115	63	120	26	128
	135	112	173	115	89	118	93	126
	140	58	168	64	l 98	66	65	64
	224	56	202	64	19	66	24	63
BEST	206	56	204	64	101	64	20	57
	227	55	132	63	45	64	7	56
	130	53	201	61	36	57	34	54
	155	51	219	58	7	57	4	53
	131	51	130	56	34	56	1	52
	204	48	245	55	3	56	3	51
	226	48	223	48	6	52	5	49
	129	46	131	48	4	51	6	46

SAMPLING

Another factor included in the study was wheel location. Samples were taken from the wheelpath and from outside the wheelpath. This factor was included to investigate the effect of wheel load on the strength and material characteristics of the pavement.

Thus there were three factors in the study: pavement type (recycled or resurfaced), condition (good or bad), and location (wheelpath or outside). Each was present at two levels, resulting in a 2³ factorial design of experiment. A layout of the design of experiment is given in Table 2. At each location that was selected, at least six 4-in.-diameter cores were obtained; three cores were taken from the wheelpath and three more from outside the wheelpath. The cores were sliced into their respective pavement layers before testing. A complete analysis was carried out on all the cores obtained.

FIELD EVALUATION

A plot of the Dynaflect values at the selected test locations is shown in Figure 3. The best and worst Dynaflect data (W1) for the recycled half are much lower than those from the nonrecycled half. Lower deflection values indicate better pavement strength. When tested shortly after construction,

TABLE 2 LAYOUT OF SAMPLING DESIGN

TYPE	RECY	CLED	CONVENTIONAL			
CON- DITION	W/PATH	O/WPATH	W/PATH	O/WPATH		
GOOD	А	С	Е	G		
BAD	В	D	F	Н		

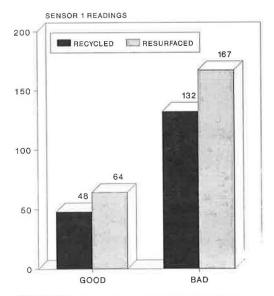


FIGURE 3 Dynaflect values of test sections.

the deflection values on the recycled section were only slightly lower than the values on the resurfaced section (I), indicating that the two pavement structures are roughly structurally equivalent.

A pavement condition survey was also carried out at each of these locations using the method of the U.S. Army Corps of Engineers (3). This survey takes into account every visible distress and quantifies them according to a standard that yields an index called the Pavement Condition Index, or PCI. The PCI is measured on a scale of 1 to 100, where the higher number represents a better pavement. The results of this survey are given in Table 3, and a plot of the PCI values is shown in Figure 4. Figure 4 shows that the recycled pavement on the whole is performing better. The PCI value on the bad section of the recycled pavement is slightly higher than the good section, which can be attributed to variations in measuring distress, since only the selected sites were surveyed. Also, the PCI is a surface performance rating, which does not necessarily correspond to the Dynaflect measurements of structural strength. The major distresses in the recycled sections were low levels of transverse and longitudinal cracks and rutting, whereas in the nonrecycled section the predominant distresses were medium to low levels of widening cracks along the joint, transverse and longitudinal cracks, and rutting. Some low levels of alligator cracking were also observed in the latter. Visually, the nonrecycled section is showing more prevalent and more severe distress.

LABORATORY EVALUATION

In the laboratory, several tests were conducted to obtain characteristics of the pavement and its materials and to evaluate them with regard to performance. The tests included bulk specific gravity of cores (ASTM D2726-89), Marshall stability (ASTM D1559-89), maximum Rice specific gravity (ASTM D2041-90), quantitative extraction of bitumen from bituminous paving mixtures (ASTM D2172-88), recovery of asphalt from solution by Abson method (ASTM D1856-79), pene-

TABLE 3 TEST RESULTS

Core	Dynfl	Ave.	Pen.	KinV	Rut	% Air	Asph.	PCI	RICE	Ave.
#		Marsh			Depth	Void	%		Sg	Bsg
A31	48	1035	18.0	814	0.25	7.9	5.91	70	2.501	2.3031
B31	132	1029	20.0	772	0.25	6.3	6.10	75	2.469	2.3141
C31	48		19.5	831	0.25	7.6	6.65	70	2.484	2.2937
D31	132	748	23.0	685	0.25	7.7	6.07	75	2.481	2.2897
E31	64	631	21.0	669	0.25	5.5	5.57	59	2.514	2.3753
F31	167	703	25.5	576	0.25	4.4	5.42	57	2.506	2.3960
G31	64	548	21.0	676	0.25	7.6	5.16	59	2.517	2.3252
H31	167	711	25.0	581	0.25	5.7	5.43	57	2.513	2.3701
A32	48	1151	18.0	874	0.25	3.4	4.59	70	2.477	2.3928
B32	132	1043	27.0	594	0.25	3.1	5.09	75	2.503	2.4241
C32	48	1131	20.5	832	0.25	1.6	5.32	70	2.445	2.4059
D32	132	1396	21.0	727	0.25	2.8	4.51	75	2.490	2.4206
E32	64	720	14.5	1121	0.25	8.1	4.77	59	2.450	2.2509
F32	167	493	20.0	739	0.25	7.8	5.19	57	2.457	2.2639
G32	64	603	15.0	915	0.25	7.9	4.92	59	2.463	2.2687
H32	167	459	22.0	808	0.25	7.4	5.74	57	2.445	2.2631
A33	48	575	27.0	701	0.25	7.9	7.28	70	2.427	2.2347
B33	132	525	33.5	591	0.25	7.1	6.88	75	2.451	2.2757
C33	48	519	29.5	688	0.25	6.2	6.34	70	2.416	2.2656
D33	132	477	27.0	709	0.25	8.3	6.09	75	2.470	2.2641

tration of bituminous materials (ASTM D5-86), and kinematic viscosity of asphalts (ASTM D2170-85). The test results are summarized in Table 3. The base layer of the recycled pavement was analyzed and not the base of the nonrecycled pavement, because that was the old pavement. Only the average results are given for each test, which was conducted in duplicate or triplicate according to the ASTM test methods mentioned. Appropriate plots have been made to elucidate the superior performance of the recycled pavement given in the next section.

ANALYSIS OF RESULTS

The test results obtained in this study are in agreement with previously published reports (4,5) that cold in-place recycled pavements perform well and are viable for medium- to low-

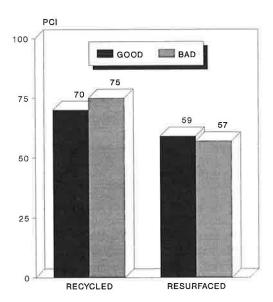


FIGURE 4 PCI values of test sections.

traffic-volume highways. In addition, they are stiffer and hence perform better than conventional pavements (5,6). Figures 3 and 4 show plots of Dynaflect and PCI data, respectively, confirming these findings. The higher PCI values also point to a more structurally sound pavement system.

A plot of penetration values of recovered asphalt from the surface, binder, and base layers is shown in Figures 5, 6, and 7, respectively. A comparison is also made for samples from the wheelpath and from outside the wheelpath. The general trend is for the bad sections to have higher penetration values in the surface and binder layers. Soft asphalt in these areas may contribute to greater rutting there. There is no obvious trend in the base layer. The asphalt from outside the wheel-

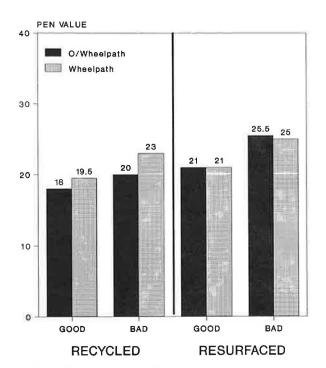


FIGURE 5 Penetration of surface.

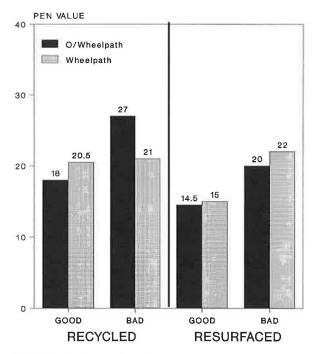


FIGURE 6 Penetration of binder.

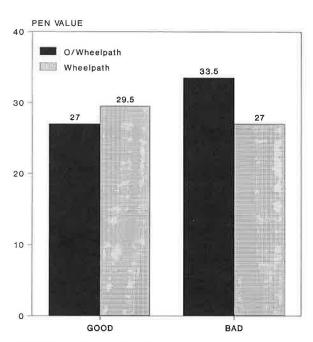


FIGURE 7 Penetration of base.

path generally has lower penetration values, perhaps because it is less traveled and hence has more air voids that allowed more oxidation to occur. Figure 8 shows the predictable result of penetration versus kinematic viscosity, and the relationship for each layer is similar, forming almost parallel curves.

A plot of Marshall stability versus percentage of air voids in Figure 9 shows another expected result. Lower air void content corresponds to higher Marshall stability, because the pavement is more compact and dense. The higher Marshall

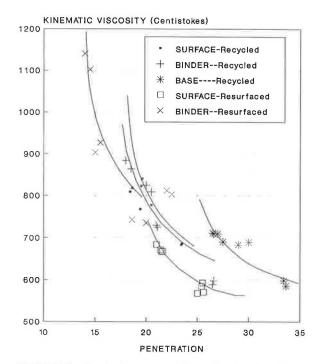


FIGURE 8 Penetration versus kinematic viscosity of recovered asphalt.

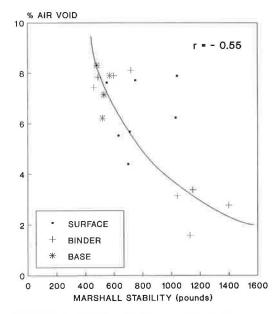


FIGURE 9 Relationship between Marshall stability and air void content.

values also correspond to the recycled half of the pavement, where distress is lower. A plot of the Marshall values is shown in Figures 10 through 12 for the different locations. Figure 13 shows the relationship of penetration versus Marshall stability, where higher penetration, hence softer asphalt, produces lower Marshall stabilities. All these plots verify earlier expectations for recycled pavements and conform with field evaluation.

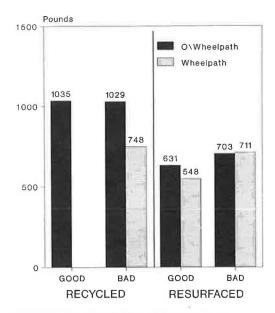


FIGURE 10 Marshall stability of surface.

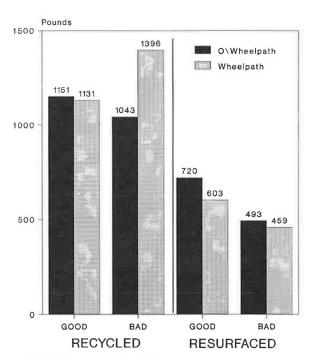


FIGURE 11 Marshall stability of binder.

The tests indicate that after 5 years in service since cold inplace recycling, the pavement is in very good condition and is performing better than the pavement that received the standard conventional resurface treatment. The laboratory and field observations indicate that the pavement will last at least another 5 years before major rehabilitation work is required. The western half of the project, which received the standard treatment, is showing greater distress and will need treatment much sooner.

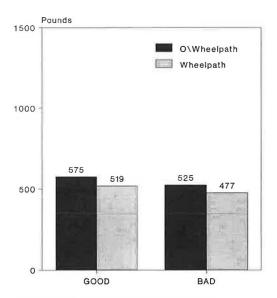


FIGURE 12 Marshall stability of base.

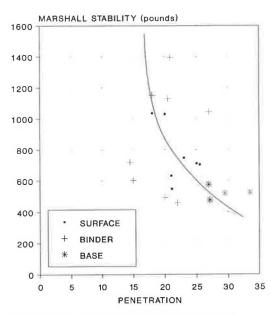


FIGURE 13 Relationship between Marshall stability and penetration values.

CONCLUSION

The cold in-place recycling project constructed on a trial basis in 1986 on SR-38 in Indiana can be called a success, because after 5 years in service it has been shown to perform better than a stretch of initially identical pavement that was rehabilitated conventionally. The recycled pavement shows less distress and has a better PCI and greater support values from the Dynaflect tests than the other. It has needed very little maintenance work since construction ended and still performs well. Test results confirm findings by other researchers about similar projects and indicate that this technique is indeed an alternative for rehabilitating medium- to low-volume asphalt pavements.

REFERENCES

 R. S. McDaniel. Cold In-Place Recycling of Indiana State Road 38. In *Transportation Research Record 1196*, TRB, National Research Council, Washington, D. C., 1988, pp. 234–242.
 K. Majidzadeh and V. Kumar. *Manual of Operation and Use of*

Dynaflect for Pavement Evaluation. Report FHWA/OH-83/004. Federal Highway Administration, U. S. Department of Trans-

portation, 1983.

M. Y. Shahin and S. D. Kohn. Pavement Maintenance Management for Roads and Parking Lots. Report CERL-TR-M-294. Construction Engineering Research Laboratory, U. S. Army Corps of Engineers, 1981.

 J. A. Epps, NCHRP Synthesis of Highway Practice 160: Cold-Recycled Bituminous Concrete Using Bituminous Materials. TRB, National Research Council, Washington, D. C., 1990.

 J. A. Epps, D. N. Little, and R. L. Terrel. NCHRP Report 224: Guidelines for Recycling Pavement Materials. TRB, National Re-

search Council, Washington, D. C., 1980.

 D. N. Little and J. A. Epps. Evaluation of Certain Structural Characteristics of Recycled Pavement Materials. *Proc.*, Association of Asphalt Paving Technologists, Vol. 49, 1980.

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In Situ Cold Recycling of Bituminous Pavements with Polymer-Modified High Float Emulsions

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The New Mexico State Highway and Transportation Department began an in situ cold recycling (ISCR) program in 1984. On more than 1,850 lane-mi of Interstate, primary, and secondary highways, this effort has been successful in reusing valuable resources (asphalt and aggregate), reducing the transportation and handling of materials, minimizing environmental pollution, and rehabilitating pavements with many types of distresses at minimal cost. The development of the ISCR design, specification and construction methods, and the selection of high float styrene-butadiene-styrene polymer-modified emulsions to overcome many of the problems (rutting, reflective cracking, and moisture damage) previously encountered with conventional rejuvenators are discussed. Annual statewide tours of ISCR projects have documented overall excellent rideability, minimal rutting, and a delay of reflective cracking by 5 to 6 years over that in control sections.

In 1984 New Mexico had many miles of roadway to be rehabilitated or reconstructed with limited funding. Faced with rising asphalt prices, falling revenues, and diminished quality aggregate sources in some areas, the New Mexico State Highway and Transportation Department (NMSHD) began an in situ cold recycling (ISCR) program. Because existing cold recycling practices and conventional emulsions did not give the desired results, NMSHD sought to improve the technique. New methods were adopted for design, construction, and quality control, and new rejuvenator emulsions (high float polymer modified) were used.

PROJECT DESIGN

To realize any benefit from ISCR, an appropriate mix design procedure and pertinent field quality control tests must be developed. Two widely different approaches have been used in cold mix recycling.

The first anticipates that the recycled asphalt pavement (RAP) will vary markedly with depth, history (age, environment, patches, seals, etc.), and the construction equipment used during the recycling process. It concludes, therefore, that a formal laboratory mix design approach is futile. The amount of recycling additive is estimated from previous experience or from a rudimentary Marshall procedure, but without serious consideration for the amount or hardness of the asphalt already in the RAP. Construction is begun, and field adjustments are made on the basis of experience, appearance, and

workability. If the mix is too dry, it is often used as untreated base. If too much additive is applied, the mixture quickly loses air voids and becomes susceptible to deformation under traffic.

The second approach calls for a highly sophisticated design process coupled with rigorous quality control during every phase of ISCR. Milling machines are brought to the project long before the actual job to obtain numerous representative field samples for the design. The asphalt is extracted from each of the RAP samples to determine hardness, and then the rejuvenating additives are carefully chosen to provide a finished binder of some predetermined asphalt grade. Gradations of the extracted aggregate samples are carefully determined, and traditional volumetric designs are run to determine amounts of virgin aggregates and additive that will be used. This approach usually specifies rigorous quality control testing at the job site to guarantee that the laboratory design is achieved.

The no-design approach guarantees that the benefits of ISCR will not be optimized, and serious failures will almost certainly occur. Unfortunately, the highly sophisticated design method is extremely expensive and taxes already strained laboratory resources. The high variability of materials even within one project makes it extremely difficult to generate one perfect design. Costs for obtaining initial samples using milling equipment are also high, and these small milled areas may cause traffic problems or suffer structural failure before the project is begun. Thus the high cost and effort of the latter approach defeats the primary purpose of using ISCR as an inexpensive alternative for pavement rehabilitation.

NMSHD has adopted a site selection, mix design, quality control strategy that falls between these two extremes. Using the general guidelines discussed later, 105 ISCR projects incorporating 1,853.9 pavement lane-mi have been successfully completed since 1984. There have been no serious failures to date. These projects, some of which are listed in Table 1, consist of 1,331.7 lane-mi of primary and secondary roads and 522.2 lane-mi of Interstate pavement.

PROJECT SELECTION

Initial selection of a project suitable for ISCR is made by reviewing the original construction design documents and then discussing the maintenance history with appropriate district personnel. Information gathered includes traffic, original pavement thickness (including base materials), overlays, seal

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TABLE 1 SUMMARY OF IN SITU RECYCLING PROJECTS

			Recyc	le	Emulson	Average Compress					Traffic Time of		Existina	Pavement	New We	earing	Course
Control		Lane		Emulsion	Added	Section 10 section		Cost (\$/s	q yd)				(inches)		11011 111		Thic
No	Highway	Miles	(in)	Grade	(%)	(psi)	Density		Emulson	Total	ADT		Surface	Base	Туре	OGF	C (in)
307	110	30,12	3	HFE 150	1.70	238	120.4	1.4	0.73	2.13	7655	30.6	0	W6,E6/6	PMBP	Υ	3.5
312	US 60	18-4	3	HFE 150	4.20	199	142	1.25	1.19	2.44	962	13.5	1.75	7	PMBP	Υ	1.5
721	NM 28	14.58	3	HFE 150S	1.50	380.2	131.9	1.25	0.29	1.54	2301	2.7	4.75	4	PMBP	Y	3
730	NM 90	3.1	2.5	HFE 150S	1.08	438	123.5	1.95	0.34	2.29	1232	12	2	7	PMBP	Y	1.5
856		52.96	4	HFE 150S	2.36	376	133	1.05	1.24	2.29	18809	4	4	14.5	PMBP	Υ	2
910		9.12	3	HFE 150				1.16		1.16	2511	23	3	6	PMBP	Υ	3.5
130	US 82	8.6	3,5		1.80	N/A	129.85	1		1					PMBP	Υ	2
301		10.8	3	ASSESSMENT OF THE PROPERTY.	3.00	379	124	0.95	0.94	1.89	1305	24.2	2.5	UTB3,Sub4	PMBP	Υ	2.5
2301		8.1		HFE 300	2.44	N/A	142.1		181/TON								
857		7.22	4.0		1.49	294	131.6	1.5	0.54	2.04	1074	14.3	6	6.5	PMBP	-	3
1939	US 70	16.04			1.73	445.13	140,21	1.25	0.75	2	N/A	N/A	4	6		Y	4
1722		15.38	3		1.69	487	132	1.25	0.49	1.74	1243	8	4.8	4.5	PMBP	Y	2
1045	US 54	11.62			2.16	378	137	1.3	1	2.3	4379	24.8	4	UTB3.5,Sub4		Y	2
564	US 70	12.78	4	HFE 150	2.78	336	135.5	1.45	1.13	2.58	3285	14.7	4.5	6	PMBP	Υ	3
1541		16	7	HFE 150	1.94	227	136	1.45	0.61	2.06	1478	14.1	4.5	6	PMBP	Y	2.5
313 545	US 70 US 70	19.54	3	HFE 150S	0.59 2.80	535 273	130.5	1.1	0.11	1.21	2114	22.5 30	2.0&2.5	11.0&12.0	PMBP PMBP	Y	2.5
	US 70	19.6	4				134.8	1.13	1.27	2.4	3789		5.5	15	PMBP		
1568 1202	US 82				2.25	394	134.7	1.2	0.99	2.19	1849	23	4.5	6		Y	2.5
539	US 82	16.06 16.14		HFE 150 HFE 150	3.70 4.44	246 277	122.6	1.25	0.1B 1.13	1.43	895 740	12.9 27.1	2	6	PMBP PMBP	Y	3
201	US 60	30.88		HFE 150	4.44	236	123	1.3		1.9	8028		3.5	9	PMBP	N	2
	US 82	27.88				236	119 122	1.05	0.85			13.1			PMBP	Y	3
718		11	3	HFE 150 HFE 150S	3.10 1.87	418	128.99	2	0.29	1.64	2675 871	22 9	3	5,5 5	PMBP	Y	2.5
542	US 70	19.8	4		1.42	328.2	131	1	0.55	1.55	2070	22	4	6	PMBP	Y	3.5
543	US 70	20	4	HFE 150S	1.42	344	131		0.55	1.55	1919	22	4	6	PMBP	Y	3.5
1538	US 60	12.8	3.5	HFE 1503	3.70	330	131	1.35	1.21	2.56	286	12.9	3.5	6	PMBP	Ý	1.5
525		24.64	3.5		1.11	417	138	1.55	0.09	1.59	3093	10.6	3.6	4	PMBP	Y	3
1716		14.56		HFE 1503	1.20	324	137.27	1.25	0.05	2.2	1194	15.4	4.5	6	PMBP	Ý	2.5
1378		29.2	4.5	HFE 150	2.40	193	131	1.65	0.42	2.07	1954	13.5	4.5	6	PMBP	Ý	3
379		29.56		HFE 150	2.20	255	130	1.65	0.46	2.11	1582	13.5	4.5	6	PMBP	Ÿ	3
381		29.28	4	HFE 150	2.89	334	131.1	1.25	0.76	2.01	1502	10.5	5.5	8	PMBP	Ý	2.5
380	US 285	30	4	HFE 150	2.90	243	127	1.25	0.93	2.18	1401	13.2	4.5	8	PMBP		3
382	US 285	17.16	4.5	HFE 150	2.35	367	133.5	1.25	1.15	2.4	1138	12.2	4.5	6	РМВР		2
720	NM 6	20.9	3	HFE 150S	3.30	213.17	140.2	1	1.1	2.1	717	3	2	6	PMBP	Υ	2.5
725	NM 4	6.1	3	HFE150S/3		194	134	1.2	0.89	2.09	755	10.2	2.0-4.5	3	PMBP	Y	2.5
1294	1 25	27.72		HFE 150S		78	124.5	1.2	0.76	1.96	2855	19.7	9	8	PMBP	Y	3
257	1 25	20.36	3	HFE 150S	1.61	110.31	127.07	1.2		1.2	2532	19.7	8.0-8.5	8	PMBP	Y	3
1548	1 25	21.84	4	HFE 300S	1.21	229	145.3	1		1	2296	22.6	9.23	6	PMBP	Y	3.5
437	1 25	27.6	4.5	HFE 300S	1.52	115	133.67	0.56	0.82	1.38	3146	22.6	4.5	4	PMBP		4.5
864	1 25	18	4	HFE 300S	0.65	122.5	138.15	0.8	0.32	1.12	2695	19.3	8.5	6	PMBP	Y	4
825	1 25	28.36	4	HFE 300S	1.50	152.5	129.64	0.82	0.82	1.64	2643	22.6	5	ATB4,Sub7	PMBP	Υ	4
315	140	30.16	4	HFE 150S	1.01	244	131	1.4	0.49	1.89	7937	50.7	6	4	PMBP	Y	3
	US 64/8	18	3.5	HFE 300S	1.69	274.38	147.44	0.9	0.85	1.75	1986	12.2			PMBP	Y	4.5
284	US 54	39.72	4	HFE 150S	1.32	279	138.55	0.85	0.59	1.44	2202	24	4.35	5.04	PMBP		3
155	US 87	34.4	4	HFE 150	1.80	N/A	N/A	1.5	0.63	2.13	2010	10.5	3	6	PMBP	Y	4
204	US 87	19.22	3	HFE 150	2.11	167	125	1.2	0.48	1.68	2151	11.2	3	6	PMBP	Y	3
52	US 64	20.38	3.5	HFE 150S	1.07	294	138	1	0.3	1.3	1635	12.2	4	8	PMBP	Y	4.5
247	US 54	26.36	3	HFE 150	1.88	326	137	1.25	0.27	1.52	2312		2	3	PMBP	Y	3
572	US 54	21.4	3	HFE 150	2.63	409.5	139.9	1.15		1.15	901	17.2	3	5	PMBP	Y	3
317	NM 504	15.28	4	HFE 150S	1.79	709	144.2	2.5	0.33	2.83	1125	7	2.5	9	DBL P	N	DB
97	US 84		3.5	HFE 150S				1.5	275/TOP	V	2028	6.4	3.5	5	PMBP	Y	5
372	US 285	8.8	3.5	HFE 150S				1.65	100/TOP	V	845	20	3.5	8	PMBP	Y	3
70	140	11.44	4	HFE 150S				0.92		0.92	11624	25.1	6.5	CTB4,Sub8	PMBP		3
318	US 60		4	HFE 150S				1	245/TOP	N	610	9.4	4	8	PMBP	Y	2
268	US 60	25.18	3	HFE 150	2.50	N/A	N/A	1.4	0.63	2.03	461	10.3	2.25	6	PMBP	Y	1.5
614	NM 44	16	3.5	HFE 150S	2.68	259	134	1.4	1.06	2.46	4691	15.5	3	4	PMBP		3
547	US 180	11.52	4	HFE 150	4.00	297	128	1.92	1.36	3.28	528	7	2	7	PMBP	Y	1.5
652	NM 4	6.74	4	HFE 150	2.78	N/A	129	2	1.03	3.03	1250	10.2	1.5	8	PMBP	Y	2.5

coats, amount and type of patching, and so forth. There is also a thorough field examination of base and subgrade conditions. Pavements with extensive base or subgrade problems are not good candidates for ISCR and are immediately rejected.

Once the initial review indicates that a site is suitable for ISCR, 8 in. cores are taken every ½ mi in both wheelpaths and lane centers of the existing pavement for laboratory analysis and mix design. These samples are selected to be representative of the major portion of the project, not of isolated or failed areas.

LABORATORY ANALYSIS AND MIX DESIGN

There is no standard mix design procedure of ISCR. An NCHRP study of two proposed methods found that "a precise laboratory design (even if obtainable) is not critical for achieving a successful recycling project. At best, it can only serve as a general guideline for an initial job-mix formula, with adjustments being made following an evaluation of mix quality, including such factors as workability, coating, plasticity, and ease of compaction" (1).

However, certain basic principles applicable to all asphalt paving projects should be followed as part of the designselection-specification process:

- 1. Examination of field samples for evidence of stripping;
- 2. Crushing of RAP to required size;
- 3. Selection of grade of recycling emulsion and prediction of approximate amount of additive required;
- 4. Mixing, compaction, and testing of trial mixtures using the appropriate additive to optimize formulation for binder content, density/air voids, and stability. Determination whether additional virgin aggregate, either from base course or blended during milling, is required to increase pavement thickness, increase air void content, or improve mixture stability;
- 5. Estimation of strength parameters for characterizing load-carrying capabilities of the ISCR mix. A study at Purdue University found that the structural coefficient for a layer of recycled emulsion mix should range from 0.17 to 0.44, with a median value of 0.29 (2). New Mexico uses 0.25. When the minimum unconfined compressive strength of the ISCR mix exceeds 250 psi, a structural coefficient of 0.30 appears to be a valid estimate. Other studies of performance conducted by Oregon State University and Oregon Department of Transportation indicate that the ISCR structural layer coefficient may be considered equivalent to that of a conventional hotmix pavement (3); and
- 6. Field adjustments as necessary to achieve proper coating of emulsion on the aggregate, ensure sufficient workability and proper density, and so forth.

New Mexico no longer tests mixtures during the preliminary design phase, but rather delays all laboratory work until after the ISCR project has been let to contract.

NMSHD has determined that a 1.0 percent polymer-modified high float emulsion should be included as a bid item to establish a separate price for the liquid additive, but the exact quantity is not determined until the design and initial construction phases are completed. This system allows tailoring the job to the contractor and its equipment without unduly affecting material costs after the contract price has been established.

RECYCLING AGENT—HIGH FLOAT POLYMER-MODIFIED EMULSIONS

On the basis of extensive prior cold mix experience, NMSHD began ISCR operations in 1984 using conventional high float anionic mixing grade emulsions. This philosophy was somewhat different from the more specialized aromatic oil rejuvenating emulsions used widely at that time for cold recycling applications. High float asphalt emulsions were selected because they create a gel structure in the asphalt residue after the water evaporates. These materials are believed to improve temperature susceptibility, reduce draindown or flow of the asphalt cement, resist deformation due to traffic, and improve resistance to "washing off" when wetted (4). NMSHD specified two grades of high float emulsions (HFE-150 and HFE-300), which conform to AASHTO specifications but were slightly modified to provide greater flexibility in residue penetration and mixing ability to meet specific field conditions.

After using these conventional high float emulsions for approximately 2 years, a project was built using a styrenebutadiene-styrene (SBS) (STYRELF) polymer-modified high float emulsion. It was found that the polymer further enhanced the positive characteristics of the high float emulsion residue. The SBS polymer network develops a strong, elastic matrix within the asphalt residue (5). Only a very small amount of virgin binder can be added without overasphalting a 100 percent RAP mixture. Since this residue must be relatively soft to adequately coat the RAP (including fines generated by the mill head), the quality of this virgin binder is of utmost importance. The higher cohesive strength imparted by the polymer rapidly develops load bearing capacity, which prevents the recycled pavement from rutting soon after being opened to traffic. The polymer improves the long-term resistance of the finished product to moisture damage, which is particularly important for preventing raveling, cracking, and potholes (6). Although NMSHD normally covers ISCR projects with conventional hot mix or a double penetration surface treatment (double chip seal) to seal the pavement as soon as moisture conditions allow, this has not always been possible. In some cases the ISCR pavement has been left open to traffic during the winter, and no apparent deterioration was ob-

Polymers also allow the use of low viscosity (soft) asphalts, which are better able to soften the aged asphalt cement in the RAP over a period of time, without suffering the early distresses of permanent deformation commonly encountered with unmodified soft materials. The softer asphalts can be more easily dispersed in finely graded mixes, and they are particularly valuable when mixes contain highly absorptive aggregates. It is important to remember that cold recycling relies heavily on the consistency of a very small quantity of virgin binder. At ambient temperatures there is not the rapid blending of the recycling agent with the aged asphalt as when using hot recycling methods.

Table 2 gives the properties of the polymerized high float emulsions.

SPECIFICATIONS

The success of any in situ recycling program begins with the specifications, ideally simple and straightforward and providing flexibility to optimize materials and construction variables throughout each individual project. Given the wide variability encountered, specifications must be somewhat generic. One choice is method specification, which lays out precisely how the work will be performed, the type of equipment used, and the end result in general terms. An alternative is end-result specification, which defines the required mixture criteria and in-place density but leaves the means for achieving these results to the contractors and suppliers.

Since the RAP is typically nonuniform and critical field conditions (temperature, humidity, wind, moisture content in pavement, etc.) vary on an hourly basis, it is not possible to predefine all operational parameters. Hence, New Mexico has chosen a combination of methods and end-result specifications that covers the following topics:

- 1. Overall description of the job;
- 2. Job mix formula with an initial estimate of the emulsion grade and amount;
- 3. Pulverization requirements for the RAP, including strict limitations on the maximum allowable size in the recycle mix;
- 4. Laboratory mix design requirements, including methods to determine optimum emulsion content and grade;
- 5. Field change order flexibility, which enables the user agency to vary the amount and grade of emulsion (virgin binder) as required to meet existing conditions. This includes the right to request special emulsion additives such as coating enhancers, dispersants, and antistripping agents where laboratory or field results indicate they will enhance performance:
 - 6. Mixture laydown and aeration standards;
- 7. Compaction requirements, including size, type, and sequence of rollers, in-place moisture content, and minimum density of the finished mix;
- 8. Quality control criteria, including inspection, sampling, and testing;
 - 9. Weather limitations;
 - 10. Traffic control and safety restrictions; and
- 11. Protection of the newly laid ISCR surface from traffic if required to prevent abrasion or raveling. This may include fog sealing with a dilute emulsion or other techniques acceptable to the user agency.

CONSTRUCTION PROCEDURES

The ISCR operation generally uses a train of portable equipment, including milling machine, crusher with suitable aggregate screening decks, pugmill mixing chamber with controls for quantity of aggregate and emulsion, and emulsion tank or attachments, which connect an emulsion transport directly to the recycling train. The laydown machine with attachment to pick up the windrow of recycled mix usually follows a short distance behind. The compaction equipment, primarily heavy pneumatic rollers, may follow at some distance behind the laydown operation, as moisture content and emulsion break rate are critical variables in achieving density.

An ISCR project is begun with the job mix formula developed by the laboratory. The recycled mixture is observed for coating, dispersion, and balling of the fines. The application rate is then adjusted up or down as needed to achieve desired results. Where necessary, the grade of emulsion may be changed, or specific emulsion additives may be used to improve the end product. It is important that metering equipment be calibrated periodically to ensure that the correct amount of emulsion is being introduced.

The amount of water added during the milling operation should also be carefully controlled. Some water is needed to cool the mill head, and the same moisture can help disperse the emulsion throughout the mix and aid compaction. However, too much moisture can inhibit compaction and may also cause the emulsion to coat only the fines. Spilling water on the subgrade is also to be avoided, because the excess moisture will eventually migrate to the surface, creating soft spots, rutting, shoving, or other problems typical of moisture damage.

The laydown machine should operate as close to the milling/mixing train as possible, because the recycled mix is generally most workable when the moisture content is high and the emulsion is not completely broken. However, it is preferable to operate the entire ISCR process at the lowest moisture content possible, because this reduces the required aeration/drying time before compaction can begin.

Another critical variable for ISCR is the compaction procedure. NMSHD determined early in its cold recycling work that conventional roller patterns established for hot mix asphalt concrete do not work and may create serious problems. Initial breakdown with a steel wheel roller seals the surface and traps water in the mix, resulting in the various moisture damage failures discussed previously. To prevent moisture entrapment, the compaction procedure requires breakdown to be done with pneumatic rollers. In addition, ISCR mixes tend to "fluff," which implies that the uncompacted lift is quite thick compared with the same amount of conventional hot mix. Heavier rollers are essential to good compaction. New Mexico specifies a minimum weight of 30 tons for the pneumatic rollers on ISCR jobs. The pneumatic roller continues to work a section until it has "walked out of the mix"

TABLE 2 PHYSICAL PROPERTIES OF POLYMER-MODIFIED HIGH FLOAT EMULSIONS USED FOR ISCR

Emulsion	HFE I	150S	HFE 3 grade		HFE 300S grade III	
	Min	Max	Min	Max	Min	Мах
Saybolt Furol Viscosity 122 F (sfs)	50		50		50	
Sieve Test (%)		0.10		0.10		0.10
Oil distillate volume of emulsion(%)		3		7		7
Asphalt Content (%)	65		65		65	
Tests on Residue From Distillation:						
Float Test @ 140 F (seconds)	1200		1200		1200	
Penetration on residue @ 77 F	150	300	300		300	
Solubility in TCE (%)	97.5		97.5		97.5	
Tensile Stress @ 800% elongation:						
at 39.2 F (kg/cm2)	1					
at 14 F (kg/cm2)			0.05		0.15	

(i.e., indentations are leveled out and no further deformation occurs under the rubber tires).

Moisture content is also critical during compaction. If there is not enough, the mixture is harsh and will not compact. With too much moisture, the mixture cannot compact because of excess fluids and no air voids, or sealed-in water leads to future problems under traffic. If moisture levels are correct, compaction should begin just as the emulsion begins to "break," or lose moisture. This phenomenon is highly dependent on ambient air temperature and humidity and usually varies from 45 min to 2 hr. Compaction is rarely delayed more than 2 hr simply because the rollers fall too far behind the milling train. If the rollers are delayed longer than 2 hr, the mix begins to stiffen and additional passes are often required to achieve density.

When the pneumatic roller has "walked out" of a section, the density is checked with a nuclear density gauge. Then a steel wheel roller is used to level the surface and achieve final density. To ensure that this compactive effort is sufficient, the following procedure has been established to develop a target density for the recycled mix:

A sample of loose recycled mix is collected from the roadway just ahead of the first roller. This material is placed in an oven at 140°F (60°C) for two hours or until the mixture reaches the prescribed temperature, whichever comes first. The mix is then compacted immediately using the standard 50 blow Marshall procedure. The bulk specific gravity of the compacted specimen is determined. The target for roadway compaction is 96% of the Marshall compacted field briquette.

Using this procedure, a rolling pattern is developed for the project. The same Marshall compacted specimens are also tested for unconfined compressive strength to confirm that the resulting field mix conforms to the original laboratory design or can withstand expected traffic loadings. Fortunately, the recycled mixture can be expected to gain strength with time. Table 3 gives results for unconfined compressive strength at time of construction and after some months time lapse for six ISCR projects. It seems reasonable to predict from these data that the unconfined compressive strength will approximately double during the first 2 months in service.

NMSHD has prepared a video tape covering ISCR procedures and guidelines as a training aid for construction personnel (7). Copies of this tape are available on request.

RESULTS AND CONCLUSIONS

Table 1 includes a partial listing of New Mexico's 1853.9 lane miles of ISCR projects with related details such as location, length, thickness, costs, amount and type of emulsion, pavement structural features, traffic, type of wearing surface, and laboratory results for such parameters as in-place densities and unconfined compressive strength. The authors will supply additional details on request. Records for each project are kept by the Materials Division of NMSHD.

To evaluate ISCR performance, annual statewide tours are made of the projects. To date, the rideability has been characterized as excellent, rutting minimal, and resistance to cracking greatly improved. For example on one project (US Route 64 at Raton Pass), the traffic lanes were rehabilitated using ISCR, whereas the shoulders were not. The entire road surface was then overlaid with hot mix. After 1 year reflective cracking was evidenced in the shoulders, but it was 5 years before there was any sign of cracking in the traffic lanes.

Although the ISCR pavements are generally sealed, in cases where it has been impossible to seal the same season because of weather conditions, there has been no evidence of pavement deterioration.

The construction-related details and early performance (no failures) of ISCR projects using polymer were so encouraging that polymer-modified high float emulsions have been specified for all ISCR projects in New Mexico since 1986. Initial compression strengths averaged 259 psi on studied projects, whereas cores taken after about 1 to 1.5 years averaged 490 psi. Table 3 indicates that the ISCR approximately doubles in strength with time.

ISCR is unquestionably an effective method to conserve valuable resources and save dollars by salvaging old, worn out asphalt pavements. To ensure success, specifications must be clear but flexible, laboratory design should be effective but limited in scope, the user-contractor-supplier communication channels must remain interactive, and quality control must permeate every portion of the field operation to overcome the extreme variability in pavement and environmental conditions encountered during construction.

Beyond the conventional guidelines commonly recommended for ISCR, NMSHD has developed certain practices that are believed to be critical to their unparalleled success with such techniques, including the following:

TABLE 3 TYPICAL POSTCONSTRUCTION STRENGTH GAINS EXPERIENCED USING HIGH FLOAT EMULSIONS FOR ISCR

	Compressio (psi)	Time lapse		
Project	at con- struction	of core	before coring (months)	
ST-F-013-2 (208	297	610	2	
ST-ETF-033-1 (205)	368	727	6	
IR-010-1(43)13	238	432	17	
SP-ETF-046-(2206)	167	360	12	
ST-F-024-3 (202)	328	676	4	

During construction, 50 blow Marshall briquettes molded at 140 F and broken in straight down, unconfined compression. Roadway cores are also broken in straight down unconfined compression.

- 1. Careful selection of projects to avoid those few roadways that are not suitable for recycling. Factors such as excessive patching, base/subgrade failures, weak subgrades, and so forth eliminate a pavement from consideration for ISCR.
- 2. Selection of type, grade, and amount of virgin binder. SBS polymer-modified high float emulsions have performed well and now are used exclusively in New Mexico ISCR.
- 3. Size and sequencing of rollers. Specifications require that 30+ ton pneumatic rollers be used for breakdown and continue until the roller "walks out" of the mix. Steel wheel rollers, either static or vibratory, are used for finish rolling to smooth the surface and achieve final density.
- 4. A target density based on 96 percent of 50-blow Marshall specimens compacted from the field mix is used to predict adequate compaction of the roadway.

REFERENCES

 V. P. Puzinauskas and R. N. Jester. NCHRP Report 259: Design of Emulsified Asphalt Paving Mixtures. TRB, National Research Council, Washington, D.C., 1983.

- A. J. Van Wijk. Structural Comparison of Two Cold Recycled Pavement Layers. In *Transportation Research Record 954*, TRB, National Research Council, Washington, D. C., 1984.
- T. V. Scholz, G. Hicks, D. F. Rogge, and D. Allen. Use of Cold In-Place Recycling on Low Volume Roads. Draft for Proc., 5th International Conference on Low Volume Roads, Oregon State University and Oregon Department of Transportation, April 1990.
- J. C. Hardin, S. D. Ban, and H. E. Schweyer. Rheology of Asphalt Emulsion Residues. *Proc.*, Vol. 48, American Association of Asphalt Paving Technologists, 1979.
- H. W. Muncy, G. N. King, and J. B. Prudhomme. Improved Rheological Properties of Polymer Modified Asphalts. *Asphalt Rheology: Relationship to Mixture*. ASTM STP 941 (O. E. Briscoe, ed.), 1985.
- G. N. King, H. W. Muncy, and J. B. Prudhomme. Polymer Modification: Binder's Effect on Mix Properties. *Proc.*, Vol. 55, American Association of Asphalt Paving Technologists, 1986.
- 7. D. Dekker. Cold Mix In Place Recycling (video). New Mexico Engineering Research Institute, 1987.

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Evaluation of New Generation of Antistripping Additives

Kimo Pickering, Peter E. Sebaaly, Mary Stroup-Gardiner, and Jon A. Epps

A laboratory evaluation of antistripping additives is presented. The three additives included one lime additive added to the aggregates and two liquid additives mixed with the asphalt cements. Each additive was evaluated at various levels of concentration. Control mixtures with no additives were also evaluated. The laboratory evaluation program consisted of measuring the unconditioned resilient modulus at various temperatures and the moistureconditioned resilient modulus and tensile strength. The ratios of the unconditioned to the conditioned values were also used in the evaluation. On the basis of the results of the laboratory evaluation program, lime reduces the stripping potential of asphalt concrete mixtures. The liquid additives did not provide any significant improvement in the measured strength parameters (resilient modulus and tensile strength) of asphalt concrete mixtures. Therefore, the liquid additives have not proven to be effective antistripping additives.

Premature pavement failures in the forms of raveling, rutting, and cracking have been a common occurrence in the western United States for the past several years. This type of damage, which requires millions of dollars in repairs, can most often be attributed to the loss of adhesion between the asphalt cement and the aggregate surface. Also referred to as stripping, the loss of adhesion is the result of moisture infiltrating the asphalt concrete layer. Partial or complete stripping leads to a strength loss on the order of 70 to 95 percent (1).

Several methods may reduce the moisture sensitivity of asphalt concrete mixture. Lime added to the aggregate or liquid antistripping chemicals added to the asphalt cement are commonly used throughout the United States as antistripping agents. It is believed that the lime changes the surface chemistry and polarity of the aggregate surface, producing a stronger adhesion with the asphalt cement (1). Liquid antistripping additives act as surfactants and allow the asphalt to coat the aggregate particles more easily and, at the same time, to displace adsorbed water on or near the surface of the aggregate particles (2).

BACKGROUND

Stripping in asphalt concrete pavements has been a nagging problem for many state agencies and materials engineers for the past several decades. It causes millions of dollars in pavement damage. Research efforts have been expanded to min-

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imize or reduce the problem by the use of antistripping additives. The first use of such additives can be dated back to the early 1930s, when stripping was recognized as an adhesion problem (2).

The principal objective of any antistripping agent is to strengthen the bond between the asphalt cement and the aggregate surface. Chemical antistripping additives are readily soluble in asphalt cement and are designed to decrease the surface tension between the asphalt and aggregate surface, thereby allowing the aggregate to be more easily wetted by the asphalt (3). These types of chemicals are known as surface active agents, or surfactants. Common examples are the soaps and detergents used to impart wetting characteristics to all types of aqueous solutions.

Materials such as hydrated lime have proven to be effective in reducing the stripping potential of asphalt concrete mixtures. Hydrated lime functions in part like mineral filler and can also help to alter the chemistry of an aggregate surface. Several theories have been recognized as to why lime is so effective. First, lime improves the bonding of calcium with silicates in aggregate. Second, there is a possible interaction with the acidic portions of the asphalt. Third, aggregates with clay coatings have ion exchange and pozzolonic reactions between the calcium in lime and the silica in clay (1). Previous research studies have shown that the effect of lime on the moisture sensitivity of asphalt-aggregate mixtures depends on other variables: types of lime, methods of applying lime to the mixtures, changes in aggregate sources, and air voids present in the pavement.

RESEARCH PROGRAM

The objectives of this research program were to conduct a laboratory experiment to evaluate the effectiveness of (a) the various antistripping agents in reducing or eliminating the stripping potential of asphalt concrete mixtures and (b) the two liquid antistripping chemicals against the Type N hydrated lime.

DESIGN OF LABORATORY EXPERIMENT

The design of the research program consisted of selection of the materials (i.e., additives, asphalt, and aggregates), design of mixtures, and selection of laboratory tests.

Table 1 gives a summary of the experimental design. Each asphalt concrete mixture (i.e., aggregate source) was tested

TABLE 1 TEST MATRIX

		Anti Strip Agent								
1		Lir	ne	I	iquid	I	Li	iquid	п	
1		1	2	0.5	1	2	0.5	1	2	
Helms	L	L	L	L	ML	ML	L	ML	ML	
Doyle	L	L	L	L	ML	ML	L	ML	ML	

0.5, 1, and 2 = Represent the percent of antistrip agent.

 $L={\sf Test}$ sequence $L, {\sf Temperature}$ and Moisture sensitivity, ${\sf M}={\sf Test}$ sequence ${\sf M}, {\sf Limited}$ mix design

with no additive (control), and with all four additives at various percentages. This setup allowed for several multiple comparisons, which will be discussed in the data analysis section.

MATERIALS

One lime product representing a normally hydrated Type N lime was tested in this program. Two new generations of antistripping chemicals were used as additives to the asphalt cement. Liquid Additive I is a new generation additive produced by Exxon Chemical, and Liquid Additive II is a product of Unichem International. An AR- 4000 asphalt cement grade was used to prepare all the mixtures. It was supplied by Witco's Golden Bear refinery located in Oildale, California. Aggregates were obtained from two sources located in northern California and northern Nevada. The northern Nevada aggregate (Helms aggregate), which has a history of stripping potential, was obtained from a river deposit in Sparks, Nevada. The total gradation consists of crushed coarse aggregates and a blend of crushed and natural sand. The California aggregate was obtained from a limestone quarry in Doyle,

California (Doyle aggregate). The aggregate gradation shown in Figure 1 was selected to provide a gradation that would meet three standard specifications: Nevada Department of Transportation Type II, Caltrans 3/4-in. maximum size, and ASTM dense mixture ½-in. maximum size.

MIXTURE DESIGNS—TESTING SEQUENCE

The basic Hveem mix design presented in the Asphalt Institute's Manual Series No. 2 (4) was followed with two exceptions. First, samples were extruded from the molds and allowed to cool to 77°F, then the resilient modulus and the bulk specific gravity were determined. The resilient modulus was determined for a loading frequency of 0.33 Hz, with a load duration of 0.1 sec and a rest period of 2.9 sec. Second, the samples were reheated to 140°F for 2.5 hr (± 0.5 hr) before determining their Hveem stability. Once the stability was determined, samples were once again cooled to 77°F, and the indirect tensile strength was determined. All testing was performed according to the applicable ASTM standards.

Because the gradation was fixed, and therefore not adjusted to achieve the minimum of 35 stability, the selection of the optimum asphalt cement content was based solely on the binder content required to achieve 4 percent air voids. However, for all selected optimum asphalt contents, the minimum stability value of 35 was achieved.

LABORATORY TESTS

As discussed earlier, the major cause of asphalt concrete stripping is the infiltration of water through the pavement surface. The strength of asphalt concrete is drawn from the bonding

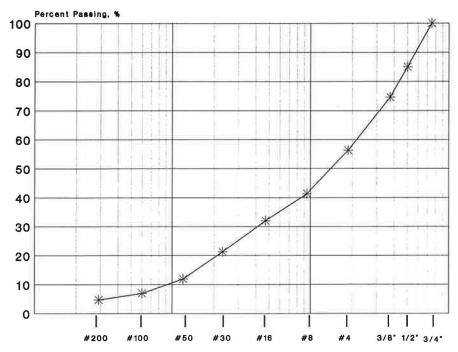


FIGURE 1 Gradation used in research project.

between the asphalt cement and the aggregates. The existence of moisture in asphaltic concrete causes major damage to the asphalt cement-aggregate bonding and therefore reduces the overall strength of the mixtures. Therefore, any laboratory experiment conducted to evaluate the effectiveness of antistripping agents must investigate the effects of the agents on the overall strength of the mixtures under severe water infiltration conditions. For the laboratory experiment conducted in this research, the resilient modulus and indirect tensile strength were identified as representative values of the overall strength of asphalt concrete mixtures. The resilient modulus and tensile strength of the original mixtures and mixtures with antistripping agents are evaluated under various levels of temperature and moisture conditioning.

The moisture-conditioning sequence used in this program is a modification of the Lottman moisture-conditioning procedure (5). It consisted of subjecting the test specimens to a vacuum saturation of 24 in. Hg for approximately 10 min to achieve at least 90 percent saturation. The samples were then wrapped in plastic and placed in a 0°F freezer for 15 hr followed by a thawing period of 24 hr in a 140°F water bath. After the thawing period the samples were brought down to testing temperature by placing them in a 77°F bath for 2.5 hr (\pm 0.5hr). The resilient modulus and indirect tensile strength tests were then performed on the conditioned samples. The temperature conditioning testing sequence consisted of determining the resilient modulus of each set of three specimens at 34°F, 77°F, and 104°F.

The method of adding the antistripping agents to the asphalt concrete mixture differs from one agent to another. In the case of lime, the specified percentages of lime were mixed with the prewetted aggregates (5 percent water by dry weight of aggregate). Finally, the aggregate-lime mixtures were heated before mixing with the binder. When either liquid additive is used, the desired amount (0.5, 1, or 2 percent) was added directly to the asphalt cement at the required mixing temperature and stirred continuously for 2 to 4 min. The asphalt—liquid additive mixture is then used in the preparation of the asphalt concrete mixtures.

DATA ANALYSIS

As discussed earlier, the laboratory testing program consisted of evaluating the resilient modulus of the various mixtures under different temperatures and evaluating the resilient modulus and indirect tensile strength of the various mixtures before and after moisture conditioning. The overall objective of this research was to evaluate the effectiveness of the various antistripping additives. Therefore, the influence of the various agents on the physical properties of the mixtures had to be evaluated. The following data analyses were conducted:

- 1. Repeatability analysis of the individual laboratory tests,
- 2. Evaluation of the effects that antistripping agents have on the resilient modulus of mixtures at various temperatures,
- 3. Evaluation of the effects that antistripping agents have on the values of unconditioned and conditioned resilient modulus and the corresponding ratios, and
- 4. Evaluation of the effects that antistripping agents have on the values of unconditioned and conditioned indirect tensile strengths and the corresponding ratios.

Repeatability Analysis

The repeatability analysis consisted of evaluating the mean, standard deviation, and coefficient of variation (CV) of each set of three replicate measurements. The CV is the ratio of the standard deviation to the mean times 100. Most CV values varied between 5 and 15 percent except for the modulus at 34°F. This range of CV values is acceptable for laboratory testing data and will be considered in the next part of the data analysis (i.e., evaluation of antistripping additives).

Temperature Susceptibility of Resilient Modulus Values

The test results for this portion of the research program are given in Tables 2 through 4. Evaluation of the test results was based on comparisons made with the control mixtures. It can be seen from Table 2 that, in the two cases (Helms and Doyle), increasing the percentages of lime resulted in a slight increase in stiffness at all temperatures.

Table 3 gives the test results for both aggregates using the Liquid I additive. At ½ percent a slight increase at 77°F and 104°F was noticed along with a decrease of value at 34°F for both aggregates. A general slight increase at all temperatures was seen for both aggregates at the 1 percent concentration level. At 2 percent, the results varied between the Helms and Doyle aggregate sources. Combining this data with the repeatability analysis results, it can be concluded that there was roughly no appreciable increase of stiffness for either aggregate source.

In analyzing Liquid II, individual variability between the aggregate sources is noticed (Table 4). There was no increase of resilient modulus values for either aggregate at $\frac{1}{2}$ and 2 percent. At 1 percent concentration, a slight increase of modulus values was noticed at $34^{\circ}F$ and $77^{\circ}F$ for both aggregate types.

Moisture Sensitivity and Resilient Modulus Values

Figures 2 and 3 show that the resilient modulus after moisture conditioning is significantly improved with the addition of lime

TABLE 2 TEST RESULTS FOR EVALUATION OF TEMPERATURE SUSCEPTIBILITY (LIME, AVERAGE OF THREE REPLICATES)

Aggregate Source Additive	Air Voids	Resilient Modulus, Ksi at Temperature, (°F)				
		34°F	77°F	104°F		
Helms No Additive (Control)	9,9	3322	272	31		
Lime - 1%	9.0	3580	295	31		
Lime - 2%	9.4	4238	329	33		
Doyle No Additive (Control)	8.4	3816	264	33		
Lime - 1%	7.6	5372	321	38		
Lime - 2%	7.9	6132	360	50		

TABLE 3 TEST RESULTS FOR EVALUATION OF TEMPERATURE SUSCEPTIBILITY (LIQUID I, AVERAGE OF THREE REPLICATES)

Aggregate Source Additive	Air Voids	Resilient Modulus at Temperature, (°F)			
		34°F	77°F	104°F	
Helms No Additive (Control)	9.9	3322	272	31	
Liquid 1 - 0.5%	10.1	2145	289	32	
Liquid I - 1.0%	7.9	5491	435	37	
Liquid 1 - 2.0%	7,8	4781	276	31	
Doyle No Additive (Control)	8.4	3816	264	33	
Liquid I - 0,5%	8.5	3550	311	35	
Liquid 1 - 1.0%	9.0	4048	412	40	
Liquid I - 2,0%	8.4	3964	258	30	

TABLE 4 TEST RESULTS FOR EVALUATION OF TEMPERATURE SUSCEPTIBILITY (LIQUID II, AVERAGE OF THREE REPLICATES)

Aggregate Source Additive	Air Voids	Resilient Modulus, Ksi at Temperature, (°F)			
		34°F	77°F	104°F	
Helms No Additive (Control)	9,9	3322	272	31	
Liquid II - 0,5%	9.6	1979	249	23	
Liquid II - 1.0%	8.4	4492	280	26	
Liquid II - 2.0%	8.8	3445	219	21	
Doyle No Additive (Control)	8.4	3816	264	33	
Liquid II - 0.5%	9.4	3949	255	25	
Liquid II - 1,0%	9.4	4153	277	26	
Liquid II - 2 0%	8.9	3177	237	26	

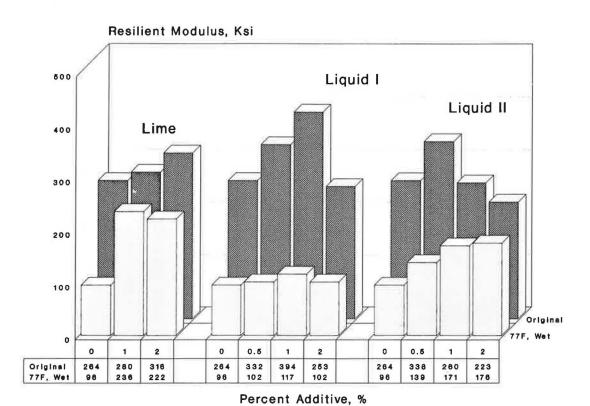


FIGURE 2 Comparison of resilient modulus before and after moisture conditioning for Helms aggregate.

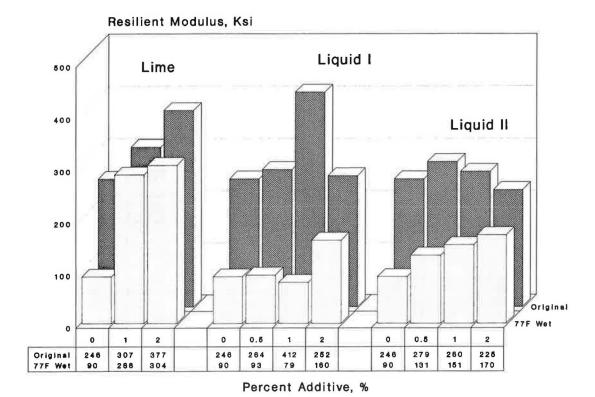


FIGURE 3 Comparison of resilient modulus before and after moisture conditioning for Doyle aggregate.

in any amount for both aggregate sources. It can also be seen that the modulus for lime mixtures for each aggregate is still increasing for the unconditioned specimens and has not leveled off. This indicates that the addition of more lime may be advantageous. For both aggregate sources, the ratios of conditioned to unconditioned resilient modulus values are at least 70 percent for all percentages of lime products (Figures 4 and 5).

In the case of the Liquid I antistripping agent, Figures 2 and 3 show that the 1 percent concentration level has significantly increased the unconditioned modulus of the mixtures with both types of aggregates. However, after conditioning there is a retention of only 30 and 20 percent for the Helms and Doyle mixtures, respectively. Figures 4 and 5 indicate that Liquid I does not generally decrease the moisture sensitivity of the mixture for either aggregate at any concentration level. Although at 2 percent some increase of the ratios is noticeable, the additive does not prove as effective as both lime products. Liquid II indicates some potential to be an effective antistripping agent. Although at most concentration levels the mixtures are not as stiff as those of the limes (Figures 2 and 3), their retention values improve with higher percentages of the additive (Figures 4 and 5). The Helms mixture has a high ratio of 80 percent, whereas the Doyle mixture has a high value of 76 percent. An observation unique to the Liquid II data is that the highest retention ratios occurred at concentration levels different from those that produce the highest unconditioned modulus values.

Moisture Sensitivity and Tensile Strength Values

The test results are shown in Figure 6 through 9 (tensile strength values and tensile strength ratios). Figures 6 and 7 show that the tensile strength after moisture conditioning is significantly improved with the addition of limes in any amount for both aggregates. The tensile strengths for lime mixtures steadily increases with increasing percentages of lime for both aggregates. A tensile strength ratio of 104 is achieved with the Doyle mix when using 2 percent lime, whereas with the Helms mixture the 1 and 2 percent concentration levels have very close ratios (Figures 8 and 9). Both ratios are above 80 percent and are considered highly acceptable. A ratio of more than 100 means that the mixture has no moisture sensitivity at all, a good indication of an optimal mix.

In the case of Liquid I, the tensile strength data are similar to the resilient modulus results. Liquid I at first shows some promising unconditioned results with strengths of 115 and 116 psi at 2 percent for the Helms and Doyle mixtures, respectively. However, after conditioning, relatively poor retained strengths are again achieved with this additive. At 1 percent for the Helms mixture, the retained strength is 70 percent, but one must consider its low unconditioned value of 72 psi compared with the lime products.

The Liquid II additive produced initial tensile strengths as high as those of the lime products, with the Helms mixture maximizing at ½ percent and the Doyle at 1 percent concentrations. The Helms mixture has increasing ratios (Figure 8);

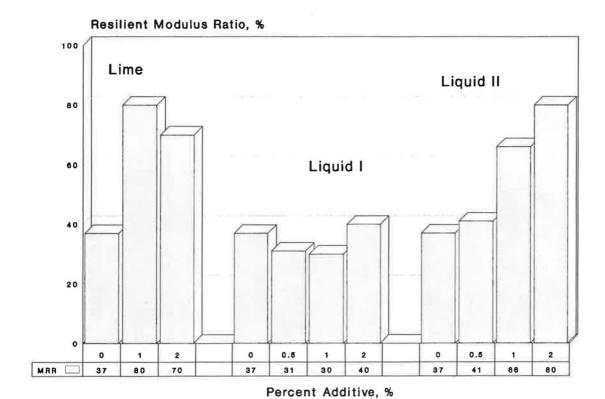


FIGURE 4 Comparison of resilient modulus ratios for Helms aggregate.

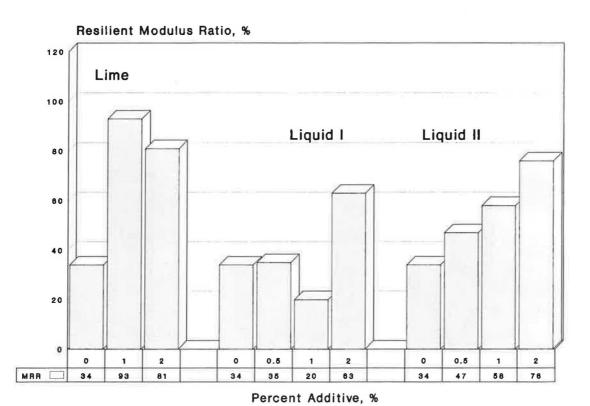


FIGURE 5 Comparison of resilient modulus ratios for Doyle aggregate.

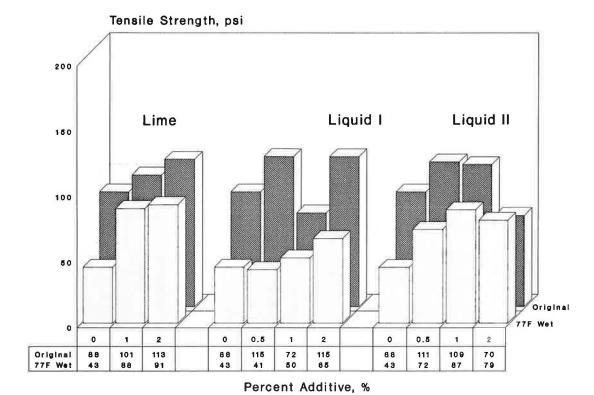


FIGURE 6 Comparison of tensile strength before and after moisture conditioning for Helms aggregate.

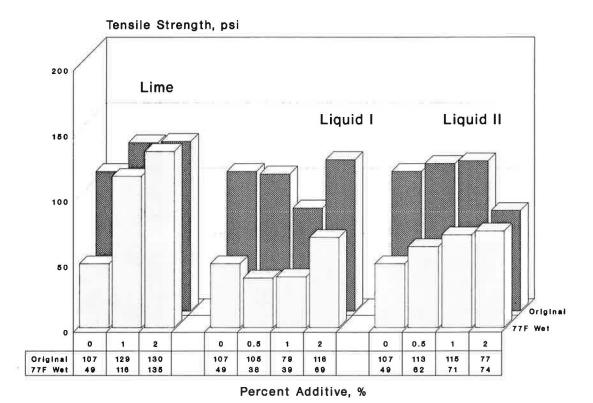
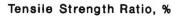
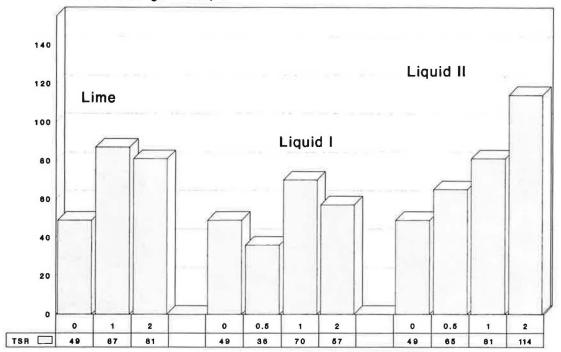


FIGURE 7 Comparison of tensile strength before and after moisture conditioning for Doyle aggregate.





Percent Additive, %

FIGURE 8 Comparison of tensile strength ratios for Helms aggregate.

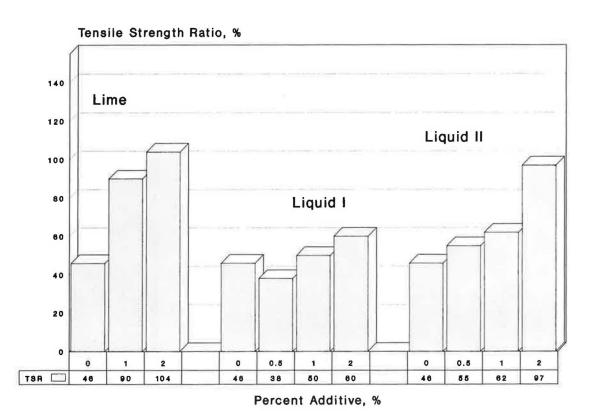


FIGURE 9 Comparison of tensile strength ratios for Doyle aggregate.

however, a large drop of unconditioned strength occurs at 2 percent with an original value of 70 psi. This high ratio coupled with the low unconditioned tensile strength is not considered highly effective. The same concept holds true for the Doyle mixture with a retained strength of 97 percent (Figure 9).

SUMMARY AND CONCLUSIONS

Three different types of antistripping additives were evaluated in this laboratory testing program. The evaluation consisted of measuring the effect of additives on the resilient modulus at various temperatures and the moisture-conditioned and unconditioned resilient modulus and tensile strength values. On the basis of the analysis of the laboratory data, the following conclusions can be drawn:

- 1. Lime additive indicates a consistent increase in the resilient modulus at all tested temperature levels for both types of aggregates (Helms and Doyle).
- 2. The effect of the Liquid I additive on the resilient modulus at various temperatures was inconsistent. In general, the 1 percent concentration was the most effective in increasing the resilient modulus at the various temperatures.
- 3. The effect of the Liquid II additive on the resilient modulus at various temperatures indicates a weak trend. The modulus values increased as a function of concentration level up to a level of 1 percent. After 1 percent the modulus values decreased (at 2 percent).
- 4. Lime indicates similar effects on the conditioned and unconditioned resilient modulus and tensile strength values of the mixtures made with both aggregate types. In general, both Lime I and II proved to be effective antistripping agents.
- 5. Liquid I additive showed a significant increase in the unconditioned modulus. However, it showed little retention in the modulus values after moisture conditioning. In the case of tensile strength some increase was obtained depending on

the aggregate type. On the basis of its laboratory performance, the Liquid I additive did not prove to be an effective antistripping agent.

6. The Liquid II additive showed a consistent improvement in the unconditioned modulus and tensile strength values. High ratios of conditioned to unconditioned values were obtained; however, these high ratios did not coincide with the unconditioned values. Although the Liquid II additive showed some increase in the modulus, tensile strength, and ratios, its effectiveness as an antistripping additive is highly questionable.

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REFERENCES

- 1. M. Stroup-Gardiner, H. Waite, and J. A. Epps. Four Variables Affecting the Performance of Lime in Asphalt-Aggregate Mixtures. In *Transportation Research Record 1115*, TRB, National Research Council, Washington, D. C., 1987, pp. 12–22.
- J. S. Coplantz, J. A. Epps, and L. Quilici. Antistrip Additives: Background for a Field Performance Study. In *Transportation Research Record* 1115, TRB, National Research Council, Washington, D. C., 1987, pp. 1–11.
- 3. D. G. Tunnicliff and R. E. Root. *Use of Antistripping Additives in Asphaltic Concrete Mixtures—Laboratory Phase.* TRB, National Research Council, Washington, D. C., 1984.
- 4. Mix Design Methods for Asphalt Concrete and Other Hot-Mix Types. Manual Series No. 2. The Asphalt Institute, 1986.
- R. P. Lottman. NCHRP Report 192: Predicting Moisture-Induced Damage to Asphaltic Concrete. TRB, National Research Council, Washington, D. C., 1978.

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Correlation Between Performance-Related Characteristics of Asphalt Cement and Its Physicochemical Parameters Using Corbett's Fractions and HPGPC

S. W. BISHARA AND R. L. McReynolds

The study of a set of 20 asphalt cements reported in a recent publication is modified and extended to cover additional parameters and a precise statistical evaluation of correlations. For a given asphalt, the whole, original material is subjected to high performance gel permeation chromatography (HPGPC) using a gravimetric finish. The experimental conditions used are kept uniform for all molecular size distribution (MSD) analyses in this study. Another part of asphalt is fractionated by ASTM D 4124. Each Corbett fraction is subjected to HPGPC analysis. The asphaltene compatibility index (ACI) is determined as proposed by Branthaver et al. A part of original asphalt is subjected to 16hr TFOT, and MSD of the aged sample is determined. Two asphalts from the same refinery showed MSD and ACI data that are outliers with respect to the other 18 asphalts. For many correlations, excluding these two asphalts causes the correlation coefficient, r, to change appreciably because r is not resistant (i.e., one or more extreme entries can be influential). Several correlations are evaluated. Depending on the number of asphalts involved, n, the numerical value of r indicates whether a correlation is not significant, significant, or highly significant; the sign of r indicates the direction of slope of the regression line. Considering only the significant and highly significant correlations, the sign of associations tested indicate that a high LMS/MMS ratio [the molecular size index (MSI)] for original asphalt accompanies a low ACI, a minimum temperature susceptiblity, a high resistance to aging (minimum change of MSI due to 16-hr TFOT), and a high viscosity, 135°C. From the MSD of original asphalt, eight with the highest MSI show common parameters that predict a promising field performance.

Fourteen out of every 15 mi of surfaced roads in the United States is topped with asphalt. The amount spent annually on asphalt pavement is \$10 billion, of which \$3 billion is for the binder itself (I).

Asphalt is an extremely complex material with no specifications for its chemical composition or structure. There is mounting evidence that the physical properties by themselves are not sufficient to predict pavement performance (2). Garrick and Wood (3) reported having asphalts with similar rheological properties but different chemical composition. Other investigators (4,5) pointed out asphalt cements with same specifications but different chemical compositions.

An important property of asphalt (and other polymers) is the molecular size distribution (MSD). In a previous inves-

tigation (6), a set of 20 original asphalt cements (n = 20) was studied. The set covers a wide viscosity range (500 to 2,200 poises) and was collected from 14 different refineries. The rheological properties were measured. The MSD of the whole set was carried out; the chemical analysis of 11 out of the 20 samples was achieved using Corbett (7) ASTM D4124, Method B. Although some parameters [e.g., penetration viscosity number (PVN), 135°C; viscosity, 135°C; and viscosity temperature susceptibility (VTS)] indicated correlations with MSD that are highly significant, other parameters, such as penetration, 25°C; viscosity, 60°C; viscosity ratio, 60°C; and colloidal instability index (8) indicated nonsignificant correlations with MSD. Classification of a correlation between two variables as highly significant, significant, or not significant follows the generally accepted practice of considering a correlation highly significant if for a given number of pairs of variables (n-2) the value of r, irrespective of the sign, is beyond its tabulated value at the 1 percent level of significance (p = 0.01). The correlation is not significant if the value of r is less than its reported value at the 5 percent level of significance (p = 0.05); the correlation is significant if the value of r exceeds the value reported at the 5 percent level (9-11).

The previous study (6) has dealt with the MSD of whole asphalt samples. To better understand the structure of asphalt cement, however, fractionation by various procedures may be involved (12-15). Branthaver et al. (12) analyzed asphalts by ion-exchange chromatography (IEC). The neutral (nonpolar) fraction from IEC was then analyzed by preparative high performance gel permeation chromatography (HPGPC) (13) into seven fractions; each of the two middle fractions was separated into saturate and aromatic components by chromatography on silica gel (14). Each of the two parts, saturate and aromatic, was then analyzed by supercritical fluid chromatography (SFC) to get the range of carbon numbers. Stegeman et al. (15) applied residual oil supercritical extraction (ROSE) to fractionate asphalt. Each fraction was then analyzed by ASTM D4124, Method B. Ramond et al. (16) reported that the grade of asphaltenes and their tendency to associate to form agglomerates (interaction index) are behind the changes of rheological properties that occur during hot

In the present work, the Corbett method was used to fractionate the 20 original asphalts used previously (6). Each fraction was then analyzed by semipreparative HPGPC according

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TABLE 1 PHYSICAL PROPERTIES OF 20 VIRGIN ASPHALTS

* Sample	Pen 25 ^O C, mm	Vis. 60°C, poises	Vis.60°C (after TFOT) poises	Vis. Ra- tio	Vis. 135 ⁰ C, est	VTS	PVN 60°C	PVN 135 ^O C
82-1214	93	1678	3966	2.36	416	3.454	-0.33	-0.25
1332	79	1141	3056	2.68	271	3.659	-0.99	-1.06
1522	85	1727	5051	2.93	398	3.500	-0.45	-0.42
2746	87	1204	2765	2.29	305	3.579	-0.79	-0.83
83-3203	65	2149	6335	2.95	395	3.591	-0.64	-0.73
3284	99	1464	3530	2.41	369	3.500	-0.37	-0.36
85-1230	85	1311	3253	2.48	359	3.454	-0.74	-0.57
3695	94	1408	3693	2,62	352	3.522	-0.50	-0.49
3890	94	858	1822	2.12	248	3.614	-1.03	-1.02
4116	96	1368	3844	2.81	321	3,591	-0.50	-0.60
86-3063	93	1353	3960	2.93	360	3.489	-0.56	-0.4
3113	96	1265	5127	4.05	326	3.545	-0.58	-0.5
87-2040	94	881	2361	2.68	223	3.727	-1.00	-1.18
88-2381	85	1275	1435	1.12	307	3.591	-0.77	-0.78
2192	162	501	1248	2.49	233	3.432	-0.74	-0.5
2483	119	979	3214	3.28	301	3.492	-0.51	-0.4
2570	130	707	1386	1.96	360	3.204	-0.72	-0.0
2787	93	1123	2483	2.21	450	3.216	-0.76	-0.1
3680		1090	2295	2.10	343	3.432	-0.60	-0.40
3719		994	2589	2.60		3.557	-0.73	-0.72

*The first two digits refer to the year sample was received.

**PVN, 60° C = $\frac{-1.5(6.489 - 1.59 \log_{10} \text{ Pen } 25^{\circ}\text{C} - \log_{10} \cap 60^{\circ}\text{C})}{1.05 - 0.2234 \log_{10} \text{ Pen } 25^{\circ}\text{C}}$ Ref.(25)

***PVN, 135° C = $\underline{-1.5(4.258 - 0.79674 \log_{10} \text{ Pen } 25^{\circ}\text{C} - \log_{10} \text{ n } 135^{\circ}\text{C})}$ Ref.(3) 0.795 - 0.1858 \log_{10} Pen 25°C

to the method described earlier (6,17). Compatibility tests were carried out on the whole, original asphalt samples according to Branthaver et al. (18). The original asphalts were aged by the 16-hr TFOT method (ASTM D1754), then subjected to semipreparative HPGPC analysis. Physical properties of the whole asphalts before and after aging are given in Tables 1 and 2. The reasonably large number of asphalt cements studied and the fact that they are randomly selected (random here means that the asphalts cover a wide viscosity range and a large number of refineries) make it possible to consider the correlation coefficient, r, to be an unbiased estimate of μ , the correlation coefficient for the population of asphalt cements, that is, generalize the association relationships to cover the wide range of asphalt cements. Another objective is to use MSD data to pinpoint from among the 20 asphalt cements those with potential for good field performance.

EXPERIMENTAL

Apparatus

A Waters HPGPC system was used. This consisted of a solvent delivery system, a U6K injector, a Digital Equipment

Corporation computer, a printer, and a Waters system interface module. A Phenomenex, 5- μ m, 500-Å phenogel semi-preparative column (300 \times 22.5 mm) with THF as solvent was used.

Procedure

Fractionation of Original Asphalts

Carried out by ASTM D4124, Method B. n-Hexane was used instead of n-Heptane.

Molecular Size Distribution Analysis

Preparation of Sample Solution For whole, original or laboratory-aged asphalt, weigh accurately (to within 0.01 mg) an asphalt sample in the range of 2.0 to 2.5 g. Add about 25 mL of THF and sonify for 15 min at room temperature. Transfer quantitatively to a 50-mL volumetric flask and complete to volume with THF. An aliquot is then filtered through 0.2- μ m membrane. An exact volume (100 to 200 μ L) chosen

TABLE 2 PHYSICAL PROPERTIES OF VIRGIN ASPHALTS AFTER 16-hr TFOT

Sample	Pen 25 ^O C, mm	Vis. 60°C, poises	Vis. 135 ^o C, cst	VTS	PVN 60 ^O C	PVN 135 ^O C
82-1214	41	19456	1190	3.568	0.79	0.31
1332	22	11656	704	3.773	-0.58	-0.94
1522	30	56352	1270	3.875	1.27	0.66
2746	20	20456	910	3.773	-0.22	-0.71
83-3203	24	39750	1269	3.761	0.62	-0.15
3284	30	25203	1256	3.614	0.54	0.05
85-1230	23	12747	930	3.602	-0.44	-0.58
3695	30	22308	1035	3.716	0.43	-0.19
3890	24	11395	641	3.841	-0.48	-0.98
4116	27	35449	1180	3.773	0.69	-0.13
86-3063	38	47083	1230	3.841	1.50	0.27
3113	24	93751	1442	3.943	1.37	0.00
87-2040	30	17172	696	3.920	0.20	-0.68
88-2381	22	16711	889	3.727	-0.26	-0.66
2192	50	20657	798	3.875	1.18	-0.01
2483	30	70334	1593	3.784	1.47	0.35
2570	42	8251	879	3.489	0.02	-0.06
2787	35	10632	827	3.625	-0.02	-0.33
3680	40	12425	1024	3.523	0.33	0.08
3719	30	17600	914	3.727	0.22	-0.34

to contain 6 to 7 mg of the asphalt material is injected in the HPGPC system.

For asphaltenes, polar aromatics, naphthene aromatics, and saturates, after fractionation of an original whole asphalt by ASTM D4124, each of the four fractions is kept in a small vial under helium. The dried material is dissolved in THF. An aliquot is filtered through 0.2- μ m membrane, and a suitable volume (100 to 200 μ L) is injected.

HPGPC Analysis The following conditions were maintained for analysis of all types of material, that is, whole asphalts (original and aged) as well as the four fractions generated by the ASTM D4124 method. The mobile phase consisted of 90 percent (v/v) THF plus 10 percent (v/v) pyridine flowing at a rate of 6 mL/min. The semipreparative phenogel column is maintained at 50°C. (For analysis of saturates, a Waters LC Spectrophotometer, Model 481, was used to check whether absorption of UV light at 345 nm would occur with any of the 20 samples studied.)

The material eluting from the column is collected in a series of small, glass, accurately weighed (to within 0.01 mg) petri dishes at 4.5 to 8.0, 8.0 to 11.0, and 11.0 to 14.5 min from injection. These designate the large molecular size fraction (LMS), medium molecular size fraction (MMS), and small molecular size fraction (SMS), respectively. Repeat the injection and collect each fraction in the corresponding petri dish.

The petri dishes are set aside to allow the mobile-phase solvents to evaporate at room temperature, then heated in an oven for 90 min at 200°C. The petri dishes are then cooled in a desiccator to constant weight.

The time required to equilibrate the column and consequently eliminate "tailing" effects increases from naphthene aromatics to polar aromatics to asphaltenes.

Determination of Compatibility

The asphaltene compatibility index (ACI) was determined as recommended by Branthaver et al. (18).

Aging of Original Asphalts

This was achieved by 16-hr TFOT (ASTM D1754).

RESULTS AND DISCUSSION

A previous Kansas investigation (6) dealt mainly with whole asphalt cements. The MSD was found to correlate, at different levels of significance, with the performance-related properties studied. The direction (sign) of association between MSD (considered as the independent variable) and any of the parameters tested (considered as the dependent variable) always favors a high percentage of LMS, together with low percentages for each of MMS and SMS in order to attain the desirable performance properties of low temperature susceptibility and high resistance to aging. Since the SMS content was found to be small (less than 10 percent), a high LMS/MMS ratio [the molecular size index (MSI)] appears to be an optimum characteristic.

However, the highly complex nature of asphalt has long suggested its fractionation to permit a better understanding of asphalt behavior. In the present study, the whole asphalt was fractionated by ASTM D4124, then HPGPC was used to analyze each fraction. Table 3 gives the results of the analysis of whole asphalts by the Corbett method (7) together with the colloidal instability index, $I_{\rm c}$ (8).

Of the four Corbett fractions, asphaltenes have received the most attention. Because it is composed mainly of highly polar compounds, or associations thereof, asphaltenes are believed to have a significant role in asphalt behavior. To study MSD of asphaltenes, and to establish comparisons with MSD of the whole asphalts, the conditions applied previously (6) for the whole asphalts were first used. But the following modifications were deemed necessary:

- 1. The mobile phase used in the previous study (95 percent THF + 5 percent pyridine) could not overcome the tendency of the smaller, polar molecules of asphaltenes to be retained within the column—probably by an adsorption mechanism—and elute after the total permeation volume (19). Increasing the pyridine content of the mobile phase to 10 percent, together with maintaining the GPC column at 50°C, eliminated the phenomenon almost entirely.
- 2. The time periods reported earlier (6) for collecting the fractions were selected to provide nearly equal weight of material in each fraction. However, for these fractions to more

TABLE 3 ANALYSIS OF 20 ORIGINAL, WHOLE ASPHALTS INTO	
FOUR FRACTIONS ACCORDING TO ASTM D4124, METHOD B, USING	G
n-HEXANE AS SOLVENT INSTEAD OF n-HEPTANE	

	Fraction, %*					
Sample Number	Asphaltenes	Polar Aromatics	Naphthene Aromatics	Saturates	I _C **	
82-1214	16.0	28.1	37.7	16.8	0.50	
1332	13.5	30.5	46.1	9.8	0.30	
1522	16.2	28.0	35.5	17.1	0.52	
2746	12.1	37.3	37.4	12.0	0.32	
83-3203	20.2	32.2	35.6	11.4	0.47	
3284	21.2	29.8	37.1	11.1	0.48	
85-1230	8.9	38.8	39.7	10.6	0.25	
3695	20.0	29.1	38.2	11.9	0.47	
3890	12.8	33.0	38.5	13.5	0.37	
4116	20.0	26.9	35.1	15.9	0.58	
86-3063	16.7	29.0	36.1	17.7	0.53	
3113	17.4	31.0	34.5	15.5	0.50	
87-2040	15.2	29.7	38.0	16.5	0.47	
88-2381	15.6	34.5	38.2	8.7	0.33	
2192	15.2	27.2	36.0	21.0	0.57	
2483	18.7	26.2	36.0	17.6	0.58	
2570	6.8	42.4	38.2	9.6	0.20	
2787	5.9	44.8	39.8	6.8	0.15	
3680	12.3	34.8	37.4	14.1	0.37	
3719	18.8	29.6	39.3	11.0	0.43	

Average of two results.

The colloidal instability index,

I_C = Asphaltenes, \$ + Saturates, \$ Naphthene Aromatics, \$ + Polar Aromatics, \$

accurately represent LMS, MMS, and SMS components, the cut points are adjusted so that the selective permeation region (between total exclusion and total permeation) is divided into three more or less equal time periods—4.5 to 8.0, 8.0 to 11.0, and 11.0 to 14.5 min from injection, to represent LMS, MMS, and SMS contents, respectively.

Because these modifications proved suitable to study MSD of the asphaltenes fraction (and hence the other three less polar fractions), the set of 20 whole, original asphalts was subjected to MSD analysis under the new conditions, which were then applied all over the present work (i.e., for each of the four Corbett fractions and the laboratory-aged asphalts).

MSD of Whole, Original Asphalts

As expected, changing conditions of analysis caused the MSI to be different from that reported previously for the same set of samples (6). Decreasing (by $\frac{1}{2}$ min) the period over which the LMS fraction is collected, while increasing (by $\frac{1}{2}$ min) the period of collecting the MMS fraction caused the MSI for any given asphalt to be smaller than that reported earlier (6). However, Table 4 indicates that the group of samples that

possess the highest MSI is nearly the same for the two studies. Under the present conditions, excluding sample numbers 88-2570 and 2787 for reasons mentioned later, eight asphalts with MSI of ≥ 0.42 have the common parameters: I_c between 0.25 and 0.58; viscosity, 60°C, between 1,200 and 1,700 poises; viscosity, 135°C, between 300 and 420 cst; viscosity ratio, 60°C, from 2.3 to 2.9; VTS from 3.45 to 3.59; PVN, 60°C, between -0.33 and -0.79; and PVN, 135° C, between -0.25and -0.80. Interestingly, these ranges are essentially the same as those reported previously (6). The only detectable change is in the lower limit of absolute viscosity, which increased from 1,100 to 1,200 poises. This and other evidence (20) confirm the statement that absolute viscosity appears to be a function of asphalt preparation, because it is a "made to order" property (18). This work and others (6,21) have found that correlation between viscosity, 60°C and MSD is not significant (Table 5).

Correlation Between MSD of Whole Original Asphalts and Their Physicochemical Parameters

The correlation coefficient, r, for the relationship between MSD (LMS, MMS, or LMS/MMS) and some physicochemical

TABLE 4 MSD OF 20 ORIGINAL, WHOLE ASPHALT CEMENTS

Sample		Fraction,		(LMS/MMS)
Number	IMS	MMS	SMS	
82-1214	29.5	69.4	1.1	0.43
1332	20.9	77.8	1.3	0.27
1522	31.0	66.7	2.3	0.47
2746	29.3	69.3	1.5	0.42
83-3203	27.5	70.0	2.5	0.39
3284	33.7	64.3	2.0	0.52
85-1230	30.4	68.0	1.6	0.45
3695	32.8	65.3	2.0	0.50
3890	20.4	75.8	3.7	0.27
4116	30.4	65.8	3.9	0.46
86-3063	30.9	67.5	1.6	0.46
3113	26.8	72.4	0.8	0.37
87-2040	24.6	72.5	2.9	0.34
88-2381	25.3	72.2	2.6	0.35
2192	28.3	70.2	1.5	0.40
2483	26.1	72.5	1.4	0.36
2570	41.3	57.9	0.7	0.71
2787	46.9	52.3	0.7	0.90
3680	25.7	70.1	4.1	0.37
3719	25.9	71.2	2.8	0.36

parameters of the 20 original asphalts is given in Table 5. To judge how credible a correlation might be, the numerical value of r has to pass the t test (9-11). The following equation is used to calculate t.

$$t = r[(n-2)/(1-r^2)]^{1/2}$$
 (1)

1. For the significant and highly significant correlations, the numerical value of r under the MSI for a given parameter is usually higher than, or at least equal to, the r value for either the LMS or MMS fraction alone. The correlation between asphaltenes content and MSI is significant, but correlation of asphaltenes content with each of the LMS or MMS fraction is not significant. The same observation is true for the colloidal instability index. On the other hand, for those correlations that are nonsignificant (e.g., penetration, 25°C; viscosity, 60°C;

TABLE 5 CORRELATION COEFFICIENT, r^* , FOR THE MSD DATA AND SOME PHYSICOCHEMICAL PARAMETERS FOR 20 ORIGINAL ASPHALTS

Physicochemical		MSD Dat	a**
Parameter	IMS	MMS	(LMS/MMS)≕MSI
Penetration, 25°C	0.17 ^a	-0.15 ^a	0.14ª
Viscosity,60°C	0.03 ^a	-0.04ª	-0.01ª
Viscosity,135°C	0.69 ^c	-0.68°	0.67 ^c
Viscosity Ratio,60°C	-0.14 ^a	0.19 ^a	-0.18ª
VIS	-0.83°	0.81°	-0.83 ^c
PVN,60°C	0.32ª	-0.32ª	0.24ª
PVN,135°C	0.78 ^C	-0.77 ^C	0.75 ^c
Asphaltenes, %***	-0.41ª	0.39ª	-0.48 b
Polar Aromatics,%	0.57 ^C	-0.57 ^C	0.62
Naphthene Aromatics,%	-0.11ª	0.14ª	-0.04ª
Saturates,%	-0.34ª	0.34ª	-0.39ª
Colloidal Instability,I _C	-0.39 ^a	0.38ª	-0.47 b

^{*}In calculating the value of r, the LMS (MMS or LMS/MMS) is considered the independent variable, and the physicochemical parameter is considered the dependent variable.

^{**}The SMS content hardly exceeded 4% and therefore correlation coefficients for this fraction were not calculated.

^{***}n-Hexane insolubles.

Not significant correlation.

bSignificant correlation.

^CHighly significant correlation.

TABLE 6 MSD OF THE SATURATES FRACTION OF 19 ORIGINAL ASPHALT CEMENTS

Sample		Fraction,		MSI
Number	LMS	MMS	SMS	
82-1214	12.5	86.2	1.2	0.15
1332	0.0	100.0	0.0	0.00
1522	10.3	89.7	0.0	0.12
2746	6.0	91.5	2.4	0.07
83-3203	5.3	94.7	0.0	0.06
3284	1.4	97.8	0.8	0.01
85-1230	11.3	87.7	1.0	0.13
3695	2.7	94.3	3.0	0.03
3890	5,8	92.6	1.6	0.06
4116	10.5	86.6	2.8	0.12
86-3063	8.5	89.2	2.2	0.10
3113	11.5	86.7	1.8	0.13
87-2040	5.1	93.5	1.4	0.06
88-2381	8.9	91.1	0.0	0.10
2192	4.4	94.7	0.9	0.05
2570	16.0	81.3	2.7	0.20
2787	19.9	80.1	0.0	0.25
3680	8.4	91.6	0.0	0.09
3719	0.0	100.0	0.0	0.00

PVN, 60°C; and naphthene aromatics, percent) the numerical value of *r* under MSI is even smaller than the corresponding value under LMS or MMS fraction. Therefore, the use of MSI to describe the MSD is advantageous.

- 2. The physical parameters measured, or derived from measurements, at the high temperature (135°C) show highly significant correlations with MSI. Physical parameters measured, or derived from measurements, at low temperature (25°C for penetration, 60°C for the rest) show correlations with MSI that are not significant.
- 3. The MSI shows a highly significant correlation with polar aromatics content, a significant correlation with asphaltenes content as well as with colloidal instability index, but a non-significant correlation with naphthene aromatics and saturates content. However, the correlation with saturates content is significant at the 10 percent level of significance. Thus, if we can accept the situation that there is less than a 10 percent chance that the saturates content and MSI for the population of asphalt cements are noncorrelated (i.e., there is more than a 90 percent chance they are correlated), this leaves only the naphthene aromatics fraction with a correlation that is not significant. At the other extreme, polar aromatics seem to have the strongest influence on MSI of whole asphalt.

MSD of Saturates

In accordance with the gravimetric procedure used for analysis of this and all the other types of fractions, the milligram amounts of material eluting from the HPGPC column were heated in an oven for 90 min. But only the material collected from the saturates fraction generated the distinct odor of burning asphalt. None of the other fraction types behaved similarly.

One of the main advantages of a gravimetric finish (17) over an UV absorption method for MSD analysis is inability of the latter to account for components of the saturates fraction—usually 10 to 25 percent of an asphalt. For the set of 20 samples investigated, the maximum absorbance reading at 345 nm was practically zero; saturates of only two asphalts showed a maximum absorbance of 0.024. However, this slight absorbance might be attributed to a trace of naphthene aromatics contaminating the saturates (22).

The MSD of saturates is given in Table 6; the saturates from one asphalt sample were lightly colored, indicating presence of a minute quantity of naphthene aromatics, and therefore were not included. For any given asphalt, the MSI of saturates is smaller than the corresponding value for whole asphalt. Table 6 indicates that saturates are composed mainly (85 to 100 percent) of medium-size molecules; the only exception is the two unique asphalts 88-2570 and 2787.

MSD of Naphthene Aromatics

The naphthene aromatics fraction of one asphalt cement was accidentally lost. Table 7 gives MSD of 19 naphthene aromatics fractions. The MSI for naphthene aromatics of any given asphalt is always larger than that obtained for saturates (Table 6) but still smaller than that of the whole asphalt cement (Table 4).

TABLE 7 MSD OF THE NAPHTHENE AROMATICS FRACTION OF 19 ORIGINAL ASPHALT CEMENTS

Sample		Fraction,	Ł	MSI
Number	IMS	MMS	SMS	
32-1214	18.3	81.4	0.3	0.22
1332	12.1	87.1	0.8	0.14
1522	17.2	81.4	1.4	0.21
2746	26.2	72.8	1.0	0.36
83-3203	10.2	88.2	1.6	0.12
3284	11.3	87.0	1.7	0.13
85-1230	16.8	81.4	1.8	0.21
3695	11.1	88.2	0.7	0.13
3890	14.2	84.6	1.2	0.17
4116	15.0	84.6	0.4	0.18
86-3063	16.8	81.3	1.9	0.21
87-2040	13.2	85.9	0.9	0.15
88-2381	18.0	80.1	1.9	0.22
2192	27.1	71.7	1.2	0.38
2483	12.9	87.2	0.0	0.15
2570	35.7	63.3	1.1	0.56
2787	42.1	56.3	1.6	0.75
3680	17.5	80.3	2.1	0.22
3719	8.1	91.6	0.3	0.09

MSD of Polar Aromatics

Table 8 gives MSD of the polar aromatics of 19 asphalt cements. In comparison with the three other Corbett fractions, MSI of the polar aromatics fraction is the closest to MSI of the whole asphalt material (Table 4).

MSD of Asphaltenes

Tables 9 and 10 indicate the following:

- 1. The MSI of an asphaltene fraction is higher than the corresponding value of any of the other Corbett fractions. Typically, about two-thirds (60 to 70 percent) of an asphaltene fraction is composed of large-size molecules.
- 2. For any given asphalt, the MSI increases going from saturates to naphthene aromatics to polar aromatics to asphaltenes. Thus, as the polarity increases, the MSI also increases. All of the fractions of the 20 asphalts investigated met this criterion.

Stegeman et al. (15), using ROSE method for fractionation, reported a progressive increase in molecular size with fraction number. They also found that the asphaltenes content is highest in the bottom fraction, whereas saturates are highest in the first fraction; the naphthene aromatics content peaks in a lighter fraction than polar aromatics. Although the fractionation techniques and the routes followed are different (Stegeman et al. used ROSE then Corbett, this study used

TABLE 8 MSD OF THE POLAR AROMATICS FRACTION OF 19 ORIGINAL ASPHALT CEMENTS

Sample		Fraction,	t	MSI
Number	LMS	MMS	SMS	****
82-1214	31.6	66.3	2.2	0.48
1332	31.3	67.8	0.9	0.46
1522	36.9	60.2	2.9	0.61
2746	28.2	71.1	0.7	0.40
83-3203	29.5	68.4	2.1	0.43
3284	34.3	57.9	7.7	0.59
85-1230	36.8	61.6	1.5	0.60
3695	29.4	69.2	1.4	0.42
3890	33.2	63.6	3.1	0.52
4116	34.6	63.9	1.5	0.54
86-3063	28.9	68.6	2.5	0.42
87-2040	26.8	70.8	2.4	0.38
88-2381	29.5	67.0	3.5	0.44
2192	30.3	66.5	3.3	0.46
2483	29.4	69.6	1.1	0.42
2570	61.7	38.0	0.3	1.62
2787	43.6	52.7	3.6	0.83
3680	37.2	61.4	1.5	0.61
3719	27.5	72.1	0.4	0.38

TABLE 9 MSD OF THE ASPHALTENES FRACTION OF 20 ORIGINAL ASPHALT CEMENTS

Sample		Fraction	. 8	MSI
Number	LMS	MMS	SMS	1201-15-16
82-1214	71.7	24.3	4.0	2.95
1332	57.8	37.2	5.0	1.55
1522	67.9	27.4	4.7	2.48
2746	59.7	32.1	8.0	1.86
83-3203	61.6	32.0	6.4	1.93
3284	66.8	26.5	6.7	2.52
85-1230	63.0	32.0	4.9	1.97
3695	64.9	31.7	3.3	2.05
3890	60.7	33.9	5.5	1.79
4116	67.7	28.5	3.8	2.38
86-3063	68.8	26.2	5.0	2.63
3113	60.9	30.7	8.4	1.98
87-2040	61.9	32.8	5.2	1.89
88-2381	61.4	35.1	3.5	1.75
2192	69.1	25.0	5.9	2.76
2483	61.5	31.4	7.0	1.96
2570	59.7	29.7	10.6	2.01
2787	62.4	28.3	9.3	2.20
3680	57.1	33.4	9.4	1.71
3719	65.1	29.0	5.9	2.24

Corbett then HPGPC), the two studies come to the same conclusion about the interrelationship between MSD of asphalt and polarity of constituents.

- 3. For a given asphalt, the MSI for polar aromatics is the nearest in magnitude to the corresponding value of the whole asphalt (Table 4). From the four fractions, MSI of polar aromatics is the closest to the MSI of whole asphalt (compare Tables 4 and 10).
- 4. The 88-2570 and 2787 asphalts have the highest MSI for the first three fractions but not for asphaltenes. The dominance of large molecules in these two whole, original asphalts as opposed to other asphalts (6,21) may then be mainly attributed to a higher-than-normal percentage of large molecules in the naphthene aromatics and polar aromatics fractions; the same is true but to a lesser degree in the saturates fraction.

Correlation Between MSI of Corbett Fractions and Physicochemical Parameters of Original Asphalts

To find r for these relationships, the MSD of a Corbett fraction is considered the independent variable, and the physicochemical property is considered the dependent variable. Table 11, like Table 5, indicates that the MSI has generally nonsignificant correlations with parameters measured, or derived from measurements, at low temperature (25°C for pen-

TABLE 10 VARIATION OF MSI ACROSS THE CORBETT FRACTIONS OF ORIGINAL ASPHALT CEMENTS

	MSI						
Sample Number	Saturates	Naphthene Aromatics	Polar Aromatics	Asphaltenes			
82-1214	0.15	0.22	0.48	2.95			
1332	0.00	0.14	0.46	1.55			
1522	0.12	0.21	0.61	2.48			
2746	0.07	0.36	0.40	1.86			
83-3203	0.06	0.12	0.43	1.93			
3284	0.01	0.13	0.59	2.52			
85-1230	0.13	0.21	0.60	1.97			
3695	0.03	0.13	0.42	2.05			
3890	0.06	0.17	0.52	1.79			
4116	0.12	0.18	0.54	2.38			
86-3063	0.10	0.21	0.42	2.63			
3113	0.13		*****	1.98			
87-2040	0.06	0.15	0.38	1.89			
88-2381	0.10	0.22	0.44	1.75			
2192	0.05	0.38	0.46	2.76			
2483		0.15	0.42	1.96			
2570	0.20	0.56	1.62	2.01			
2787	0.25	0.75	0.83	2.20			
3680	0.09	0.22	0.61	1.71			
3719	0.00	0.09	0.38	2.24			

etration, 60°C for the rest). Parameters measured, or derived from measurements, at high temperature (135°C) show highly significant correlations with MSI of at least some of the fractions. For example, only MSI of saturates correlates with viscosity, 135°C; MSI of saturates, polar aromatics, and asphaltenes correlates with PVN, 135°C; and MSI of saturates, naphthene aromatics, and polar aromatics correlates with VTS. The MSI of each of the fractions, except asphaltenes, correlates with MSI of the whole asphalt. However, if the asphaltenes fraction of the two unique asphalts (88-2570 and 2787) is excluded, *r* increases to 0.61, indicating a highly significant correlation.

Correlation Between Corbett Fractions' Content and Physicochemical Parameters of Original Asphalts

It is interesting to note that MSI of whole asphalt correlates with MSI of naphthene aromatics (r = 0.82, Table 11) but does not correlate with the content of naphthene aromatics (r = -0.04, Table 5). Table 12 gives the correlation between Corbett's fraction content (independent variable) and the physicochemical parameter (dependent variable). The fraction content generally correlates with some of the parameters measured, or derived from measurements, at low temperature (60°C). The asphaltenes fraction content contributes much to temperature susceptibility, as evidenced by the significant and highly significant correlations with PVN, 60°C and VTS, respectively. Shiau et al. (23) have also reported that the asphaltenes content contributes much to temperature susceptibility. Polar aromatics correlate highly significantly with VTS. The naphthene aromatics percentage correlates with PVN, 60°C.

TABLE 11 CORRELATION COEFFICIENT, r, FOR THE RELATIONSHIP BETWEEN MSI OF EACH CORBETT FRACTION AND SOME PHYSICOCHEMICAL PARAMETERS OF ORIGINAL ASPHALTS

	MSI					
Physicochemical Parameter	Saturates	Naphthene Aromatics	Polar Aromatics	Asphaltenes		
Penetration,25 ⁰ C	0.06	0.33	0.32	0.34		
Viscosity,60°C	0.00	-0.34	-0.27	0.16		
Viscosity,135 ^O C	0.59	0.35	0.33	0.33		
Viscosity Ratio,60°C	0.10	-0.36	-0.31	0.15		
VTS	-0.71*	-0.79*	-0.75*	-0.30		
PVN,60°C	0.16	-0.14	0.00	0.63*		
PVN,135 ^O C	0.63*	0.03	0.58*	0.63*		
MSI of Whole Asphalt	0.72*	0.82*	0.66*	0.28		
				(0.61)*,a		

^{*}Indicates a highly significant correlation; the rest are not significant correlations.

 $^{^{}lpha}\!\!$ Obtained after excluding the two asphalts 88-2570 and 2787.

TABLE 12 CORRELATION COEFFICIENT, r, FOR THE RELATIONSHIP BETWEEN CORBETT'S FRACTION CONTENT AND SOME PHYSICOCHEMICAL PARAMETERS OF ORIGINAL ASPHALTS

	Fraction, 8					
Physicochemical Parameter	Saturates	Naphthene Aromatics	Polar Aromatics	Asphaltenes		
Penetration, 25°C	0.44	-0.22	-0.10	-0.13		
Viscosity, 60°C	-0.11	-0.22	-0.17	0.43		
Viscosity,135°C	-0.31	-0.15	0.35	0.03		
Viscosity Ratio, 6000	0.48*	-0.35	-0.46**	0.43		
VIS	0.21	0.06	-0.59**	0.57**		
PVN,60°C	0.29	-0.56*	-0.36	0.48*		
PVN,135°C	-0.03	-0.31	0.00	-0.21		

^{*}Significant correlation.

From Tables 11 and 12, neither the penetration, 25°C, nor the viscosity, 60°C is correlated with the content or with MSI of any of the four Corbett fractions. The viscosity, 135°C does not correlate with the percentage of any of the fractions, but it correlates with MSI of saturates. Viscosity ratio does not correlate with MSI of any of the four fractions, but it correlates with saturates and polar aromatics content. The VTS correlates with polar aromatics content as well as its MSI. It also correlates with MSI of saturates and naphthene aromatics, as well as with asphaltenes content. Not surprisingly, VTS correlates with both the MSI and the content of some of Corbett fractions, since VTS involves measurements at the two temperatures of 60°C and 135°C. The PVN, 60°C correlates with asphaltenes content as well as its MSI; it also correlates with naphthene aromatics content. The PVN, 135°C correlates with MSI of saturates, polar aromatics, and asphaltenes.

The positive association between viscosity ratio and saturates content (Table 12) indicates that a small percentage of saturates favors a low viscosity ratio. The polar aromatics content is negatively associated with viscosity ratio, and therefore a low viscosity ratio would be favored by a high percentage of polar aromatics. A low VTS would be favored by

a high content of polar aromatics (negative association) and by a low asphaltene percentage (positive association). Therefore, a low percentage of saturates and of asphaltenes together with a high percentage of polar aromatics would favor the desired properties of low viscosity ratio and low VTS. Interestingly, the same requirements would automatically favor a low colloidal instability index, I_c (8). The fourth term in the equation that describes I_c , namely naphthene aromatics content, has nonsignificant correlation with each of the viscosity ratio and VTS (Table 12).

Correlation Between MSI and Percentage of Corbett Fractions

Table 13 indicates that the polar aromatics fraction content has a highly significant correlation with its MSI. The other three fractions have nonsignificant correlations. In further contrast to the other fractions, the polar aromatics fraction has the following features: (a) the content has a highly significant correlation with MSI of the whole asphalt, and (b) the content correlates significantly with the resistance to aging (viscosity ratio) and highly significantly with temperature sus-

TABLE 13 CORRELATION COEFFICIENT, r, FOR THE RELATIONSHIP BETWEEN MSI AND PERCENTAGE OF CORBETT FRACTIONS—COMPARISON WITH THE VALUE OF r FOR WHOLE ASPHALT

Fraction Content	MSI of Corbett Fraction	MSI of Whole Asphalt
Saturates	-0.14	-0.39
Naphthene Aromatics	0.06	-0.04
Polar Aromatics	0.62**	0.62**
Asphaltenes	0.27	-0.48*

^{*}Significant correlation.

^{**}Highly significant correlation; the rest are not significant.

^{**}Highly significant correlation; the rest are not significant.

Sample Number	ACI*	
82-1214	0.938**	71.
1332	1.712	
1522	0.827	
2746	1.619	
83-3203	0.959	
3284	0.717	
85-1230	2.004	
3695	0.964	
3890	1.726	
4116	0.730	
86-3063	0.879	
3113	1.220	
87-2040	1.061	
88-2381	1.317	
2192	0.760	
2483	1.068	
2570	3.188	
2787	3.641	
3680	1.090	
3719	1.217	
Asphaltene compatibili	ty index as define	d by Branthaver et al. (18
ACI =	Isooctane Aspha	ltenes, % X

^{**}Each entry is the average of two determinations.

ceptibility (VTS) [i.e., polar aromatics is the only fraction that correlates with both the susceptibility and aging resistance (Table 12)].

Compatibility of Asphalts

According to the widely accepted micellar model, associations of polar, aromatic molecules are dispersed in a nonpolar solvent phase. The physical properties of asphalt appear to be largely determined by the ability of the solvent phase to disperse the associated phase. The state of dispersion (compatibility) is one way of characterizing each of these phases (14). Table 14 indicates that the majority of asphalts investigated have an ACI in the range of 0.7 to 2.0. Only the two unique asphalts 88-2570 and 2787 have an ACI well beyond this range. As expected, these two asphalts have the lowest n-heptane asphaltenes.

Effect of Some Physicochemical Parameters on ACI

Table 15 (top part) indicates that, excluding the two unique asphalts, the percentage of each of the four Corbett fractions

is at least significantly correlated with ACI; those of naphthene aromatics and asphaltenes demonstrate a highly significant correlation. Although not directly included in the calculation of ACI, the content of saturates, naphthene aromatics, and polar aromatics appears to affect the state of dispersion. On the other hand, MSI of the same three fractions does not seem to influence ACI (r is practically zero); only MSI of asphaltenes shows a highly significant correlation with ACI (r = -0.65). The direction of association predicts that a low ACI would be favored by a high MSI for asphaltenes (i.e., the predominance of large asphaltene molecules would favor a relatively incompatible system). Branthaver et al. (18) reported that the asphaltene molecular weight, obtained by vapor phase osmometry in toluene or pyridine at 60°C, does not relate to compatibility. The conclusion may then be that distribution of molecular sizes within asphaltenes, rather than asphaltene molecular weight, affects compatibility. In GPC analysis, the size parameter that is important is not molecular weight, but the average volume that a molecule assumes in a dynamic-flowing environment.

$$V_{h} = [\cap]M \tag{2}$$

TABLE 15 CORRELATION BETWEEN ACI AND SOME PHYSICOCHEMICAL PARAMETERS OF ASPHALTS-INFLUENCE OF TWO UNIQUE ASPHALTS

Physicoc	chemical		n	Correlation	Coefficient, r
Paramete	er	*	**	*	**
	Percentage	20	18	-0.66	-0.55^{α}
Saturate	MSI	19	17	0.64	-0.03
March 43	Percentage	20	18	0.44	0.62°
Naphther Aromatic		19	17	0.80	0.05
	Percentage	20	18	0.90	0.56 ^{\alpha}
Polar Aromatic	s MSI	19	17	0.70	0.00
	Percentage	20	18	-0.86	-0.75 ⁰⁰⁰
n-Hexane Asphalte		19	17	-0.26	-0.65 ⁰⁰⁰
n-Heptar Asphalte	ne enes (Percentage)	20	18	-0.84	-0.68 ^{αα}
$I_{\mathbf{C}}$		20	18	-0.88	0.56 ^{\alpha}
MSI Whol	e Asphalt	20	18	0.67	-0.52^{α}
Penetrat	ion, 25 ⁰ C	20	18	0.03	-0.34
Viscosit	су, 60 ⁰ С	20	18	-0.33	-0.22
Viscosit	cy,135°C	20	18	0.28	-0.28
Viscosit	y Ratio, 60°C	20	18	-0.34	-0.22
VIS		20	18	-0.64	0.26
PVN, 60°	Pc C	20	18	-0.42	-0.66°
PVN, 135	o _C	20	18	0.29 88-2570 and 2	-0.53 ^α

Including 88-2570 and 2787 asphalts. "Decluding 88-2570 and 2787 asphalts.

 α Denotes significant correlation; $\alpha\alpha$ denotes highly significant correlation. Correlations involving the two unique asphalts are not marked, and reported only for comparison.

The correlation coefficient, r, for the parameters above the dividing line was obtained by considering the physicochemical parameter as independent variable, and ACI as dependent variable; the opposite applies for parameters below dividing line.

where V_h is the hydrodynamic volume, \cap the intrinsic viscosity, and M the molecular weight. The molecular volume is determined by molecular weight, structure, configuration, and solute associations (24). An aromatic molecule with rather long, not highly branched side chains would have a large molecular volume but a relatively small molecular weight.

The colloidal instability index, I_c, shows a significant correlation with ACI; the positive association implies that a low I_c favors a low ACI. The MSI of whole asphalt correlates significantly with ACI; a high MSI favors a low ACI. The significant correlation and negative association between MSI of whole asphalt and ACI mutually confirm the validity and significance of the two new parameters.

The two parameters of MSI and ACI indicate that the values obtained for two asphalts 88-2570 and 2787 are out of the range found for the 18 other asphalts. One common rule of thumb for identifying suspected outliers (11) singles out values falling at least 1.5 times the interquartile range (IQR) above the third quartile, Q₃ (75th percentile) or below the first quartile, Q_1 (25th percentile). Applying the 1.5 \times IQR rule to the set of 20 asphalts indicates that an MSI > 0.62 and an ACI > 2.422 is an outlier. Each of the two asphalts have an MSI and an ACI that is higher than the calculated value.

When outliers exist, the first option (11) is to search for a cause and have the data corrected. In the present case, however, outliers are caused by factors associated with crude source, refinery processes, additives, or some other related criteria that are beyond the scope or control of this study. Moore and McCabe (11) state, "If some other abnormal condition caused the outlier, we can delete it from the data with a clear conscience."

Calculating r after excluding the two asphalts 88-2570 and 2787 (Table 15, top part) shows that the correlation coefficient can be significantly affected, to the point where the sign of association is reversed, that is, correlation between ACI and each of I_c, and MSI of whole asphalt. The effect of excluding the two asphalts is also evident from the correlation between ACI and MSI of Corbett fractions. The least affected are correlations between ACI and Corbett fraction's content. The two asphalts have fraction contents that are generally not much different from the whole set (Table 3), but MSI of their Corbett fractions (except asphaltenes) is quite different (Tables 6 through 9). That the value of r after excluding the two asphalts is different from that obtained for the whole set is not unexpected, however, because r is not resistant [i.e., one or more entries can greatly influence the correlation coefficient (11)]. A basic scatter plot of Y versus X shows that the two asphalts possess physicochemical parameters data (MSI of whole asphalt, ACI, I_c, MSI of saturates, naphthene aromatics, and polar aromatics) that are extreme in value. Such data may therefore be influential, and removing them would markedly change the position of regression line (11).

Effect of ACI on Some Physical Parameters

Branthaver et al. (18) reported that parameters that measure compatibility should correlate with variations in viscosity with temperature, but not necessarily with absolute viscosity. Our findings confirm this statement. Table 15 (lower part) indicates that correlation between ACI and both the absolute viscosity and kinematic viscosity is not significant. On the other hand, ACI correlates significantly with PVN, 135°C and highly significantly with PVN, 60°C; a low ACI favors a high PVN.

Excluding 88-2570 and 2787 caused r to change, sometimes markedly as for the correlation between ACI and each of viscosity, 135°C; VTS; and PVN, 135°C. From Table 15 (lower part), excluding the two asphalts significantly changes correlations involving parameters measured, or derived from measurements, at high temperature (135°C). These parameters have been shown to correlate highly significantly with MSI of whole asphalt (Table 5). Indeed, the two asphalts have proved to possess a unique MSD as opposed to the other 18 asphalts (Table 4).

Viscosity Ratio as a Measure of Resistance to Aging

The correlation between ACI and viscosity ratio is not significant (Table 15). Furthermore, viscosity ratio proved to be the least correlated to any of the parameters studied [e.g., it is not significantly correlated to MSI of whole asphalt (Table 5), MSI of any of Corbett fractions (Table 11), or the naphthene aromatics or asphaltenes content (Table 12)]. Resistance to aging may alternatively be evaluated by another parameter, such as MSI before and after aging, as shown later.

Effect of Laboratory Aging on MSI

As expected, the 16-hr TFOT caused MSI of original asphalt to increase (compare Tables 4 and 16). The rate of change (MSI of aged asphalt divided by MSI of original asphalt) may serve as a measure of the resistance to aging. The 10 original asphalts with MSI of ≥ 0.42 suffer the least change in MSD (rate of increase ranging from 1.10 to 1.50).

Correlation Between Resistance To Aging and Some Physicochemical Parameters for 18 Asphalts

The correlation between MSI of original asphalt as independent variable and rate of change of MSI as dependent variable has a coefficient of -0.55 (Table 17). The numerical value reflects a significant correlation even at the 2 percent level; the negative association indicates that original asphalts with

TABLE 16 MSI OF LABORATORY-AGED ASPHALT; RATE OF CHANGE OF MSI DUE TO 16-hr TFOT

Sample Number	MSI	MSI aged MSI original
82-1214	0.66	1.53
1332	0.46	1.70
1522	0.59	1.26
2746	0.63	1.50
83-3203	0.57	1.46
3284	0.71	1.37
85-1230	0.67	1.49
3695	0.56	1.12
3890	0.40	1.48
4116	0.67	1.46
86-3063	0.58	1.26
3113	0.86	2.32
87-2040	0.58	1.71
88-2381	0.61	1.74
2192	0.59	1.48
2483	0.65	1.81
2570	1.00	1.41
2787	1.28	1.42
3680	0.52	1.41
3719	0.63	1.75

a high MSI tend to have a minimum change in their MSD due to aging (i.e., manifest a high resistance to aging).

Table 17 indicates that resistance to aging (expressed as rate of change of MSD) is not correlated with either the percentage or the MSI of any of Corbett fractions. However, the MSI of polar aromatics and asphaltenes manifest *r* values that are not too small; the sign of association suggests that a high MSI for either fraction should accompany a low rate of change of MSD. The temperature susceptibility parameters are not correlated with the rate of change of MSD or with viscosity ratio. Viscosity ratio correlates significantly with I_c.

Correlation Between Temperature Susceptibility and Some Physicochemical Parameters for 18 Asphalts

The MSI of whole asphalt correlates significantly with VTS and highly significantly with PVN, 135°C; a high MSI favors a low VTS and a high PVN, 135°C (Table 18). The I_c correlates nonsignificantly with VTS and PVN. No correlation exists between temperature susceptibility and content of any of Corbett fractions except that of naphthene aromatics, which correlates significantly with PVN, 135°C. A low naphthene aromatics content favors a high PVN. Only MSI of asphaltenes correlates with temperature susceptibility; it correlates significantly with VTS and highly significantly with PVN, 135°C.

TABLE 17 CORRELATION COEFFICIENT, r, FOR SOME PHYSICOCHEMICAL PARAMETERS AND THE RESISTANCE TO AGING FOR 18* ASPHALT CEMENTS

Physicoche	mical	Resistar	nce to Aging	
Parameter**		Rate of Change of MSD	Viscosity Ratio	
MSI of Whole Asphalt		-0.55^{α}	0.11	
ACI		0.26	-0,22	
I _C		-0.04	0.52^{α}	
	-Percentage	-0.03	0.42	
Saturates	MSI	0.11	0.12	
Naphthene	-Percentage	0.02	-0.33	
Aromatics	MSI	-0.13	-0.28	
Polar	-Percentage	0.03	-0.42	
Aromatics	MSI	-0.40	-0.11	
Asphaltene	Percentage	-0.03	0.34	
Asphartene	MSI	-0.32	0.14	
Penetratio	on, 25 ^o C	0.06	0.04	
Viscosity,	60 ⁰ C	-0.27	0.14	
Viscosity,	135 ^o C	-0.36	0.12	
VIS		0.29	-0.03	
PVN, 60°C		-0.22	0.28	
PVN, 135 ^O C		-0.29	0.16	

^{*}For r=18; df=16 and r=0.468 and 0.590 at the 5% and 1% levels of significance, respectively.

A high MSI for asphaltenes favors a low temperature susceptibility. Correlation between the two temperature susceptibility parameters and MSI of polar aromatics is significant at the 10 percent level; a high MSI favors a low temperature susceptibility.

The penetration, 25°C and viscosity, 60°C do not correlate with susceptibility, whereas viscosity, 135°C correlates significantly with VTS and highly significantly with PVN, 135°C—in exactly the same way that the MSI of whole asphalt correlates with susceptibility (Table 18). Table 5 has also indicated that viscosity, 135°C and MSI of whole asphalt show a highly significant correlation. Thus, viscosity, 135°C; MSI of whole asphalt; and temperature susceptibility are all at least significantly intercorrelated.

CONCLUSION

For the set of 20 asphalts studied, the following conclusions are drawn:

1. MSD (expressed as MSI or LMS/MMS) of whole asphalt correlates significantly with I_c and highly significantly with ACI; viscosity, 135°C; VTS; and PVN, 135°C.

- 2. For any given asphalt, there is a continuous increase in MSI of Corbett fractions going from saturates to naphthene aromatics to polar aromatics to asphaltenes. None of the 20 asphalts is an exception. Therefore, a direct relationship exists between MSI and polarity.
- 3. Some of the parameters measured, or derived from measurements, at 60°C (e.g., viscosity ratio; VTS; and PVN, 60°C) correlates significantly with the percentages of some Corbett fractions. Parameters measured, or derived from measurements, at 135°C (e.g., VTS; PVN, 135°C; and viscosity, 135°C) correlate highly significantly with MSI of some Corbett fractions. A high MSI for asphaltenes favors a low temperature susceptibility.
- 4. Polar aromatics fraction has some distinct features. It is the only fraction that shows a correlation between content and MSI. The content is highly significantly correlated with MSI of whole asphalt, with VTS, and significantly correlated with viscosity ratio. The MSI is the closest to that of whole asphalt.
- 5. Two asphalts, 88-2570 and 2787, showed MSI, ACI, as well as MSI of Corbett fractions (except asphaltenes) that are markedly different from the 18 other asphalts. Applying the 1.5 times IQR rule for detecting outliers to the set of 20 samples shows that whole asphalts with MSI and ACI > 0.62

^{**}The physicochemical parameter is considered the independent variable, and resistance to aging as the dependent variable.

 $[\]alpha$ Denotes a significant correlation.

TABLE 18 CORRELATION COEFFICIENT, r, FOR SOME PHYSICOCHEMICAL PARAMETERS AND THE TEMPERATURE SUSCEPTIBILITY FOR 18 ASPHALT CEMENTS

Physicochem	ical _	Temperatur	e Susceptibility
Parameter*		VIS	PVN, 135°C
MSI of Whole Asphalt		-0.52^{α}	0.71 ⁰⁰⁰
Ic		-0.13	-0.45
Săturates Pe	ercentage	-0.33	0.33
	SI	-0.34	0.39
Naphthene	ercentage	0.34	-0.50^{α}
Aromatics M	SI	-0.36	0.15
Polar (Po	ercentage	0.04	-0.27
Arcmatics	SI	-0.45	0.42
Asphaltenes	Percentage	0.06	0.30
Aspharceles	L _{MSI}	-0.53 ^{\alpha}	0.65 ^{αα}
Penetration	, 25 ^o C	-0.45	0.28
Viscosity,	60 ^o C	-0.06	0.34
Viscosity,	135 ^o C	-0.50^{α}	0.71 ^{αα}

^{*}The physicochemical parameter is considered the independent variable, and the temperature susceptibility the dependent variable.

and 2.422, respectively, are outliers—a condition that applies to the two asphalts. Furthermore, data for MSI of saturates, naphthene aromatics, and polar aromatics fractions indicate that at least one of the two asphalts is an outlier. Consequently, many correlations change appreciably after excluding the two asphalts. The most noticeable change occurs for correlations between ACI and each of MSI (of whole asphalt and of Corbett fractions) and parameters measured, or derived from measurements, at 135°C.

Excluding the two asphalts (i.e., n = 18), the following conclusions are drawn:

- 6. Of the MSI of Corbett fractions, only that of asphaltenes correlates with ACI and with temperature susceptibility. A high MSI for asphaltenes favors a low ACI and a low temperature susceptibility (VTS and PVN, 135°C).
- 7. Of the percentage of Corbett fractions, only that of naphthene aromatics correlates with PVN, 135°C. None correlates with VTS or with resistance to aging.
- 8. The ACI correlates significantly with each of MSI of whole asphalt; PVN, 60° C; PVN, 135° C; and I_{c} . A low ACI accompanies a high MSI for whole asphalt; a high PVN, 60° C; PVN, 135° C; and a low I_{c} .
- 9. MSI of whole asphalt correlates significantly with ACI, temperature susceptibility, and resistance to aging (expressed as rate of change of MSD due to 16-hr TFOT), and correlates highly significantly (r=0.65) with viscosity, 135°C. A high MSI (≥ 0.62) for whole asphalt favors a minimum temperature susceptibility; a high resistance to aging; and a high viscosity, 135°C.

- 10. Viscosity, 60°C and penetration, 25°C do not correlate with any of the parameters studied: temperature susceptibility, resistance to aging, ACI, MSI of whole asphalt, or the percentage, the MSI of any of Corbett fractions.
- 11. Viscosity ratio correlates only with I_c (at the 5 percent level) but not with any of the other parameters. The rate of change of MSD of an asphalt due to aging may also serve as a measure of aging—a primarily chemical process. Rate of change of MSD due to 16-hr TFOT correlates significantly (at the 2 percent level) with MSI of whole asphalt; a high MSI for whole asphalt favors a minimum rate of change of MSI.
- 12. Because n is reasonably large and the original asphalts tested are randomly selected (random here refers to the fact that samples cover a wide range of viscosity and a large number of sources), the value of r calculated herein for a given correlation is an unbiased estimate of μ , the correlation coefficient for population of asphalt cements.
- 13. To put the preceding significant/highly significant associations together and select from among the set of 18 asphalt cements those with potential for good field performance, the analysis started with MSI of whole asphalt, because it correlates at least significantly with temperature susceptibility and resistance to aging (as expressed by rate of change of MSI). Eight asphalts with MSI ≥ 0.42 have the following characteristics: MSI of 0.42 to 0.52; ACI of 0.72 to 2.00; $I_{\rm c}$ of 0.25 to 0.58; viscosity, 135°C of 305 to 416 cst; VTS of 3.45 to 3.59; PVN, 135°C of -0.25 to -0.81; rate of change of MSI from 1.12 to 1.53; and MSI of asphaltenes from 1.86 to 2.95. To use the specified ranges for selection from among a

^{\alpha}Denotes a significant correlation.

 $[\]alpha\alpha$ Denotes a highly significant correlation.

wider collection of asphalts, the $1.5 \times IQR$ rule for identifying outliers may be used to find upper and lower permissible limits of variation (e.g., MSI ≥ 0.62 and ACI ≥ 2.422).

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Fractionation by Corbett's method and asphaltene compatibility indices were carried out by Hope Alban, who also translated from French one of the referenced papers, and entered the manuscript on the word processor.

REFERENCES

- D. J. Kulash. From the Director. FOCUS, Strategic Highway Research Program, No. 23, Aug. 1987.
- V. P. Puzinauskas. Experience with Chemical Techniques on Bitumen for the Characterization of Asphalt Cement. An Overview. Presented at the ACS 22nd Midwest Regional Meeting, Wichita, Kan., Nov. 1987.
- 3. N. W. Garrick and L. E. Wood. Relationship Between HPGPC Data and the Rheological Properties of Asphalts. In *Transportation Research Record 1096*, TRB, National Research Council, Washington, D.C., 1986, pp. 35–41.
- Washington, D.C., 1986, pp. 35-41.
 B. Brulé, G. Ramond, and C. Such. Relationship Between Composition, Structure, and Properties of Road Asphalts: State of Research at the French Public Works Central Laboratory. In *Transportation Research Record 1096*, TRB, National Research Council, Washington, D.C., 1986, pp. 22-34.
- S. W. Bishara and R. L. McReynolds. The Use of HPGPC with UV Detection for Determining the MSD of Asphalt Cement After Quantitative Correction for Molar Absorptivity Variation and Saturated Oils. In *Transportation Research Record 1269*, TRB, National Research Council, Washington, D.C., 1990, pp. 40-47.
- S. W. Bishara, R. L. McReynolds, and E. R. Lewis. The Interrelationship Between Performance-Related Properties of Asphalt Cement and Their Correlation with Molecular Size Distribution (MSD). Presented at the 70th Annual Meeting of the Transportation Research Board, Washington, D.C., 1991.
- 7. L. W. Corbett. Analytical Chemistry, 41, 576, 1969.
- C. Gaestel, R. Smadja, and K. A. Lamminan. Contribution à la Connaissance des Propriétés des Bitumes Routiers. Rev. Generale des Routes et Aerodromes, 466, 1971, pp. 85–94.
- G. W. Snedecor. Statistical Methods. Collegiate Press, Inc., Ames, Iowa, 1938.
- 10wa, 1938.10. R. L. Anderson. Practical Statistics for Analytical Chemists. Van
- Nostrand Reinhold Co., New York, 1987.D. S. Moore and G. P. McCabe. *Introduction to the Practice of Statistics*. W. H. Freeman and Co., New York, 1989.
- J. F. Branthaver, M. W. Catalfomo, and J. C. Petersen. Separation of SHRP Asphalts by Ion Exchange Chromatography. Symposium on Asphalt Chemistry and Characterization, 200th National Meeting of ACS, Washington, D.C., Aug. 26–31, 1990;

- Division of Petroleum Chemistry, ACS, Vol. 35, No. 3, 1990, pp. 376-381.
- 13. J. F. Branthaver, J. J. Duvall, and J. C. Petersen. Separation of SHRP Asphalts by Preparative Size Exclusion Chromatography. Symposium on Asphalt Chemistry and Characterization, 200th National Meeting of ACS, Washington, D.C., Aug. 26–31, 1990; Division of Petroleum Chemistry, ACS, Vol. 35, No. 3, 1990, pp. 407–414.
- 14. F. A. Barbour and J. F. Branthaver. Supercritical Fluid Chromatography Separation of SHRP Asphalt Non-Polar Fractions. Symposium on Asphalt Chemistry and Characterization, 200th National Meeting of ACS, Washington, D.C., Aug. 26–31, 1990; Division of Petroleum Chemistry, ACS, Vol. 35, No. 3, 1990, pp. 421–430.
- 15. J. R. Stegeman, A. L. Kyle, B. L. Burr, H. B. Jemison, R. R. Davison, C. J. Glover, and J. A. Bullin. Compositional and Physical Properties of Asphalt Fractions Obtained by Supercritical Extraction. Symposium on Asphalt Chemistry and Characterization, 200th National Meeting of ACS, Washington, D.C., Aug. 26–31, 1990; Division of Petroleum Chemistry, ACS, Vol. 35, No. 3, 1990, pp. 431–439.
- G. Ramond, A. Bononi, and M. Pastor. Modification des caractéristiques rhéologiques des bitumes au cours de leur évolution à l'enrobage. *Bull. liaison Labo. P. et Ch.*, 168, July-Aug. 1990, pp. 47-56.
- 17. S. W. Bishara and R. L. McReyonds. A New Approach for the Determination of MSD of Asphalt Cement Using HPGPC. Fuel Science and Technology International (forthcoming).
- J. F. Branthaver, J. C. Petersen, J. J. Duvall, and P. M. Harnsberger. Compatibilities of SHRP Asphalts. Presented at the 70th Annual Meeting of the Transportation Research Board, Washington, D.C., 1991.
- B. Brulé. Contribution of Gel Permeation Chromatography (GPC) to the Characterization of Asphalts. In *Liquid Chromatography* of *Polymers and Related Materials II*, (J. Cazes and X. Delamare, eds.). Marcel Decker, Inc., New York, 1980, pp. 215–248.
- 20. S. W. Bishara and R. L. McReynolds. In preparation.
- C. J. Glover, R. R. Davison, J. A. Bullin, J. W. Button, and G. R. Donaldson. Chemical Characterization of Asphalt Cement and Performance-Related Properties. In *Transportation Research Record* 1171, TRB, National Research Council, Washington, D.C., 1988, pp. 71–81.
- 22. S. W. Bishara and E. Wilkins. Rapid Method for the Chemical Analysis of Asphalt Cement: Quantitative Determination of the Naphthene Aromatic and Polar Aromatic Fractions Using High Performance Liquid Chromatography. In *Transportation Research Record 1228*, TRB, National Research Council, Washington, D.C., 1989, pp. 183–190.
- 23. J.-M. Shiau, M. Tia, B. E. Ruth, and G. C. Page. Characterization of Age-Hardening Potential of Asphalts by Using the Corbett-Swarbrick Asphalt Fractionation Test, Presented at the 70th Annual Meeting of the Transportation Research Board, Washington, D.C., 1991.
- 24. H. V. Drushel and W. W. Schultz. Effect of Solvents and Temperatures on the Separations of Asphaltenes by Gel Permeation Chromatography. Presented at the Symposium on Techniques for Characterization of Residual Fuels, Division of Analytical Chemistry, American Chemical Society, San Francisco, Calif., Aug. 1980.
- D. W. Christensen and D. A. Anderson. Effect of Amine Additives on the Properties of Asphalt Cement. Association of Asphalt Paving Technologists, Vol. 54, 1985, pp. 593–615.

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Reaction Rates and Hardening Susceptibilities as Determined from Pressure Oxygen Vessel Aging of Asphalts

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Many attempts have been made to simulate road aging with laboratory tests. These have generally not been successful. One reason is that other causes of road failure, such as heavy traffic or construction deficiency, can obscure the effect of the asphalt binder. But other reasons are that an adequate model relating laboratorymeasured properties to changes in the road is not available and that the oven test temperatures that have been used may be too high to relate oven aging to road aging. Use of the lowertemperature pressure oxygen vessel offers promise of approximating road conditions. It is shown that no test run at a single elevated temperature can be used to simulate road aging and that at road temperature the rate is still too low for practical testing. It is also shown, however, that the oxidative aging mechanism is constant at temperatures up to 82.2°C and that it approximates aging of the asphalt binder in laboratory cores. If this is borne out with road cores, extrapolation of higher temperature data may provide more accurate predictions of road aging.

From the standpoint of understanding and predicting asphalt performance, the properties of aged asphalt are more important in many respects than the properties of the original material. Years after placement, when a roadway is deteriorating, the asphalt properties may be greatly different from the original properties, and, as a result, if failure is due to the asphalt cement, it will be the properties of this aged material rather than those of the original that are causing the problem. This has been recognized for many years, and many test procedures have been devised to predict aging.

Aging really occurs in two modes: first, there is the very rapid aging that occurs in the hot-mix plant at high temperature on the aggregate surface. This is followed by the much slower, low-temperature aging in the road. It is likely that the mechanisms in these modes of aging are quite different. This is borne out by the persistent failure of tests that were developed to predict hot-mix aging to predict road aging accurately.

There are now two widely recognized hot-mix aging procedures: the thin film oven test (TFOT), ASTM D1754-83, and the rolling thin film oven test (RTFOT), ASTM D2872-80. Numerous variations have been made on the TFOT and RTFOT methods with many intended to simulate road aging as well. Vallerga et al. (1) proposed tilting the oven used for the TFOT and provided an effect similar to that of the RTFOT.

The recognition that thinner films may be desired to simulate hot-mix aging led to the development of microfilm tests (2,3). Thinner films correspond better to actual pavement mixtures, harden faster, and lessen any diffusion effects that may be present in thicker films. The microfilm durability test of Griffin et al. (4) was designed to simulate road aging but has been correlated with aging from the hot-mix process as well as road aging (5,6).

Schmidt and Santucci (7) developed a rolling microfilm test in which benzene-dissolved asphalt was cast in bottles and aged in a manner similar to that used in the RTFOT. The modification of adding a circulating fan, as suggested by Schmidt (8), has been maintained and is called the rolling microfilm circulating (RMFC) test. Schmidt (9) has also used several modifications to the rolling microfilm test at various temperatures and rates of air circulation as well as in the presence of aggregate in an effort to simulate hot-mix and road aging.

Kemp (10) studied several durability tests and their relationship to field hardening. Kemp stated that no laboratory process seemed to predict performance satisfactorily. The void content of the mix is the only parameter that seems to give any prediction of the aging process for pavements with more than 50 months of service. This leads to the conclusion that voids must be constant before an aging test can predict actual field hardening. The best correlation with the field data came from the RMFC test for 48-hr exposures. The RMFC test was modified by Petersen (11). The test is run at 112.8°C using a larger sample and is purported to represent about 10 years of field aging.

Evidence exists that aggregate can catalyze aging (12-17). Jennings (12) studied the aging process using gel permeation chromatography (GPC). Jennings stated that the change in the molecular size distribution of an asphalt due to aging is a function of aggregate, additives, exposure, and time.

The most realistic road aging procedures appear to be the low-temperature (compared with the hot-mix temperatures) pressure oxygen vessel (POV) tests, in which lower temperatures can be partly compensated for with respect to required test duration by higher oxygen pressure, and there are several reports using this kind of device. Lee (18) simulated the hot-mix step first by TFOT aging. This material, still in the TFOT dish, was placed in a pressure vessel and aged at 65°C at various pressures. A standard procedure was proposed by Lee (19) in which TFOT residues were aged at 65°C and 300 psi for 1,000 hr. They used a variety of criteria to correlate aging

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including viscosity, penetration, softening point, microductility, and percent asphaltenes. Though the results are promising, the rankings of the POV-aged asphalts by each criterion were not very consistent with the rankings of the field-aged asphalts, indicating that actual road aging was not being duplicated. They also found an overwhelming effect of voids that complicated relating properties to age. Jamieson and Hattingh (20) reported that POV results using 7-mm films at 65°C and 300 psi did not agree with road performance.

Edler et al. (21) reported results in which 30-µm asphalt films were contacted with oxygen at 300 psi at 65°C for 96 hr, but the results were not compared with field data. Kim et al. (22) aged compacted cores at 100 psi and 60°C up to 5 days. Comparison with field cores was on the basis of the resilient modulus and Corbett analysis of extracted material. There was generally fair agreement between the laboratory and field cores for two of three sets of cores, but they reported some problems with diffusion into cores of low air voids. It is likely that much of this work is complicated by diffusion of oxygen into cores and problems with extraction and recovery (23) of the asphalt binder from laboratory and field cores.

OBJECTIVES

As indicated, most road aging tests, which are lowertemperature variations of the oven tests that were designed to simulate the hot-mix process, do not appear to correlate to road aging. One likely reason is that the tests are still conducted much above field temperatures, so that the aging rates and mechanisms are not relevant to field aging. Another difficulty with correlation is that an adequate model relating laboratory-measured properties to changes in the road is not available.

The primary objective of this work was to determine the significance of aging temperature with respect to oxidation rate and mechanism. By so doing, a rationale would exist for designing a laboratory test that would be optimal with respect to the ease and time to conduct the test and with respect to the ability of the test to indicate or predict road aging.

APPARATUS AND PROCEDURES

The POV and sample holder designed for this work are shown in Figures 1 and 2. The vessel is made of 4-in.-diameter standard stainless steel pipe. It is 2 ft long and flanged at each end. The top flange is equipped with a 1½ in., 400-psig rupture disk with the operating pressure in the tests limited to 300 psia. The pressure vessel is wrapped with heating tape, which with a variable transformer is used to control the temperature. A thermocouple is inserted through the bottom flange to detect the temperature, which is read from a digital indicator. A control panel houses the temperature indicator as well as vacuum and pressure gauges. The pressure vessel is located behind a metal shield. All control valves are specially cleaned and teflon coated for oxygen service. Valves and tubing as shown in Figure 1 are stainless steel. The sample holder is also stainless steel. It fits inside the pressure vessel and can support 88 asphalt samples.

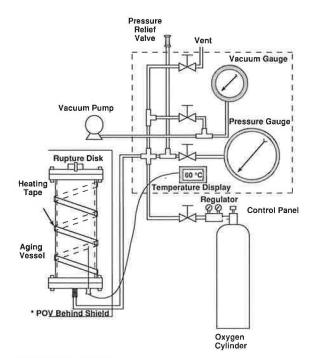


FIGURE 1 POV apparatus.

Asphalt samples for the POV oxidation test are prepared by depositing about 20 g of molten asphalt between one sheet of polyvinylchloride and one sheet of triacetate. The asphalt is then pressed between two glass plates until the asphalt between the plastic sheets formed a 6-in. circular disk to obtain a film thickness of about 1000 μm . If the asphalt cools down to the point that the pressing process becomes difficult, the whole setup is heated in an oven at a temperature of about 90°C to 100°C until the asphalt becomes soft again. A thinner film can be obtained by pressing the asphalt into a bigger disk if desired. The composite sheet is then cooled to room temperature and is cut into rectangular pieces of 1.5 in. by 2.5 in. The plastic sheets are removed from the asphalt film after

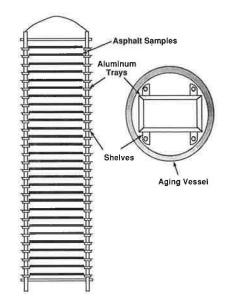


FIGURE 2 Sample holder for POV.

cooling the composite film in a freezer. The asphalt films are then deposited in 1.5-in. by 2.5-in. aluminum trays and are ready for the POV experiment.

Asphalt samples are placed in the stainless steel sample holder. The sample holder is placed inside the aging vessel and the flange is bolted down securely. With the venting valve, oxygen feed valve, and high-pressure gauge valve closed and the vacuum valve opened, a vacuum is applied to the aging vessel to remove the air inside. The vacuum valve is then closed. The vessel is slowly pressurized to 300 psia oxygen from an oxygen cylinder with the oxygen feed valve and highpressure gauge valve open. When the desired oxygen pressure is reached, the oxygen feed valve is closed. Power to the heating tape is then turned on to heat the pressure vessel to the desired aging temperature. During the heating process, excess pressure above the aging pressure can be vented off by opening the venting valve momentarily. When the desired period of oxidation time is attained, oxygen in the vessel is slowly vented to the atmosphere. Asphalt samples are taken from the vessel for further analysis at specified time intervals. The short time required to depressurize, remove the samples, and return to operating pressure and temperature is small compared with the total aging time.

Infrared analyses were made of all samples with a Nicolet 60 SXB Fourier transform infrared spectrometer (FT-IR) using an attenuated total reflectance (ATR) procedure described by Jemison et al. (24). The growth in the carbonyl band (from 1820 cm⁻¹ to 1650 cm⁻¹) was used as a measure of oxidation. Dynamic viscosities were measured at several temperatures using a dynamic mechanical spectrometer RMS-800 RDSII from Rheometrics Inc. Measurements were made with 0.984-in. (25-mm) parallel plates operating in a dynamic mode with a 0.0394-in. (1-mm) gap. Operation and data acquisition are monitored by an IBM PS-2 Model 30 computer with a RECAPIII computer analysis package, also from Rheometrics Inc. The complex dynamic viscosities were measured at low enough frequencies (at 0.1, 0.01, or 0.005 sec⁻¹, depending on the asphalt stiffness) to approach the lowfrequency limiting values designated as η_0^* .

RESULTS AND DISCUSSION

It was expected that the failure of aging procedures to represent road aging resulted primarily from using temperatures far above those experienced by the asphalts in road service, but the question remained how high a temperature could be used while still being representative of road aging. Experiments in the POV at 25°C showed extremely low aging rates, and it was obvious that some compromise must be made with higher temperatures.

To study this effect, five asphalts (AC-20, viscosity grade) were aged in the POV at 300 psia oxygen pressure at temperatures of 60°C, 71.1°C, and 82.2°C, and one asphalt, Ampet, was aged at 93.3°C. Samples were withdrawn periodically for viscosity measurements and infrared analysis. A typical infrared spectrum is shown in Figure 3. Figure 4 shows the limiting complex dynamic viscosity, η_0^* , measured at 60°C (140°F) versus aging time for Ampet at all four aging temperatures. The limiting complex dynamic viscosity, η_0^* , is defined as the limiting, essentially frequency-independent value

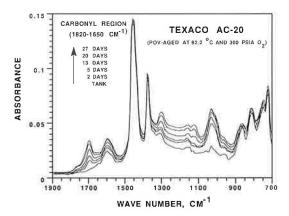


FIGURE 3 Typical FT-IR spectra using the ATR method for asphalts aged in the POV for various lengths of time.

that is reached at sufficiently low frequencies. The range of shear rate defining η_0^* is material dependent. The linear relation of log η_0^* versus aging time is characteristic of all asphalts studied, though with different slopes and intercepts. Figure 5 shows a plot for the same asphalt of carbonyl growth (the area increase above the neat asphalt between 1820 to $1650~\rm cm^{-1}$) versus time exhibiting a constant rate of carbonyl growth.

As expected, a cross plot of η_0^* versus carbonyl is linear (Figure 6). It can been seen that data from different aging temperatures combine to give a single line, strongly indicating that whereas the rate of oxidation changes with temperature, the mechanisms of oxidation do not. All five asphalts gave similar results as shown in Figure 7.

The linear relation between log viscosity and carbonyl formation was reported previously by Martin et al. (25) for asphalt extracted and recovered from road-aged asphalt. Jemison et al. (26) showed that it also applied to TFOT and RTFOT aging, but the function was not the same as that resulting from hot-mix plant aging. The POV data further confirm that this is a universal characteristic of asphalt oxidation for each asphalt as long as the aging mechanism is unchanged.

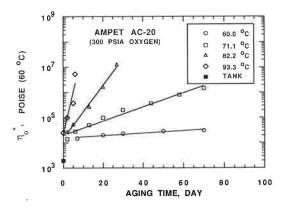


FIGURE 4 Dynamic viscosity hardening of an Ampet AC-20 asphalt with time at four temperatures.

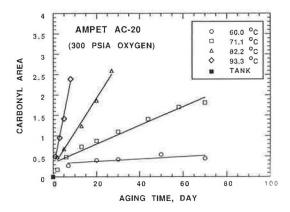


FIGURE 5 Growth of the infrared carbonyl band with time at four temperatures.

This relation also appears to hold for viscosity measurements over a wide range of temperatures. Figure 8 shows log viscosity at other temperatures versus carbonyl area. This of course means that changes in viscosity temperature susceptibility are independent of the aging temperature within this range, further confirmation of a constant aging mechanism.

This does not necessarily mean that the aging mechanism of the neat asphalt in the POV is the same as that on the road. The results of Jemison et al. (26) indicate that, at least in the hot-mix plant, the aggregate may play a role. Tables 1 and 2 give results obtained with two asphalts used in the Strategic Highway Research Program (SHRP). The asphalts were aged in a laboratory hot-mix process in the presence of aggregate (either SHRP RL or RB), compacted into cores, and then aged either in a POV or in a forced-draft oven (27). The aging times and temperatures are given in the table. The cores were then subjected to physical property tests (not the subject of this paper) and subsequently to asphalt extraction and recovery using a recently developed procedure designed to minimize solvent aging of the asphalt and to maximize asphalt extraction and subsequent solvent removal (23,28, 29). The recovered asphalt was compared with neat samples of the same asphalts after aging in the POV at 93.3°C and 300 psia oxygen (Figures 9 and 10). As can be seen, the differences, if any, are small, indicating little difference be-

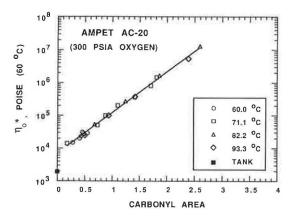


FIGURE 6 Hardening of the dynamic viscosity with growth of the carbonyl band at three temperatures.

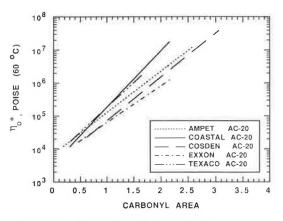


FIGURE 7 Comparison of the hardening susceptibilities of five asphalts.

tween POV aging of neat asphalt and core aging at temperatures below about 93.3°C.

The SHRP AAG-1 asphalt is remarkable in that when oxidized to the same extent as the other asphalts, the viscosity has not increased nearly as much. For example, when the carbonyl area of AAG-1 has increased by 2 units, then it has hardened to a viscosity of about 30 kPoise (Figure 9). On the other hand, for the same carbonyl growth, AAK-1 (Figure 10) has hardened to about 10,000 kPoise. There are very dramatic differences.

These results of Figures 4 through 10 show that hardening as the asphalt ages involves two important and independent factors. The first, the slope of the log viscosity versus carbonyl area plot, as in Figures 6 and 7, is a measure of the sensitivity of the asphalt viscosity to carbonyl formation. This is called the hardening susceptibility. The second, the slope of the carbonyl area versus time plot, as in Figure 5, is the rate of oxidation, which, as seen in Figure 5, is a strong function of temperature. An asphalt may oxidize relatively rapidly but not harden a great deal, compared with other asphalts, or it may harden considerably while oxidizing at a slower rate. Which situation holds can be an important consideration in assessing an asphalt's durability.

There is considerable evidence that the hardening susceptibility is primarily a function of compatibility. Asphalt AAG-

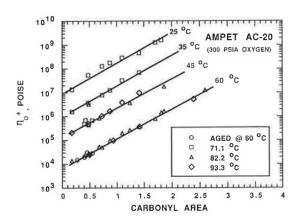


FIGURE 8 Dynamic viscosity versus carbonyl area for a single asphalt at four temperatures.

TABLE 1 LIMITING COMPLEX DYNAMIC VISCOSITY AND CARBONYL AREA FOR SHRP ASPHALT AAK-1 FOR DIFFERENT AGING CONDITIONS

- Sample	e ^a —	Pre-A	Aging —	-	- Aging			Properties -	
Aggregate	Air Voids (%)	Time (days)	Temp. (°C)	Time (days)	Temp. (°C)	Ovenb	η ₀ * (25°C,0.01s ⁻¹) (10 ⁶ poise)	η_0^* (60°C,0.1s ⁻¹) (10 ³ poise)	Carbonyl Area
Neat		0		0			1,987	3,992	0.000
RL	9.20	2	40	0		***	11.98	20.00	0.209
RL	9.10	2	40	7	107		270.2	1976.	1.350
RB	8.00	2	40	0			11.16	17.36	0.229
RB	8.90	2	40	7	107		183.4	868.0	1.318
RB	6.57	2	60	0			11.19	17.92	0.271
RB	6.42	2	60	7	107		150.9	560.9	1.071
RL	***	0		2	60	300	30.47	46.86	0.378
RL	***	0		7	25	300	15.45	34.43	0.291
RB		0		7	60	100	40.46	62.34	0.537
Neat	***	0	W-W1	0.5	92.2	300	60.00	88.00	0.568
Neat	***	0		1.0	92.2	300	170.0	300.0	0.933
Neat	***	0		2.0	92.2	300	***	40,000	2.736

^a Aggregates are SHRP RB or RL.

TABLE 2 LIMITING COMPLEX DYNAMIC VISCOSITY AND CARBONYL AREA FOR SHRP ASPHALT AAG-1 FOR DIFFERENT AGING CONDITIONS

- Sample	e ^a	— Pre-A	iging —		Aging			Properties -	
Aggregate	Air Voids (%)	Time (days)	Temp.	Time (days)	Temp.	Oven ^b	η_0^* (25°C,0.01s ⁻¹) (10 ⁶ poise)	η ₀ * (60°C,0.1s ⁻¹) (10 ³ poise)	Carbonyl Area
Neat	***	0		0			2.448	2.500	0.000
RL	7.91	2	60	0			13.80	8.334	0.490
RL	7.54	2	60	7	107		165.0	83.95	2.071
RB	***	0		2	60	300	22.43	12.00	1.165
RB	***	0		7	25	300	11.78	8.132	0.940
RL	***	0		7	60	100	28.00	13.77	1.278
Neat	***	0		0.5	92.2	300	35.00	16.00	1.369
Neat	***	0		1.0	92.2	300	54.00	19.00	1.682
Neat	***	0		3.0	92.2	300	130.0	45.00	2.527
Neat	***	0		5.0	92.2	300	164.0	64.00	2.83
Neat	***	0		8.0	92.2	300	250.0	106.0	3.228

^a Aggregates are SHRP RB or RL.

b The numbers indicate O2 pressure in the POV. Where no number is given, the aging was done in the FDO.

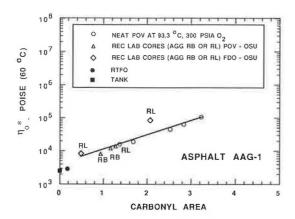


FIGURE 9 Hardening susceptibility for SHRP Asphalt AAG-1 as determined by both POV aging of neat asphalt and either POV or forced draft oven aging of laboratory-prepared cores. (L and B symbols refer to SHRP Aggregates RL and RB.)

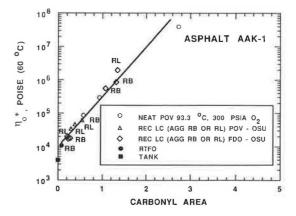


FIGURE 10 Hardening susceptibility for SHRP Asphalt AAK-1 as determined by both POV aging of neat asphalt and either POV or forced draft oven aging of laboratory-prepared cores. (L and B symbols refer to SHRP Aggregates RL and RB.)

 $^{^{\}rm b}$ The numbers indicate ${\rm O_2}$ pressure in the POV. Where no number is given, the aging was done in the FDO.

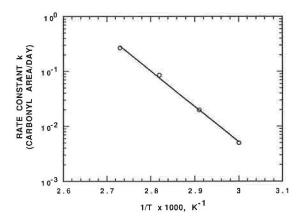


FIGURE 11 Arrhenius plot for the Ampet AC-20 asphalt.

1, for instance, is exceptionally low in both saturates and asphaltene content, which are mutually insoluble. Work by Stegeman et al. (30) has shown that the hardening ratio before and after RTFOT aging can be significantly reduced by separating asphalt into fractions on the basis of solubility and then formulating asphalts from the more mutually soluble fractions.

Chemical reaction rate constants generally vary as the exponent of the reciprocal absolute temperature. The rate of reaction is then given by this constant times a function of reactant concentrations. At the conditions in the POV, oxygen pressure is constant, and the actual oxygen uptake by the asphalt is so small compared with the number of available reaction sites that effectively the reactant concentrations are constant. If the rate of oxidation can be considered proportional to the growth rate in carbonyl are, then at temperature T

$$\frac{dA}{d\theta} \sim k \sim \exp(-E/RT)$$

where

A = carbonyl area,

k =the rate constant,

 $\theta = time$

E = a constant energy of activation unique to the reaction taking place, and

R = gas constant (1.987 cal/g mol K).

From this equation

$$\ln k \sim \frac{E}{R} \left(\frac{1}{T} \right)$$

and hence, one would expect a plot of $\ln k$ versus (1/T) to be linear.

Figure 11 shows this expected linear relationship for the Ampet asphalt and Figure 12 shows it for all five asphalts. The striking thing is not that the rates are different for each asphalt but that the slopes are different. This suggests that asphalts having different slopes are reacting with oxygen by different mechanisms. As can be seen, some asphalts that

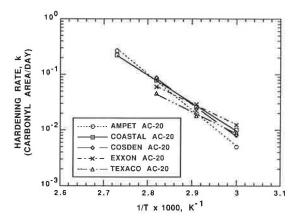


FIGURE 12 Comparison of the Arrhenius plots for the five asphalts studied. Note presence of crossover points where the relative aging rates of two asphalts interchange.

show higher rates of oxidation at elevated temperatures actually have lower oxidation rates at temperatures expected during life on the road. This reversal can carry over to physical properties as well, as shown in Figure 13, which shows the oxidative hardening of the Ampet and Texaco asphalts at 60°C and 80°C. At the lower temperature the Ampet asphalt hardens more slowly, whereas at the higher temperature the Texaco hardens more slowly. Thus no test run at a single elevated temperature is valid as a measure of expected road performance.

On the other hand, by running the POV at several temperatures, the resulting linear relations can be extrapolated to a realistic average road temperature or integrated over a typical road temperature history. To convert the rate data into a hardening (or other physical property) prediction, extrapolated rates, as could be obtained from Figure 12, can be combined with hardening susceptibilities (or analogous physical-

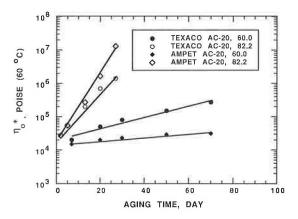


FIGURE 13 Comparison of the hardening of the Texaco and Ampet asphalts with time at two POV temperatures. At the lower temperature (140°F) the Ampet hardens more slowly, whereas at the higher temperature (180°F) the order is reversed and the Texaco hardens more slowly. The differences are significant, and relative aging of the two asphalts predicted on the basis of the higher temperature data would be grossly in error at highway conditions.

chemical property relation), such as those in Figure 7. In this way the relative expected hardening rates could be calculated from the expected road temperature profile.

CONCLUSIONS

POV aging (300 psia O_2) of asphalt films (1000 μ m thick) and subsequently measured FT-IR (ATR) spectra and complex dynamic viscosities have led to the following conclusions:

- 1. The degree of oxidation, as measured by the increase in the carbonyl peak area relative to the unaged neat asphalt, increases linearly with oxidation time at a given temperature, over a range from 60°C to 82.2°C (at least). The slope of this relation is indicative of the chemical reaction rate.
- 2. The degree of hardening due to oxidation increases essentially linearly with time and, hence, is linearly related to the carbonyl peak area. The hardening susceptibility is a key measure of the effect of oxidative aging on a physical property. This effect is probably related to the compatibility of the asphalt.
- 3. Different asphalts exhibit different reaction rates at the same temperature and, just as important, exhibit different activation energies (different slope of $\ln k$ versus 1/T). As a result, one asphalt may age faster than another at one temperature, whereas it may age slower at a different temperature. This crossover in aging rate is of profound significance in designing an asphalt road aging test. Such a test must be conducted at more than one temperature to accurately predict pavement aging.
- 4. Asphalts aged in contact with two different aggregates suggest that the reaction mechanisms are not significantly changed by the aggregate up to even 107°C.

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REFERENCES

- B. A. Vallerga, C. L. Monismith, and K. Granthem. A Study of Some Factors Influencing the Weathering of Paving Asphalts. *Proc. AAPT*, Vol. 26, 1957.
- D. F. Fink. Research Studies and Procedures. Proc. AAPT, Vol. 27, 1958.
- R. N. Traxler. Changes in Asphalt Cements During Preparation, Laying, and Service of Bituminous Pavements. *Proc. AAPT*, Vol. 36, 1967.
- R. L. Griffin, R. K. Miles, and C. J. Penther. Microfilm Durability Test for Asphalt. Proc. AAPT, Vol. 24, 1955.

- J. J. Heithaus, and R. W. Johnson. A Microviscometer Study of Road Asphalt Hardening in the Field and Laboratory. *Proc.* AAPT, Vol. 27, 1958.
- W. C. Simpson, R. L. Griffin, and T. K. Miles. Correlation of the Microfilm Durability Test with Field Hardening Observed in the Zaca-Wigmore Experimental Project. ASTM STP, 277, 1959.
- R. J. Schmidt and L. E. Santucci. The Effect of Asphalt Properties on the Fatigue and Cracking of Asphalt Concrete on the Zaca-Wigmore Test Project. *Proc. AAPT*, Vol. 38, 1969.
- R. J. Schmidt, Laboratory Measurement of the Durability of Paving Asphalts. ASTM STP, 532, 1973.
- R. J. Schmidt. The Rolling Thin-Film Circulating Oven—An Improved Rolling Thin-Film Oven Test. ASTM STP, 532, 1973.
- 10. G. R. Kemp. Asphalt Durability Tests and Their Relationship to Field Hardening. *ASTM STP*, 532, 1973.
- J. C. Petersen. A Thin Film Accelerated Aging Test for Evaluating Asphalt Oxidative Aging. AAPT preprint volume, Session II, Feb. 1989.
- 12. P. W. Jennings. Uses of High Pressure Liquid Chromatography to Determine the Effects of Various Additives and Fillers on Characteristics of Asphalt. Report FHWA/MT-82/001. Montana Department of Highways, 1982.
- 13. J. C. Petersen, F. A. Barbour, and S. M. Dorrence. Catalysis of Asphalt Oxidation by Mineral Aggregate Surfaces and Asphalt Components. *Proc. AAPT*, Vol. 43, 1974.
- J. C. Petersen, E. K. Ensley, and F. A. Barbour. Molecular Interactions of Asphalt in the Asphalt-Aggregate Interface Region. In *Transportation Research Record* 515, TRB, National Research Council, Washington, D.C., 1974.
- H. Plancher, S. M. Dorrence, and J. C. Petersen. Identification of Chemical Types in Asphalts Strongly Adsorbed at the Asphalt-Aggregate Interface and Their Relative Displacement by Water. *Proc. AAPT*, Vol. 46, 1977.
- E. K. Ensley, J. C. Petersen, and R. E. Robertson. Asphalt-Aggregate Bonding Energy Measurements by Microcalorimetric Methods. *Thermochimica Acta*, Vol. 77, 1984.
- A. F. Barbour, R. V. Barbour, and J. C. Petersen. A Study of Asphalt-Aggregate Interactions Using Inverse Gas-Liquid Chromatography. *Journal of Applied Chemical Biotechnology*, Vol. 24, 1974.
- 18. D. Y. Lee. Development of a Laboratory Durability Test for Asphalts. In *Highway Research Record 231*, HRB, National Research Council, Washington, D.C., 1968.
- D. Lee. Asphalt Durability Test Correlations in Iowa. In Highway Research Record 468, HRB, National Research Council, Washington, D.C., 1973.
- I. L. Jamieson and M. M. Hattingh. The Correlation of Chemical and Physical Properties of Bitumens with Their Road Performance. *Proc.*, Australian Road Research Conference, Vol. 5, 1970, pp. 293–324.
- 21. A. C. Edler, M. M. Hattingh, V. P. Servas, and C. P. Marais. Use of Aging Tests to Determine the Efficacy of Hydrated Lime Additions in Retarding Oxidative Hardening. *Proc. AAPT*, Vol. 54, 1985, pp. 118–139.
- O. Kim, C. A. Bell, J. E. Wilson, and G. Boyle. Development of Laboratory Oxidative Aging Procedures for Asphalt Cements and Asphalt Mixtures. In *Transportation Research Record 1115*, TRB, National Research Council, Washington, D.C., 1987, pp. 101–112.
- B. L. Burr, R. R. Davison, C. J. Glover, and J. A. Bullin. Solvent Removal from Asphalt. In *Transportation Research Record 1269*, TRB, National Research Council, Washington, D.C., 1990, pp. 1–8.
- H. B. Jemison, B. L. Burr, R. R. Davison, J. A. Bullin, and C. J. Glover. Application and Use of the ATR, FT-IR Method to Asphalt Aging Studies. ACS Preprints, Division of Petroleum Chemistry, Vol. 35, No. 3, 1990, pp. 490–495.
 K. L. Martin, R. R. Davison, C. J. Glover, and J. A. Bullin.
- K. L. Martin, R. R. Davison, C. J. Glover, and J. A. Bullin. Asphalt Aging in Texas Roads and Test Sections. In *Transportation Research Record* 1269, TRB, National Research Council, Washington, D.C., 1990, pp. 9–19.
- H. B. Jemison, R. R. Davison, C. J. Glover, and J. A. Bullin. Evaluation of Standard Oven Tests for Hot-Mix Plant Aging. In Transportation Research Record, TRB, National Research Council, Washington, D.C. (forthcoming).

27. C. A. Bell, Y. AbWahab, and M. E. Cristi. Investigation of Laboratory Aging Procedures for Asphalt-Aggregate Mixtures. In Transportation Research Record, TRB, National Research Council, Washington, D.C. (forthcoming).

28. B. L. Burr, R. R. Davison, H. B. Jemison, C. J. Glover, and J. A. Bullin. Asphalt Hardening in Extraction Solvents. In Transportation Research Record, TRB, National Research Council,

Washington, D.C. (forthcoming).

29. C. A. Cipione, R. R. Davison, B. L. Burr, C. J. Glover, and J. A. Bullin. Evaluation of Solvents for the Extraction of Residual Asphalt from Aggregates. In Transportation Research Record, TRB, National Research Council, Washington, D.C. (forthcoming).

30. J. R. Stegeman, A. L. Kyle, B. L. Burr, H. B. Jemison, R. R. Davison, C. J. Glover, and J. A. Bullin. Compositional and Physical Properties of Asphalt Fractions Obtained by Supercritical Extraction. ACS Preprints, Division of Petroleum Chemistry, Vol. 35, No. 3, 1990, pp. 431-439.

The contents of the report reflect the views of the authors, who are responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official views or policies of the Federal Highway Administration or the Texas Department of Highways and Public Transportation. This report does not constitute a standard, specification, or regulation.

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Evaluation of Aging Characteristics of Asphalts by Using TFOT and RTFOT at Different Temperature Levels

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The aging characteristics of 20 asphalts were investigated by using the thin film oven test (TFOT) and rolling thin film oven test (RTFOT) at three temperature levels. Infrared absorption spectroscopy and the Schweyer rheometer test as well as penetration and absolute viscosity tests were used to evaluate the characteristics of the asphalts before and after the TFOT and RTFOT. Data from the field were also used to compare with the laboratory results. On the basis of percent penetration retained and absolute viscosity ratio, the RTFOT was found to be a more severe aging process than the TFOT for oven temperatures of 285°F and 325°F. However, the two processes were not significantly different at an oven temperature of 365°F. On the basis of carbonyl ratio, a ratio of infrared absorbance at 1700 cm⁻¹ and 1600 cm⁻¹ used to express the level of oxidation in an asphalt binder, the effects of TFOT and RTFOT are not significantly different at oven temperatures of 285°F and 325°F. However, at 365°F, the TFOT is a more severe aging process than the RTFOT from the standpoint of carbonyl ratio. On the basis of low-temperature constant power viscosity, the effects of TFOT and RTFOT are not significantly different at any of the three levels of temperature. As a rough estimate, the TFOT or RTFOT procedure performed at 365°F, 3 months of natural weathering of compacted Marshall specimens, and 6 to 9 months of aging in a pavement would result in approximately the same hardening effects on a typical paving grade asphalt used in Florida. From the results of this study, it appears feasible to use TFOT or RTFOT at higher temperature to simulate the aging process on the asphalt binder in asphalt paving mixtures in service, as well as that of the hot-mixing process.

It has been recognized that the properties of asphalt binders play a major role in the performance of asphalt concrete pavements. It is important that the selected asphalt for paving not only have desirable properties at the time of placement but also have good long-term performance. Current asphalt binder specifications are not sufficient to ensure good long-term performance. The thin film oven test (TFOT) or rolling thin film oven test (RTFOT) at 325°F, adopted by almost all highway agencies, can only estimate the property changes of asphalt binders during the hot mixing process and do not provide adequate information on changes in properties during service in the pavement. It is necessary to have tests that can predict the properties of the asphalt binder during service in the pavement.

The results of previous studies on aging characteristics of asphalts and asphalt mixtures indicate that it is possible to simulate the effects of aging during service as well as that of the hot mixing process by using the TFOT or the RTFOT at higher temperatures (1,2). The potential advantages of using the TFOT or the RTFOT at higher temperatures as compared with other long-term aging tests, such as the California tilt oven and the pressure oxidation vessel tests, are that existing standard equipment could be conveniently used and that the test would require much less time to complete. The results of a study on the aging characteristics of asphalts by using the TFOT and RTFOT at three temperature levels are presented. Infrared absorption spectroscopy and Schweyer rheometer tests as well as penetration and absolute viscosity tests were used to evaluate the aging characteristics of the asphalts before and after the TFOT and RTFOT. Data from the field were also used to compare with the laboratory results. Relationships between laboratory aging and field aging were established.

TESTING PROGRAM

Testing Program on Asphalt Binders

Twenty representative asphalts commonly used in Florida and its neighboring states were selected for a laboratory testing program to evaluate their aging characteristics. For each of the asphalts, the following tests were performed on the asphalts and the asphalt residues after the TFOT and RTFOT at 285°F, 325°F, and 365°F: penetration at 77°F, absolute viscosity at 140°F, infrared spectroscopy to determine the carbonyl ratio, and Schweyer rheometer at 41°F and 77°F.

Statistical Model for Asphalt Binder Tests

The test results from asphalt binder tests were analyzed as results of a factorial experiment composed of 20 types of asphalts (ASPHALT), 3 levels of temperature (TEMP), and 2 types of test (OVEN). The study is concerned only with these 20 asphalts, 3 temperatures, and 2 tests. Therefore, ASPHALT, TEMP, and OVEN are regarded as fixed effects. The following linear model was assumed for any single measurement in the experiment:

$$Y_{ijk} = m + A_i + T_j + O_k + (AT)_{ij} + (AO)_{ik} + (TO)_{jk} + \varepsilon_{ijk}$$

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where

 Y_{ijk} = the response of the *i*th ASPHALT, *j*th TEMP, and *k*th OVEN;

m = the overall mean;

 A_i = the main effect of ASPHALT i;

 T_j = the main effect of TEMP j;

 O_k = the main effect of OVEN k;

 $(AT)_{ij}$ = the interaction of ASPHALT $i \times \text{TEMP } j$;

 $(AO)_{ik}$ = the interaction of ASPHALT $i \times \text{OVEN } k$;

 $(TO)_{ik}$ = the interaction of TEMP $j \times \text{OVEN } k$; and

 ε_{ijk} = the experimental error.

The SAS/STAT computer software was used for the statistical analyses.

TEST PROCEDURES

Infrared Absorption Spectroscopy

Infrared spectroscopic technique was used to measure changes in molecular structures of the asphalts due to aging in terms of changes in the amount of certain functional groups in them. The infrared absorption spectrum between 1600 cm⁻¹ and 1900 cm⁻¹ is of particular interest since it contains the absorption bands for the functional groups of carboxylic acids, ketones, and anhydrides (3,4). Ketones and anhydrides are formed in asphalt on oxidative aging, whereas carboxylic acids occur naturally in asphalt but increase in amount on oxidative aging.

The IR spectroscopy tests were run using a benchtop Perkin-Elmer Model 1600 Fourier transform infrared spectrophotometer. A 5 percent (weight/volume) solution in HPLC grade tetrahydrofuran (THF) was used in the tests. A sealed cell with 1-mm path length and sodium chloride windows was used. The background spectrum for the THF was first generated by scanning the pure solvent and stored in the computer. Then the spectrum for the solution was generated. Using the computer software of the system, the background spectrum was ratioed out, yielding the spectrum of the sample.

Schweyer Rheometer Test

A Cannon Schweyer constant stress rheometer was used in this research. A comprehensive review of the theoretical background for the Schweyer constant stress rheometer and the application of rheological concepts proposed by Schweyer has been presented by Tia and Ruth (5). The rheometer consists of a gas-operated pneumatic cylinder that applied a specific force to the plunger in the sample tube. An LVDT measured the movement of the plunger, and the output voltage was digitized and acquired by a data acquisition and analysis system, which was operated on an IBM 9000 minicomputer.

In the rheometer test, a sample tube is filled to approximately 2 to 6 mm from the top. The plunger is inserted into the sample tube, and the entire assembly is placed into an insulated aluminum block, which has been cooled to the desired testing temperature. Once the temperature has stabilized, the gas pressure is preset and the gas cylinder activated to apply force to the plunger and asphalt sample. The defor-

mation versus time plot is recorded for each stress level until the response becomes linear. Tests are usually conducted at a minimum of five stress levels. The shear stress and shear rates obtained at the various stress levels are used to compute the shear susceptibility and the constant power viscosity at 100 w/m³ of the asphalt.

TEST RESULTS

Penetration Test on Asphalt Binders

The results of the penetration tests are given in Tables 1 and 2. To increase the data base, data from a previous research study (I) were included in the analyses. Table 3 gives the data from the previous study.

Since the tested asphalts had a variety of penetration values in the original state, the percent penetration retained is more appropriate for comparison. Results of ANOVA on the percent penetration retained are given in Table 4. From Table 4, it is seen that all three main effects and the interaction effects except ASPHALT*TEMP are significant. The significance of the interaction between OVEN and TEMP means

TABLE 1 TFOT AND RTFOT EFFECTS ON PENETRATION

				RATION AT TEMPERATU	
ASPHALT	TEST	ORIGINAL	285	325	365
MA30A	TFOT RTFOT	65		*	(4)
MA30B	TFOT RTFOT	62	-	-	-
MA30C	TFOT RTFOT	58	•	8	-
MA30D	TFOT RTFOT	63		5	(7.1
MA30E	TFOT RTFOT	60	500	=	(4)
MA20T	TFOT RTFOT	72	60 56	52 51	42 42
MA20C	TFOT RTFOT	70	53 44	45 41	35 35
MA20V	TFOT RTFOT	84	69 64	59 52	46 45
TL20	TFOT RTFOT	72	52 42	40	32
TN20	TFOT	56	43 41	34 35 34	30 28 29
BJ30A	RTFOT TFOT	61	47	39	32
BJ20	RTFOT TFOT	70	41 53 46	34 43 37	28 33 34
CJ30A	RTFOT TFOT	66	54	45	37
MT30	RTFOT TFOT	53	45 44	39 37	33 31
CT30	RTFOT TFOT	57	37 -	33	29
TB30A	RTFOT TFOT	74	42 58	38 51	32 42
SA20	RTFOT TFOT	65	56 51	45 41	42 34
AS30	RTFOT TFOT	65	41 52	37 43	33 35
BF30	RTFOT TFOT	55	44 41	39 36	32 28
MA30	RTFOT TFOT RTFOT	61	36 49 47	31 42 38	28 34 33

TABLE 2 TFOT AND RTFOT EFFECTS ON PERCENT PENETRATION RETAINED

			RATION RET	
ASPHALT	TEST	285	325	365
MA30A	TFOT RTFOT			
MA30B	TFOT RTFOT	8	-	*
MA30C	TFOT RTFOT	¥	-	-
MA30D	TFOT RTFOT	ž	•	
MA30E	TFOT RTFOT			7
MA20T	TFOT RTFOT	83.3 77.8	72.2 70.8	58.3 58.3
MA20C	TFOT RTFOT	75.7 62.9	64.3 58.6	50.0 50.0
MA20V	TFOT RTFOT	82.1 76.2	70.2 61.9	54.8 53.6
TL20	TFOT RTFOT	72.2 58.3	55.6 47.2	44.4
TN20	TFOT RTFOT	76.8 73.2	62.5 60.7	41.7 50.0 51.8
BJ30A	TFOT RTFOT	77.0 67.2	63.9 55.7	52.5 45.9
BJ20	TFOT RTFOT	75.7	61.4 52.9	47.1
CJ30A	TFOT RTFOT	65.7 81.8	68.2	48.6 56.1
MT30	TFOT	68.2 83.0	59.1 69.8	50.0 58.5
CT30	RTFOT TFOT	69.8	62.3	54.7
TB30A	RTFOT TFOT	73.7 78.4	66.7 68.9	56.1 56.8
SA20	RTFOT TFOT	75.7 78.5	60.8 63.1	56.8 52.3
AS30	RTFOT TFOT	63.1 80.0	56.9 66.2	50.8 53.8
BF30	RTFOT TFOT	67.7 74.5	60.0 65.5	49.2 50.9
MA30	RTFOT TFOT RTFOT	65.5 80.3 77.0	56.4 68.9 62.3	50.9 55.7 54.1

TABLE 3 PENETRATION RESULTS FROM PREVIOUS RESEARCH

		<pre>% Penetration Retained Oven Temperature(°F)</pre>		ed
Asphalt	Test	285	325	365
VB	TFOT	76.5	66.2	51.5
AC-30	RTFOT	69.1	60.3	48.5
VE	TFOT	79.1	64.2	50.7
AC-30	RTFOT	68.7	56.7	50.7
VA	TFOT	75.3	61.6	46.6
AC-30	RTFOT	68.5	56.2	46.6
VD	TFOT	75.8	66.1	56.5
AC-30	RTFOT	74.2	61.3	56.5
VC	TFOT	73.8	61.5	49.2
AC-30	RTFOT	66.2	56.9	47.7
VJ	TFOT	74.7	62.7	58.7
AC-20	RTFOT	70.7	57.3	52.0
VJ	TFOT	82.4	71.6	56.9
AC-20 Mod	.RTFOT	74.5	58.8	55.9
VG	TFOT	73.9	58.0	44.3
85-100	RTFOT	68.2	54.5	48.9
VF	TFOT	85.4	76.8	63.4
85-100	RTFOT	84.1	78.0	69.5
VI	TFOT	88.9	81.5	74.1
25-35	RTFOT	88.9	77.8	66.7
VH	TFOT	71.1	57.9	40.8
85-100	RTFOT	71.1	55.3	42.1

TABLE 4 RESULTS OF ANOVA ON PENETRATION RETAINED

Source	DF	Mean Square	F Value	Pr > F
ASPHALT	25	193.30075	46.53	0.0001
TEMP	2	6056.51418	1457.87	0.0001
OVEN	1	847.64310	204.04	0.0001
ASPHALT*TEMP	50	5.60310	1.35	0.1500
ASPHALT*OVEN	24	14.04473	3.38	0.0002
TEMP*OVEN	2	128.80673	31.01	0.0001

R-Square: 0.989

that the difference between the TFOT and the RTFOT also depends on oven temperature. From Tables 2 and 3, it is seen that the RTFOT is a more severe aging process than the TFOT for oven temperatures of 285°F and 325°F. However, the two processes are not significantly different from each other at an oven temperature of 365°F.

TABLE 5 TFOT AND RTFOT EFFECTS ON ABSOLUTE VISCOSITY

			VISCOSITY AT 140°F (Poises) OVEN TEMPERATURE (°F)		
ASPHALT	TEST	ORIGINAL	285	325	365
MA30A	TFOT	3245	5068	6924	11776
	RTFOT		5789	8712	12135
MA30B	TFOT	3258	5421	8968	13237
	RTFOT		6614	9622	16243
MA30C	TFOT	3549	5229	7991	16384
0000000	RTFOT	1212121111	6762	10649	17149
MA30D	TFOT	3290	5883	9474	19796
	RTFOT		7688	13515	23200
MA30E	TFOT	3053	4941	7693	14110
	RTFOT		5877	9394	14435
MA20T	TFOT	2606	3631	4996	7496
20 of 50 10 10 10 10 10 10 10 10 10 10 10 10 10	RTFOT		4082	5571	7714
MA20C	TFOT	2861	4731	7148	13420
	RTFOT		5995	8699	13597
MA20V	TFOT	2563	4113	7223	13105
	RTFOT		4870	7302	13673
TL20	TFOT	1863	3676	6961	15884
	RTFOT		5184	9211	16838
TN20	TFOT	2028	3221	4586	7516
	RTFOT		3497	4959	7559
BJ30A	TFOT	3335	5043	7848	15501
	RTFOT	1/22/12/2004/000	6782	11893	24079
BJ20	TFOT	1976	3367	5290	11727
	RTFOT		4691	7291	10783
CJ30A	TFOT	2965	4856	6836	11648
	RTFOT		6498	8535	13681
MT30	TFOT	3410	5740	8472	16429
	RTFOT		8148	11965	17832
CT30	TFOT	2890	4973	6996	10599
	RTFOT		6423	9228	12285
TB30A	TFOT	3175	5842	9594	19253
	RTFOT		7174	12089	17734
SA20	TFOT	2057	3583	5618	9878
	RTFOT		4804	6964	9786
AS30	TFOT	3421	5247	8192	13493
	RTFOT		6384	9414	13504
BF30	TFOT	4130	5482	7924	12267
	RTFOT		6793	9998	12267
MA30	TFOT	3202	4756	6958	10583
	RTFOT		5923	8511	11492

Absolute Viscosity Test on Asphalt Binders

The results of the absolute viscosity tests are given in Tables 5 and 6. To have a larger data base, data from previous research (1) were included in the analysis. Data from the previous research are given in Table 7.

Since the original viscosities of the asphalts are quite different from each other, it is more appropriate to use viscosity ratios than absolute viscosities in the analysis. Results of ANOVA on the absolute viscosity ratios are summarized in Table 8, which indicates that all three main effects and the interaction ASPHALT*TEMP are significant.

Comparison of the two types of oven was done at three different temperatures by means of Duncan's multiple-range test at a level of significance of 0.05. The results of the Duncan's test are given in Table 9, which indicates that the RTFOT is a more severe aging process than the TFOT for oven temperatures of 285°F and 325°F. However, for an oven temperature of 365°F, the two processes are not significantly different from one another.

TABLE 6 TFOT AND RTFOT EFFECT ON ABSOLUTE VISCOSITY RATIO

		OVEN	TEMPERATU	200 1 20
ASPHALT	TEST	285	325	365
MA30A	TFOT	1.56	2.13	3.63
мазов	RTFOT TFOT	1.78 1.66	2.68	3.74 4.06
MAJOD	RTFOT	2.03	2.95	4.99
MA30C	TFOT	1.47	2.25	4.62
	RTFOT	1.91	3.00	4.83
MA30D	TFOT	1.79	2.88	6.02
MA30E	RTFOT TFOT	2.34 1.62	4.11 2.52	7.05 4.62
MAJUE	RTFOT	1.92	3.08	4.73
MA20T	TFOT	1.39	1.92	2.88
	RTFOT	1.57	2.14	2.96
MA20C	TFOT	1.65	2.50	4.69
MA20V	RTFOT TFOT	2.10 1.60	3.04 2.82	4.75 5.11
MAZUV	RTFOT	1.90	2.85	5.33
TL20	TFOT	1.97	3.74	8.53
1220	RTFOT	2.78	4.94	9.04
TN20	TFOT	1.59	2.26	3.71
	RTFOT	1.72	2.45	3.73
BJ30A	TFOT RTFOT	1.51 2.03	2.35 3.57	4.65
BJ20	TFOT	1.70	2.68	5.93
DULU	RTFOT	2.37	3.69	5.46
CJ30A	TFOT	1.64	2.31	3.93
	RTFOT	2.19	2.88	4.61
MT30	TFOT	1.68	2.48	4.82
CT30	RTFOT TFOT	2.39 1.72	3.51 2.42	5.23
C130	RTFOT	2.22	3.19	4.25
TB30A	TFOT	1.84	3.02	6.06
	RTFOT	2.26	3.81	5.59
SA20	TFOT	1.74	2.73	4.80
****	RTFOT	2.34	3.39	4.76
AS30	TFOT RTFOT	1.53 1.87	2.39 2.75	3.94
BF30	TFOT	1.87	1.92	2.97
D, 30	RTFOT	1.64	2.42	2.97
MA30	TFOT	1.49	2.17	3.31
	RTFOT	1.85	2.66	3.59

TABLE 7 ABSOLUTE VISCOSITY RESULTS FROM PREVIOUS RESEARCH

			Absolute Viscosity Ratio Oven Temperature (°F)		
Asphalt	Test	285	325	365	
VB	TFOT	1.98	3.14	7.33	
AC-30	RTFOT	2.49	4.64	10.60	
VE	TFOT	2.03	3.23	8.42	
AC-30	RTFOT	2.41	4.11	7.86	
VA	TFOT	1.77	2.96	7.15	
AC-30	RTFOT	2.11	3.17	5.47	
VD	TFOT	1.72	2.57	4.57	
AC-30	RTFOT	1.96	2.86	4.76	
VC	TFOT	1.90	3.19	7.98	
AC-30	RTFOT	2.54	3.94	8.73	
VJ	TFOT	1.85	2.93	6.02	
AC-20	RTFOT	2.07	3.93	4.78	
٧J	TFOT	1.48	1.91	3.94	
AC-20 Mod	.RTFOT	1.97	3.53	4.74	
VG	TFOT	1.51	2.19	3.80	
85-100	RTFOT	1.78	2.35	3.06	
VF	TFOT	1.37	1.86	3.07	
85-100	RTFOT	1.48	1.77	2.47	
VI	TFOT	1.47	2.14	3.93	
25-35	RTFOT	1.60	2.72	6.49	
VH	TFOT	2.36	4.70	21.40	
85-100	RTFOT	2.38	5.01	13.95	

TABLE 8 RESULTS OF ANOVA ON ABSOLUTE VISCOSITY RATIO

Source	DF	Mean Square	F Value	P r > F
ASPHALT	30	8.7368643	19.12	0.0001
OVEN	1	6.3215274	13.84	0.0004
TEMP	2	216.2998651	473.40	0.0001
ASPHALT*TEMP	60	3.9948567	8.74	0.0001
ASPHALT*OVEN	30	0.6934641	1.52	0.0848
OVEN*TEMP	2	1.3043145	2.85	0.0654

R-Square: 0.972

TABLE 9 COMPARISON OF TFOT AND RTFOT AT THREE TEMPERATURES ON THE BASIS OF VISCOSITY RATIO

TEMP.	Duncan Grouping	Mean	N	OVEN
285°F	А	2.0645	31	RTFOT
	В	1.6748	31	TFOT
325°F	Α	3.2626	31	RTFOT
	В	2.6148	31	TFOT
365°F	A	5.538	31	RTFOT
	Α	5.470	31	TFOT

^{*} Means with the same letter are not significantly different.

TABLE 10 RESULTS OF INFRARED SPECTROSCOPY

		CARBONY	/L RATIO	
A C DUAL T	ORIGINAL	TF0T 285°F	TF0T 325°F	TF0T 365°F
ASPHAL I	UKIGINAL	285 F	323 F	300 F
MA30A	0.3543	0.3480	0.3668	0.4066
MA30B	0.2548	0.2984	0.3231	0.3924
MA30C	0.2551	0.2530	0.2914	0.3561
MA30D	0.2737	0.3126	0.3479	0.4010
MA30E	0.3081	0.3619	0.3675	0.4070
MA20T	0.3775	0.3764	0.4320	0.4586
MA20C	0.3540	0.3861	0.4338	0.4983
MA20V	0.3512	0.3742	0.4075	0.4349
TL20	0.3250	0.3644	0.3924	0.4305
TN20	0.2444	0.2627	0.2958	0.3381
BJ30A	0.3370	0.2933	0.3256	0.3674
BJ20	0.3171	0.3321	0.3660	0.4048
CJ30A	0.3050	0.3468	0.3650	0.3910
MT30	0.3282	0.3126	0.3421	0.3869
CT30 TB30A	0.3584 0.4314	0.3975 0.4529	0.4469 0.4971	0.4622
SA20	0.4314	0.4529	0.4971	0.4326
AS30	0.3178	0.3620	0.3900	0.4324
BF30	0.3673	0.3741	0.4114	0.4224
MA30	0.3073	0.3404	0.3678	0.4045
VB	0.2840	0.3410	0.3680	0.3950
VE	0.3210	0.3520	0.3670	0.4340
٧A	0.2850	0.3310	0.3680	0.4240
VD	0.3130	0.3470	0.3820	0.4100
VC	0.3150	0.3600	0.3860	0.4450
٧J	0.2820	0.3190	0.3490	0.4010
VJM	0.2820	0.2960	0.3150	0.3600
VG	0.2540	0.2800	0.3100	0.3670
VF	0.7480	0.8050	0.8210	0.8850
VI	0.3590	0.3740	0.3870	0.4690
VH	0.3660	0.4030	0.4140	0.4700

ASPHALT	ORIGINAL	RTFOT 285°F	RTFOT 325°F	RTFOT 365°F
MA30A MA30B MA30C MA30C MA30C MA20T MA20C MA20V TL20 BJ30A BJ20 CJ30A MT30 CT30 TB30A SA20 AS30 BF30 MA30 VB VE VD VC VJ VJ VJ VJ	0.3543 0.2548 0.2551 0.2737 0.3081 0.3775 0.3540 0.3512 0.3250 0.2444 0.3370 0.3171 0.3050 0.3282 0.3584 0.4314 0.3178 0.3971 0.3673 0.3277 0.2840 0.2850 0.2850 0.2850 0.2850 0.2820 0.2820	285°F 0.3545 0.2812 0.2030 0.3102 0.3207 0.3836 0.3995 0.3951 0.3753 0.2479 0.3033 0.3421 0.3653 0.3286 0.3807 0.4496 0.3557 0.3491 0.3240 0.3230 0.3240 0.3230 0.3180 0.3370 0.3240 0.2930 0.2790	325°F 0.3680 0.3206 0.2487 0.3443 0.3568 0.4236 0.4467 0.4121 0.3920 0.2726 0.3359 0.3708 0.3866 0.3890 0.4512 0.3791 0.4366 0.3607 0.3660 0.3490 0.3620 0.3620 0.3640 0.3540 0.3540 0.3260	365°F 0.3711 0.3683 0.2868 0.3684 0.3867 0.4516 0.4736 0.4360 0.3761 0.3931 0.4073 0.3577 0.4288 0.4774 0.4044 0.4643 0.4601 0.3870 0.3920 0.4120 0.4120 0.4260 0.3760 0.4110 0.3630 0.3580 0.3390
VF VI VH	0.7480 0.3590 0.3660	0.7930 0.3760 0.3930	0.8380 0.4090 0.4180	0.8580 0.4740 0.4580

Infrared Spectroscopy on Asphalt Binders

Study of the infrared absorption spectra indicates that there is a definite increase in the infrared absorbance in the 1700 cm⁻¹ region as the level of oxidation increases. This is due to increasing concentration of ketones and anhydrides, which have infrared absorbances at about 1700 cm⁻¹, and carboxylic acids, which have infrared absorbance at about 1730 cm⁻¹. Ketones and anhydrides are formed on oxidative aging, and carboxylic acids occur naturally in the asphalt but increase on oxidation aging. Since these three functional groups are the most significant chemical functionalities that can be related to oxidative aging, examination of the spectra in this study was done with particular reference to them.

The spectra also indicate that the band centering at about 1600 cm⁻¹, which results primarily from aromatic carbon-carbon double bonds, can be assumed to be fairly constant, because the group is present in highly condensed stable molecules (6). Therefore, the carbonyl ratio, which is a ratio of absorbance at 1700 cm⁻¹ and 1600 cm⁻¹, can be used to express the level of oxidation. All absorption measurements were with reference to the absorption at 1900 cm⁻¹.

The results of the infrared spectroscopy are given in Table 10. Results of ANOVA on the data are summarized in Table 11, which indicates that all three main effects and interaction effects are significant.

Comparison of the effects of OVEN was done at different temperatures by means of Duncan's multiple-range test at a level of significance of 0.05. The results are summarized in Table 12, which indicates that the effects of TFOT and RTFOT are not significantly different at oven temperatures of 285°F and 325°F. But at 365°F, the TFOT is a more severe aging process than the RTFOT from the standpoint of carbonyl ratio.

Schweyer Rheometer Test on Asphalt Binders

Table 13 gives the results of the Schweyer rheometer test on the original asphalts. Nine of the 20 asphalts were used in the Schweyer rheometer test. The results are given in Table 14. Results of ANOVA on the rheometer test are summarized in Table 15, which indicates that the OVEN effect is not significant.

When the constant power viscosity versus temperature relationships for the original and aged asphalts were plotted, it was observed that asphalts of the same grade (viscosity) at high temperature (60°C) could have very different properties at lower temperature. In addition, the asphalt constant power viscosity-temperature relationship is shifted parallel to the original relationship after TFOT. The degree of parallel shifting depends on the severity of the aging process; it shifts more for a higher oven temperature than for a lower oven temperature.

ANALYSIS AND DISCUSSION

The TFOT and the RTFOT tests were adopted by AASHTO, ASTM, and almost all state highway departments and other

TABLE 11 RESULTS OF ANOVA ON INFRARED SPECTROSCOPY RESULTS

SOURCE	DF	ANOVA SS	F VALUE	PR > F
ASPHALT	30	1.57990015	646.54	0.0
TEMP	2	0.14249844	874.71	0.0
OVEN	1	0.00360800	44.29	0.0001
ASPHALT*TEMP	60	0.00950103	1.94	0.0055
ASPHALT*OVEN	30	0.01348053	5.52	0.0001
TEMP*OVEN	2	0.00223958	13.75	0.0001

TABLE 12 COMPARISON OF TFOT AND RTFOT BASED ON CARBONYL RATIO

TEMP. DU	NCAN GROUPING	MEAN	N	OVEN
285°F	A	0.359371	31	TFOT
	Α	0.356042	31	RTFOT
325°F	Α	0.389452	31	TFOT
	Α	0.384955	31	RTFOT
365°F	Α	0.434623	31	TFOT
	В	0.416023	31	RTFOT

^{*} Means with the same letter are not significantly different.

TABLE 13 RESULTS OF SCHWEYER RHEOMETER TEST ON ORIGINAL ASPHALTS

	C VA	LUE	η _j (Pa	s)
ASPHALT	41°F	77°F	41°F	77° F
MA30A	0.78	0.99	105559000	225715
	0.83	1.00	89936900	188550
MA30B	1.11	1.02	74156400	342260
	1.09	1.09	89563200	278806
MA30C	0.63	0.89	226055000	416123
	0.72	0.90	159718000	359387
MA30D	0.61	0.92	136696000	386520
	0.57	0.85	244281000	394638
MA30E	0.87	1.16	153100000	202829
	0.93	1.10	116200000	210183
MA20T	1.17	1.01	63108600	10257
	1.14	1.00	62118000	159926
MA20C	0.99	0.90	78002200	515192
	1.05	0.87	45416900	691512
MA20V	0.89	0.99	54533000	220698
	0.86	0.95	60670800	19281
TL20	0.29	0.97	1778310000	281662
	0.28	1.01	1875600000	167020
TN20	0.79	0.92	487279000	951524
	0.82	1.07	482526000	53607
BJ30A	0.79	0.99	113312000	26496
	0.94	0.89	67216800	408088
BJ20	0.83	0.78	111734000	544280
	0.85	0.73	113379000	748936
CJ30A	0.62	0.99	286575000	34969
	0.61	1.03	278523000	301660
MT30	0.64	0.85	252477000	47154
	0.63	0.85	258437000	486388
CT30	0.78	0.87	105307000	408446
	0.68	0.82	176288000	677324
TB30A	0.72	0.64	36108400	836738
	0.57	0.64	62694200	876500
SA20	0.24	0.91	1638560000	23905
	0.26	0.92	1946950000	370852
AS30	0.47	1.06	1104530000	20981
	0.45	1.01	1111200000	256713
BF30	0.70	1.14	610784000	243592
	0.68	1.04	613913000	345046
MA30	0.68	0.94	230442000	482422
	0.78	0.92	254569000	521246

governmental agencies as specification tests (7). The AASHTO and ASTM standards state:

This method indicates approximate change in properties of asphalt during conventional hot-mixing at about 302°F (150°C) as indicated by viscosity, penetration or ductility measurements. It yields a residue which approximates the asphalt condition as incorporated in the pavement. If the mixing temperature differs appreciably from the 302°F level, more or less effect on properties will occur.

Results of the tests on asphalt binders presented in this paper have demonstrated that higher oven temperatures result in increased differentials in properties. For example, the change in viscosity ratio between 325°F and 365°F is greater than that between 285°F and 325°F. The higher test temperature of 365°F would magnify the asphalt properties change compared with the lower test temperature of 325°F. On the basis of percent penetration retained and absolute viscosity ratio, the effect of RTFOT is slightly more severe than that of TFOT for oven temperatures of 285°F and 325°F, whereas the difference is not significant at 365°F. On the basis of carbonyl ratio, the effects of TFOT and RTFOT are not significantly different for oven temperatures of 285°F and 325°F, whereas the TFOT is a more severe aging process than the RTFOT at 365°F. From the standpoint of low-temperature constant power viscosity, the effects of TFOT and RTFOT are not significantly different from each other at any of the three temperatures. Therefore, there is no reason to favor the RTFOT process over the TFOT process, especially if the oven temperature is raised to 365°F.

Comparisons were made between the residues after TFOT and RTFOT at 365°F, and recovered asphalts from specimens aged in a forced-draft oven and on the roof under natural weathering. Table 16 compares the different aging effects. It appears that the TFOT and RTFOT procedures performed at 365°F, 14 days aging at 140°F in the laboratory, and 3

					CONSTANT	POWER VISCOSITY (as)				
ASPHALT		OVEN TEMPERATURE 285°F 325°F		365°F	ASPHA	ASPHALT		OVEN TEMPERATURE 285°F 325°F 365°F			
MA30	TFOT	77°F	1.206E+06	2.243E+06	4.390E+06	MA20C	TFOT	77°F	6.044E+05	1.182E+06	3.107E+06
MA30	TFOT	59°F	2.122E+07	2.620E+07	5.240E+07	MA20C	TFOT	41°F	9.972E+08	8.493E+08	2.073E+09
MA30	RTFOT	77°F	2.836E+06	3.729E+06	3.218E+06	MA20C	RTFOT	77°F	6.528E+05	6.850E+05	1.964E+06
MA30	RTFOT	59°F	4.069E+07	4.780E+07	7.040E+07	MA20C	RTFOT	41°F	6.697E+08	1.256E+09	2.473E+09
BF30	TFOT	77°F	1.135E+06	2.069E+06	6.168E+06	MA20T	TFOT	77°F	2.562E+05	2.644E+05	6.999E+05
BF30	TFOT	59°F	3.460E+07	4.523E+07	1.072E+08	MA20T	TFOT	41°F	3.216E+08	7.226E+08	9.109E+08
BF30	RTFOT	77°F	1.526E+06	3.113E+06	6.523E+06	MA20T	RTFOT	77°F	3.039E+05	6.675E+05	2.099E+06
BF30	RTFOT	59°F	4.820E+07	8.612E+07	1.253E+08	MA20T	RTFOT	41°F	4.513E+08	5.146E+08	7.916E+08
MT30	TFOT	77°F	9.139E+05	1.871E+06	2.730E+06	MA20V	TFOT	77°F	5.049E+05	5.465E+05	1.250E+06
MT30	TFOT	59°F	7.607E+07	5.210E+07	1.241E+08	MA20V	TFOT	41°F	6.767E+08	8.064E+08	2.084E+09
MT30	RTFOT	77°F	1.419E+06	1.682E+06	2.679E+06	MA20V	RTFOT	77°F	4.123E+05	6.310E+05	1.682E+06
MT30	RTFOT	59°F	6.424E+07	5.830E+07	6.512E+07	MA20V	RTFOT	41°F	4.757E+08	6.813E+08	2.054E+09
BJ30A	TFOT	77°F	6.918E+05	2.693E+06	4.878E+06	TL20	TFOT	77°F	1.737E+06	2.860E+06	6.806E+06
BJ30A	TFOT	59°F	2.661E+07	9.145E+07	1.523E+08	TL20	TFOT	41°F	1.133E+09	2.715E+09	7.570E+09
BJ30A	RTFOT	77°F	7.384E+05	3.357E+06	4.740E+06	TL20	RTFOT	77°F	2.267E+06	2.776E+06	8.567E+06
BJ30A	RTFOT	59°F	3.146E+07	6.007E+07	2.162E+08	TL20	RTFOT	41°F	2.123E+09	4.203E+09	5.028E+09
CT30 CT30	TFOT TFOT	77°F 59°F	2.240E+06 5.086E+07	2.622E+06 8.103E+07	4.091E+06 1.259E+08						
CT30 CT30	RTFOT RTFOT	77°F 59°F	2.699E+06 4.119E+07	2.618E+06 6.083E+07	4.282E+06 9.348E+07						

TABLE 15 RESULTS OF ANOVA ON RHEOMETER TEST RESULTS

41°F SOURCE	DF	ANOVA SS	F VALUE	PR >F
ASPHALT	3	3.587895479527E+19	17.76	0.0022
OVEN	1	8.045031317606E+14	0.00	0.9735
TEMP	2 3 6	1.710756580212E+19	12.71	0.0070
ASPHALT*OVEN	3	6.586750236695E+16	0.03	0.9913
ASPHALT*TEMP		1.034365879240E+19	2.56	0.1387
OVEN*TEMP	2	1.004507498132E+18	0.75	0.5136
59°F				
SOURCE	DF	ANOVA SS	F VALUE	PR >F
ASPHALT	4	8678182209014668.0	4.58	0.0322
OVEN	1	59330671960336.0	0.13	0.7325
TEMP	2	26348574128073500.0	27.83	0.0002
ASPHALT*OVEN	4	2976281389387996.0	1.57	0.2714
ASPHALT*TEMP	8	11600818804872336.0	3.06	0.0670
OVEN*TEMP	2	4348036038164.0	0.00	0.9954
77°F				
SOURCE	DF	ANOVA SS	F VALUE	PR >F
ASPHALT	8	70490652640426.259	30.01	0.0001
OVEN	1	1216989513771.125	4.14	0.0587
TEMP	2	67283685634763.368	114.57	0.0001
ASPHALT*OVEN	8	1917444626002.031	0.82	0.5994
ASPHALT*TEMP	16	30612108812535.961	6.52	0.0003
OVEN*TEMP	2	107356391528.258	0.18	0.8346

TABLE 16 COMPARISON OF AGING EFFECTS

	TFOT	RTFOT	ROOF (MC	ON.)	LAB(DA)	(S)
	(365°F)	(365°F)	3	6	14	28
PENETRATIO	N AT 25 °C					
MA	34	33	34	30	33	32
BF	28	28	32	24	30	26
BJ	32	28	30	28	28	27
CT		32	31	30	33	31
MT	31	29	26	26	29	27
ABSOLUTE V	ISCOSITY AT	140 °F (Poi	se)			
MA	10583	11492	10313	14171	9987	11426
BF	12267	12267	11310	16074	11150	15515
BJ	15501	24079	14959	20353	16136	20900
CT	10599	12285	20239	21931	16555	21399
MT	16429	17832	20225	22904	14251	19556
CARBONYL R	ATIO					
MA	.4045	.3870	.4979	-5140	.4551	.5014
BF	.4735	.4601	.5027	.5333	.4861	.5182
BJ	.3674	.3761	.3761	.4855	.5141	.4914
CT	.4622	.4288	.4643	.4944	.4371	.5010
MT	.3869	.3577	. 4396	.4705	.4414	.4880
CONSTANT P	OWER VISCOS	ITY AT 77 °F	*******			
MA	4.39E6	3.22E6	1.43E6	3.15E6	1.83E6	3.93E6
BF	6.17E6	6.52E6	2.23E6	2.82E6	2.40E6	3.35E6
BJ	4.88E6	4.74E6	1.69E6	8.15E6	4.12E6	7.18E6
CT	4.09E6	4.28E6	2.70E6	3.90E6	2.91E6	4.25E6
MT	2.73E6	2.68E6	7.30E6	8.29E6	2.31E6	4.73E6
CONSTANT P	OWER VISCOSI	ITY AT 59 °F			********	
MA	5.24E7	7.04E7	4.00E7	4.75E7	6.64E7	6.71E7
BF	1.07E8	1.25E8	4.60E8	7.13E7	8.11E7	1.42E8
BJ	1.52E8	2.16E8	5.97E7	1.13E8	1.14E8	1.00E8
CT	1.26E8	9.35E7	6.77E7	8.49E7	6.59E7	6.69E7
MT	1.24E8	6.51E7	4.84E7	9.10F7	8.09E7	9.56E7

months of natural weathering would result in approximately the same hardening effects on a typical paving grade asphalt used in Florida.

From a previous study (8), it was found that Marshall specimens that were allowed to weather naturally for 6 months would harden approximately two to three times as much as the mixtures when placed and compacted in a pavement. Since the hardening effects of the TFOT or RTFOT at 365°F are about the same as those of 3 months natural weathering, the hardening effects of the TFOT or RTFOT at 365°F should be about the same as those of 6 to 9 months aging on the roads.

SUMMARY

The major findings of the tests on asphalt binders are as follows:

- 1. On the basis of percent penetration retained and absolute viscosity ratio, the RTFOT is a more severe aging process than the TFOT for oven temperatures of 285°F and 325°F. However, the two processes are not significantly different at an oven temperature of 365°F.
- 2. The carbonyl ratio, a ratio of infrared absorbance at 1700 cm⁻¹ and 1600 cm⁻¹, can be used to express the level of oxidation in an asphalt binder.
- 3. On the basis of carbonyl ratio, the effects of TFOT and RTFOT are not significantly different from each other at oven temperatures of 285°F and 325°F. However, at 365°F, the

TFOT is a more severe aging process than the RTFOT from the standpoint of carbonyl ratio.

- 4. On the basis of constant power viscosity, the effects of TFOT and RTFOT are not significantly different from each other at any of the three temperatures.
- 5. Asphalts of the same grade at high temperature (60°C) can have very different properties at lower temperature.
- 6. Higher oven temperatures result in increased differentials in properties (i.e., the change in viscosity between 325°F and 365°F is greater than that between 285°F and 325°F.
- 7. The constant power viscosity-temperature relationship of asphalt is shifted parallel to the original relationship after aging process. The relationship shifts more for higher oven temperature than for lower oven temperature.
- 8. As a rough estimate, the TFOT or RTFOT procedure performed at 365°F, 3 months of natural weathering of Marshall specimens, and 6 to 9 months of aging in a pavement would result in approximately the same hardening effects on a typical paving grade asphalt used in Florida.
- 9. From the results of this study, it appears feasible to use TFOT or RTFOT at a higher temperature to simulate the aging process on the asphalt binder in service as well as that of the hot-mixing process.

REFERENCES

 M. Tia, B. E. Ruth, C. T. Chari, J. Shiau, D. Richardson, and J. Williams. Investigation of Original and In-Service Asphalt Properties for the Development of Improved Specifications—Final Phase

- of Testing and Analysis. Final report. UF Project 4910450420812. Department of Civil Engineering, University of Florida, Gainesville, 1988.
- 2. C. T. Chari, B. E. Ruth, M. Tia, and G. Page. Evaluation of Age Hardening on Asphalts and Mixtures. Proc., AAPT, Vol. 59, 1990,
- 3. K. O. Anderson, B. P. Shields, and J. M. Dacyszyn. Cracking of Asphalt Pavements due to Thermal Effects. Proc., AAPT, Vol. 35, 1966, pp. 247-262.
- 4. R. J. Schmidt. The Relationship of the Low Temperature Properties of Asphalt to the Cracking of Pavements. Proc., AAPT, Vol. 35, 1966, pp. 263-269.
- 5. M. Tia and B. E. Ruth. Basic Rheology and Rheological Concepts Established by H. E. Schweyer. Asphalt Rheology: Relationships to Mixtures, ASTM STP 941, 1987, pp. 118–145.
 6. A. C. Edler, M. M. Hattingh, V. P. Servas, and C. P. Marais.
- Use of Aging Test To Determine the Efficiency of Hydrated Lime

- Additions to Asphalt in Retarding Its Oxidative Hardening. Proc., AAPT, Vol. 54, 1985, pp. 118-139.
- 7. O. K. Kim, C. A. Bell, J. E. Wilson, and G. Boyle. Development of Laboratory Oxidative Aging Procedures for Asphalt Cements and Asphalt Mixtures. In Transportation Research Record 1115, TRB, National Research Council, Washington, D.C., 1987, pp. 101-112.
- 8. M. Tia, B. E. Ruth, J. Shiau, S. Huang, and D. Richardson. Evaluation of Criteria for Improved Durability of Asphalt. Final report. Department of Civil Engineering, University of Florida, Gainesville, 1990.

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Summary of Asphalt Additive Performance at Selected Sites

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In 1990, a survey of modified asphalt test pavements was conducted to examine performance during construction and service while using the various additives on the market. Representatives from 14 states, Austria, and Canada were contacted to survey the field performance of more than 30 end-to-end test pavements where one to five asphalt additives, modifiers, or modification processes have been evaluated. States contacted include Alabama, California, Colorado, Indiana, Louisiana, Maine, Minnesota, Montana, New Jersey, Texas, Utah, Vermont, and Virginia. Additives include styrene butadiene rubber (SBR) latex, Styrelf-13 (SBR reacted with asphalt), DuPont neoprene latex, styrene butadiene styrene block copolymer (Shell Kraton), polyethylene (Novophalt process), ethylene vinyl acetate (Exxon and DuPont), Solar Laglugel (nylon resin polymer), Gilsonite, asphalt-rubber (tire rubber), Dow polymers (unknown), Plus-Ride (ground tire rubber replacing aggregate), Microfil-8 (pelletized carbon black with oil), Celite (diatomaceous earth), Chemkrete, hydrated lime, fly ash, Acra 500, and fibers. Most of the results have been collected by telephone and are, therefore, qualitative. Some of those contacted have sent written reports that provide more quantitative data. Unfortunately, many of the test pavements are less than 5 years old and show no differences in performance.

The objective of this work was to rapidly summarize information on construction and relative field performance of modified asphalt test pavements in North America and Europe. In 1 week, representatives of at least 14 states, Canada, and Austria were contacted. Information was collected on more than 30 test sites where adjacent test pavements were built to evaluate one to five asphalt modifiers. Field performance of about 20 different modifiers was examined.

Most of the findings were obtained by telephone and, therefore, are qualitative and subjective in nature. Some more quantitative findings from written documents are also presented.

FIELD PERFORMANCE

Findings from this survey are summarized in Tables 1 and 2. Table 1 gives a brief description of the test pavements, field operations, and performance. Table 2 contains a brief synopsis of the perception of the individual user agency representative regarding asphalt additive performance and a prognosis of future use by the agency.

ADDITIVES AVAILABLE

Most of the known asphalt additives available in today's market (12) have been categorized by generic name in Figure 1.

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Some of these products are used routinely in bituminous paving mixtures; others are still in the experimental stage.

Polymers including neat rubbers are the most versatile and probably hold more promise to improve structural and adhesive properties of bituminous pavements than any other single category of additives on the market today. Generally, the polymer-type additives are shown to reduce binder temperature susceptibility and brittleness and increase toughness (energy of deformation) and tenacity. Polymers in asphalt mixtures have exhibited moderate improvements in Marshall stability and tensile properties but generally no significant increase in Hveem stability. (Although Hveem stability is sensitive to binder content, it is not sensitive to binder properties.) Specialized laboratory tests designed to estimate resistance to pavement cracking and permanent deformation often show significant benefits when polymers are used.

Latexes have been used experimentally in bituminous paving applications for more than 30 years and now enjoy widespread use. The term "latex" is derived from the natural rubber industry and describes the milky fluid that comes from rubber trees (or even dandelions). The term has been adopted to describe the initial product, which is synthetic rubber emulsified in water, from the rubber manufacturing process. Different types of synthetic rubber are available in the latex form. The latex most widely used in the paving industry is styrene butadiene rubber (SBR). SBR latex is routinely used in chip seals in many parts of the country. It is becoming more frequently used in hot paving mixtures. Several asphalt producers and suppliers have recently made plant modifications to facilitate the supply of SBR latex modified asphalt and thus save the user the trouble of blending the two products. The primary reason for the widespread use of SBR latex is its availability and relative cost compared with competing polymers. Neoprene latex has also been used for many years in bituminous pavements but with much less frequency than SBR

Pelletized carbon black containing about 8 percent oil as a binder for the pellets has also demonstrated positive results in improving asphalt pavement performance (13). Carbon black has been incorporated into bituminous paving mixtures by adding preweighed plastic bags of the material into the pug mill at a weigh batch hot mix plant. Heretofore, carbon black dispersed in hot asphalt cement quickly settled out when stored statically because of the difference in specific gravity of the two materials. Dispersing agents have been developed that will keep the carbon black in suspension and thus permit its use in drum mix plants (14). The dispersion process requires specialized equipment and adds significantly to the cost of the modified asphalt. More recently, a portable blending unit has

Location	Additives Tested	Date Placed	Pavement Section	Climate	Traffic	Summary of Findings/ Performance in 1990
Alabama (highway not specified)	Styrelf, Elvax, Novophalt, Gilsonite and Neoprene	1987	(Not Specified).	Moderate	Not specified	Gilsonite was troublesome to handle since it was supplied in 50-100 pound bags. It was mixed in the storage tank. Modified asphalts caused no problems during mixing, placement, or compaction. There are no significant difference in performance.
California IK-80 near Alta (elevation 3,000 ft.)	Kraton with Dutrex extender oil (furnished as an AR-4000 preblended at the refinery), Bonifibers (polyester fibers), Fiber Pave (poly propylene fibers), Carbon black, Ramflex (devulcanized rubber)	June 1985	1.8" surface course 1.2" leveling course cracked and seated PCC pavement.	Severe Winters	Not specified	Test sections ranged from 1,000 to 2,000 feet in length. Placement temperatures were above 325°F. Rocks were occasionally picked up by the roller on the polymer-modified. As a result of a few unusually mild winters, all pavements are performing well; there are no differences in pavement performance associated with the additives (1).
California (highways not specified)	Asphalt-rubber (A-R) and Plus-Ride	1987	(Not specified). Thickness of some of the A-R sections was reduced to compare to control	Cold	Not specified	Reflection cracking was more severe in the control sections and in the thinner A-R sections than in the A-R sections that were the same thickness as the control sections.
			sections.			In test sections installed at Ravendale in 1987, the A-R sections have "far outperformed" the control pavements and are exhibiting less cracking. Plus-Ride is performing equivalent to A-R.
California IH-10 East of Indio	Styrelf PAC-40 produced by Elf Aquitaine Asphalt (manufactured using Edgington AR4000 asphalt), a polymer modified AR-4000 produced by Witco (Golden Bear), and Novophalt (a blend of Paramount AR-4000 asphalt and polyethylene pellets mixed at the plant site using a special Novophalt unit).	September 1988	4.2" of previous overlay was cold planed and replaced with 4.2" of an all crushed 11/4" maximum size coarse graded AC base mix (using AR-8000), 3" of 3/4" maximum size coarse graded surface mix (also AR-8000). Modifiers were used in the coarse graded surface mix in the number 2 lane.	Desert	Not specified	Test sections were 0.25 miles in length. No unusual problems were encountered during mixing and placing. After two years, some rutting (1/4" - 3/8") has appeared in isolated areas in the Golden Bear section, likely due to high binder contents. Tests indicated lower in-place voids (3.3 percent) compared to other sections (4.3 - 5.4 percent) {2}.
California US 395 adjacent to the Caltrans Crestview Maintenance Station about 50 miles of Bishop (elevation 800 feet)	Polymer modified binders were supplied by Conoco and Witco (Golden Bear - AR 4000) and were preblended prior to arrival at the plant site. The suppliers of the modified asphalts were to use as a guideline the Caltrans proposed durable asphalt specification plus a minimum penetration at 39.2°F of 35 dmm on the residue from thee RIFO procedure.	1988	4.8" overlay with 3/4" maximum size aggregate.	Co ld	Not specified	After two years, the unmodified pavement is showing about three cracks per 100 feet in the surface while the modified pavement (Golden Bear) has none. Results from the Conoco section are not known.
Colorado (highway not specified)	AC-20R: a SBR-treated AC-10 asphalt meeting AC-20 specification after modification.	Not specified	Not specified	Cold	Not specified	After three years in service, the AC-10 section is exhibiting about twice as much cracking as the modified section.
Indiana Hwy 465, and US 41 (city of Terre Haute)	SBR latex, Novophalt, neoprene, Styrelf, asphalt- rubber, polyester fibers, polypropylene fibers.	1988	All three layers contain additives and the surface layer was overlaid over cracked and seated concrete pavement.	Cold	Not specified	There are no differences in performance of the various sections on Hwy 465. For US 41, the polymer test section is in excellent shape in spite of a low air voids. The control sections are showing significant rutting.
Louisiana Hwy 46 from Haydras to Riggio	Styrelf and SBR latex	1984	3/4" thick open graded friction course (OGFC) containing AC-30 at 6.1 or 6.7 percent binder, styrelf at 6.1 or 6.7 percent binder or latex at 6.7 percent binder.	Wann	Not specified	After 2 to 3 months in service, raveling in the control sections was detected at turnouts. After 5 years in service, the polymer modified sections began to ravel. After 6 years, the control sections are in very poor condition while the modified sections are in excellent condition. The key factor to the differences in performance is the binder film thickness, which was insufficient at 6.1 percent. The polymers have shown little positive benefit.

TABLE 1 (continued)

Location	Additives Tested	Date Placed	Pavement Section	Climate	Traffic	Summary of Findings/ Performance in 1990
Louisiana La 686	The control asphalt was Texaco AC-30. Three percent SBR latex was blended in a drum plant with AC-10, and Styrelf was preblended with AC-10.	1986	2" overlay over 3" badly cracked overlay surface treatment cement treated base.	Warm	Not specified	Within one week after construction, all sections began to crack. After four years in service, all overlays are performing similarly. The styrelf section is performing a little better than the latex, and the latex section is performing better than the control section. Extractions after one year revealed the latex binder was aging faster than the Styrelf binder.
Louisiana Florida Boulevard in Baton Rouge	Latex (metered directly into pug mill)	Fall 1987	2" overlay with 3/4" maximum size siliceous river gravel and sand (8 percent minus No. 200).	Warm	81,000 ADT	The control asphalt was an AC-30 and the modified asphalt was an AC-10. After two years, rutting in the control sections measured 0.5 inches and rutting in the latex section measured less than 0.2 inches. No flushing has occurred in either section (3).
Louisiana US 190	Ultrapave 70 latex	1988	Overlay on existing PCC pavement.	Warm	Not specified	The latex-modified asphalt concrete is controlling reflective cracking better than the control mix {4}.
Maine IH-95	Ultrapave latex, Dow polymers (identified by number) and latexes	1981 and September 1986	2" overlay	Cold	Not specified	1981 tests of Ultrapave latex are showing no significant differences in performance of the control sections and the latex sections. Test sections for the Dow polymers and latex were 1,000 feet in length. The test sections were made with asphalts modified with No. 1 polymer, No. 2 polymer, latex and blends of each polymer with latex. Some sections have performed poorly and some have performed well.
Minnesota City of Rosenville	Polysar latex +200-300 pen asphalt (sprayed into drum mix plant) The control asphalt was 120- 150 penetration grade.	1986	1" - 2,5" HMAC surface composed of 40 percent crushed stone and 60 percent gravel and sand with a maximum aggregate size of 0.5 inch.	Cold	Not specified	In all construction projects with the rubber additives, the DOT noted the presence of rubber-rich globules in the mix. These globules appeared on the finished pavement surface as binder-rich spots about the size of a silver dollar. The department is evaluating cracking, rutting, and flushing in these pavements, Currently, all pavements are performing well and there are no observable differences between the test pavements.
Minnesota Hwy 61 South of Hastings and just north of intersection with Hwys 50 and 20	Dupont neoprene latex L735A (introduced directly into drum mix plant)	May and June of 1988	1. Neoprene modified RAP mix in the binder course with unmodified virgin mix in the wearing course; 2. Modified RAP in the binder course with modified virgin mix in the wearing course; 3. Unmodified binder course with modified wearing course; and 4. Unmodified binder and wearing courses (control).	Cold	Not specified	During paving operations, the mixture showed evidence of unmixed neoprene in the form of small globs which later appeared as binder-rich spots on the paved surface. No performance results are available.
Minnesota Trunk Highway 63	Carbon black (Cabot Microfil) and sulphur to reduce reflective cracking.	1984	1.5" overlay 3" bituminous surfacing 6" crushed rock base 12" sand and gravel subbase.	Cold	1,600 ADT	The two additives were blended with a 200-300 penetration asphalt. One section contained an untreated 200-300 penetration asphalt and the control section contained a 120-150 penetration asphalt. Soon after construction, the 200-300 penetration asphalt section began flushing badly while the treated sections did not. None of the materials were successful in preventing reflective cracking. The carbon black section has the least cracking. Laboratory tests of extracted binders indicate that carbon black is retarding oxidative hardening. Rutting is minor, but the section with the 200-300 penetration asphalt is exhibiting more rutting (5).

TABLE 1 (continued)

Location	Additives Tested	Date Placed	Pavement Section	Climate	Traffic	Summary of Findings/ Performance in 1990
Montana IH 90 near Big Timber	Hydrated lime, Acra 500 (antistrip fly ash, Chemkrete and Microfil 8 (carbon black)	1983	4.8" bituminous surface course placed in two lifts.	Cold	Not specified	Two asphalts were used, a 120-150 penetration grade and a 200-300 penetration grade. The aggregate was a 3/4" maximum size crushed stone with 6 percent minus No. 200 materials. The carbon black was added at the drum plant. A significant amount was removed by the exhaust gas cleaning system and ended up floating on the surface of the sump pond. It tended to plug the feed lines from the silo to the plant. While placing the first lift, the Chemkrete mix appeared tender at times.
						During the first summer, the Chemkrete section began to flush and rut (1/4 inch). After two summers, the sections containing Chemkrete, ACRA-500 and the 200-300 asphalt had ruts greater than 1/4 inch while the other sections had ruts less than 1/4 inch. Minor cracking had appeared in all test sections. Rut depths increased after the hottest summer in 50 years (1988) [§].
New Jersey Route 41	Chemkrete, Texcrete (SBR latex), solar Laglugel, 3M #5990 (Polyethylene pellets) and Plus Ride.	August 1984	1.5" HMAC surface course.	Cold	11,200 ADT	Test sections are approximately 36 feet in width and 1740 feet in length. Generally, most of the pavement sections are performing well with no major differences between the different sections. The 3M \$5990 was the least resistant to cracking. The cold joints cracked soon after construction and the sections now has 84 feet of transverse cracks. The Texcrete section has a small amount of fatigue cracking in the wheelpath and about 10 feet of transverse cracks. None of the test sections are exhibiting significant rutting (1/8" - 1/4" ruts).
New Jersey Roule 35	Exxon EVA, Novophalt, Gilsonite, Carbon Black, Kraton, Solar Lag	November 1987	3" HMAC overlay placed in two lifts.	Cold	30,000 ADT	The only significant difference between test sections is rut depths, with the control AC-20 exhibiting the lowest rut depths. Raveling, usually a moisture related problem, is not uncommon in carbon black modified asphalt pavements. Cracking developing is most likely related to the weak substrate on which these overlays were placed.
Texas SH 121 in Fort Worth	SBR latex (Polysar, Ultrapave, and DOW) = added in the drum plant	Summer 1985	2" overlay with 3/8" maximum size lightweight synthetic aggregate and field sand) on a geotextile placed on CRCP.	Warm	12,000 ADT	Within one year after construction, rutting and flushing was significantly greater in the control section (with 8.5 percent asphalt content) than in the latex section (with 7.5 percent asphalt content). The control section had to be removed and replaced. The latex sections are still performing well after five years.
Texas US 83/77	Microfil - 8 (preblended), Polybilt-102, Ultrapave - 78		3" HMAC modified test sections and 4" HMAC control test sections with 3/8" maximum size		11 002 107	Two 1/4-mile test pavements containing each additive were placed as new construction in the outside lane of a 4-lane divided highway. The modified test sections used an AC-10 asphalt while the control section used an AC-20 asphalt.
in South Texas near Brownsville	latex, and Kraton D with Dutrex extender oil	August 1986	crushed river gravel and field sand. All mixes contained one percent hydrated lime as a slurry.	Tropical	15,000 ADT	During construction, the latex caused the mix to cling to the beds of the haul units and caused notable drag on the paving machine. After 3,5 years in service, there are no significant differences in the appearance of the test pavements that are attributable to the additives. Rutting in all the sections ranges from 0 to 1/8 inch.

(continued on next page)

been developed and will soon be available for use at mixing plants to alleviate the problems of blending Microfil in the field.

Asphalt rubber in paving mixtures as well as stress-absorbing membrane interlayers (SAMI) has been tested extensively in highway pavements (15-19). This product, composed of ground tire rubber in asphalt, typically requires 18 to 22 percent rubber

by mass of asphalt cement. The rubber must be ground to a minus number 10 sieve size, which requires significant effort and cost. The material decreases the ductility of asphalt to such an extent that it will not meet many state department of transportation specifications. It will probably not be cost-effective compared with products that contain 2 to 5 percent neat polymer. Pressure from the federal government, how-

TABLE 1 (continued)

Location	Additives Tested	Date Placed	Pavement Section	Climate	Traffic	Summary of Findings/ Performance in 1990
Texas US 75 near Sherman	Elvax-150, Novophalt, Kraton D with Dutrex extender oil, Ultrapave 70 latex, and Microfil - 8 (added in plastic bags at the pugmill). All polymers except Novophalt were preblended prior to arrival at the construction site.		3" HMAC modified test sections and 3" and 4" HMAC control sections with 5/8" maximum size crushed limestone and field sand.	Warm- Moderate	18,000 ADT with 17 percent trucks	The test pavements were 1/2-mile in length in the outside lane of a 4-lane divided facility composed of an old, cracked CRCP. All additives were blended with AC-10 and the control sections contained AC-20. After 3.5 years in service, there are no detectable differences in the visual appearance of the sections. The pavements are performing well and the ride quality is approximately equivalent.
Texas US 59/71 near Texarkana	Ultrapave latex, Chemkrete (CTI-102), Polybilt 102, and Styrelf-13	Fall 1987 through Spring 1988	2" surface mix with 3/8" maximum size crushed sandstone with field sand. 8" asphalt stabilized based with 7/8" maximum size crushed sandstone with field sand.	Moderate	10,000 ADT with 15 percent trucks	The test sections were 0.9 mile in length and comprise both lanes of a 4-lane divided facility. The control pavement contains AC-20. Latex was blended with AC-10, styrelf was preblended with soft asphalt, and the Chemkrete and Polybaishlat, and the AC-20 at the plant site. During construction, the latex and styrelf mixtures tended to stick to the tires of the pneumatic roller. After two years in service all pavements are
						performing well and the surfaces of the pavements have equivalent appearance.
Vermont Montpelier State Hwy.	Solar Laglugel - a resin and nylon base modifier supplied by Additives of New England, Inc.	October 1982	l 1/4" overlay.	Cold	Not specified	The modified mix gave off a strong odor and was stickier than the standard mix but presented no significant problems during placement. The DOT monitored reflective cracking, rutting, bituminous mix properties, ride quality, surface friction values and maintenance requirements in these pavements for five years and saw no cost effective or beneficial effects. In general, there was little difference in performance between the modified and unmodified pavements (Z).
Vermont Route 12 near the Worcester/Elmore town line between milemarker 0670 and milemarker 0701	Ultrapave latex (SBR)	September 1984	1" surface course.	Cold	Not specified	There were no significant problems with the production or placement of the HMAC. Several "fat spots" stuck to the compaction roller and pulled out leaving small "pock marks" in the pavement surface. Workers commented that the material was very sticky and stringy, making hand work more difficult. Crack counts and rutting surveys have indicated no detectable differences in the performance of the latex modified test sections.
Virginia Route 58	Polybilt - 100, Dow Downright HM 100L, Styrelf- 13, Ultrapave and a Celite 292 sedimentary diatomaceous deposit filler. The evaluation of these additives to improve the deformation and flexibility characteristics of asphalt paving mixtures was investigated.	1986	1.5" overlay over milled asphalt pavement.	Cold	Not specified	The test sections ranged in length from 0.6 to 0.8 miles. There are no discernable differences between the sections containing the various additives. Considerable stripping was evident in the pavement cores containing the Celite; this stripping is expected to affect performance eventually. The additives were not considered cost effective on this project because traffic was not severe enough to cause significant rutting in the conventional control mix (9), (10).
Virginia	Polybilt, Microfil-8 and fibers	1989	Not specified	Cold	Not specified	Test sections made from Polybilt modified asphalt concrete are performing well. Microfil-8 was used in a bridge deck; it appears to have improved resistance to plastic deformation in a fine-grained, low void mix. Fibers have been used in small installations; no valid conclusions can be made at this time.
Austria Vienna Metropolitan Hwy at Praterhochbruecke	Novopha lt	1980	Not specified	Cold	Not specified	After 8 years in service, the Novophalt section was exhibiting 0.16 to 0.24 inches of rutting; the control section was exhibiting 0.70 to 1.0 inches of rutting.

TABLE 1 (continued)

Location	Additives Tested	Date Placed	Pavement Section	Climate	Traffic	Summary of Findings/ Performance in 1990
Austria Semmering Freeway - 56 at Wartmannstetten and Oberdanegg	Novophalt - in the surface course only	1983	2.0" surface course 2.4" binder course Section I - untreated control pavement using limestone aggregate. Section II - similar to section I but with Novophalt in the surface course. Section III - Similar to Section III but basalt aggregate was used in the surface course.	Cold	Nat specified	After four years in service, Section I had 0.16 inches of rutting, Section II had 0.11 inches of rutting, and Section III had 0.22 inches. These rut depths are not considered serious; 0.5-inch ruts are usually considered the minimum for which maintenance is required.
Canada City of Edmonton Highway not specified	A polymer supplied by Imperial Oil (Exxon) and SBS rubber from Husky Oil.	1987	2" surface course	Cold	High volume, heavy truck traffic	After two years, more cracking was evident in the Imperial Polymer section than in the control section. There was no appreciable rutting in either section. A SBS rubber product from Husky Oil was used in a 2" overlay on an old portland cement concrete pavement. Spacing between cracks in the old pavement was about 50 feet. Cracks reflected through in about two months. In three years of service, no rutting occurred. Asphalts modified with the Husky product are reportedly very stable during prolonged hot storage, giving no evidence of phase separation or loss of viscosity.
Canada City of Edmonton Highway not specified	Navopha lt	1987	8" surface layer.	Co ld	High volume, heavy truck traffic	After three years, the Novophalt modified pavement is exhibiting significantly less rutting than the control pavement (0.25-inch versus 0.5-inch). There is no significant cracking in these pavements.
Canada City of Montreal Autoroute 20	Styrelf	1987	Open-Graded Hot Mix	Cold	Heavy	After 3 years pavement is performing better than materials used previously to pave this troublesome section of roadway. Styrelf controled draindown during handling of the open-graded mix (11)-

ever, may promote its use in some instances when it is not cost-effective to minimize the solid waste disposal problem with automobile tires.

As mentioned previously, most of the polymer, rubber, and carbon black additives improve the temperature susceptibility of an asphalt. This change in the rheological properties of the asphalt depends, of course, on the type of additive and the quantity added. Generally, one can expect a significant increase in binder viscosity at temperatures above 40°F and no appreciable change in consistency at temperatures below 40°F. Therefore, by using an asphalt one or two grades softer than that normally used in hot mix asphalt concrete plus an appropriate additive, one can take advantage of the original low viscosity of the asphalt in the low temperature range to increase resistance to cracking and, simultaneously, depend on the higher viscosity in the high temperature range to increase resistance to rutting on highways and depressions made by tires in parking areas.

OTHER CONSIDERATIONS IN THE USE OF ADDITIVES (12)

The primary disadvantage of the increased viscosity of the modified binders at high temperatures is that it extends into the temperature range at which asphalt concrete is mixed (275°F to 325°F). It is, therefore, often necessary to increase

the operating temperature of the mixing plant to achieve adequate coating of aggregate and provide for satisfactory compaction of the paving mixture. Plant temperature increases from 0°F to 70°F have been reported, with about 30°F being most usual. Obviously, the required temperature increase will depend on the type and quantity of additive used. This is, nevertheless, an important consideration for the paving contractor from an economic standpoint, in that more fuel will be required to operate the plant at a temperature higher than normal. Higher mixing temperatures can result in improved resistance of the mix to damage by moisture (20), which, in times past, may have been attributed to the additive.

When a given quantity and type of polymer is added to an asphalt product, the resulting physical properties of the binder will vary with asphalt source. Crude petroleums from various parts of the world vary significantly in chemical composition. Refining processes also cause variation in the chemical makeup of asphalt cement. The aromatic polymers, like SBR latex, may be more compatible with the more aromatic asphalts. Asphalts high in asphaltenes (above 25 to 30 percent) are generally not well peptized and, therefore, may exhibit poor compatibility with most polymers.

Many paving agencies specify an extraction procedure to verify the presence of the design quantity of bituminous binder. Some of the polymers and carbon black are only partially soluble or insoluble in the conventional solvents and, as a result, interfere with the extraction process and may cause

TABLE 2 COMMENTS FROM REPRESENTATIVES OF STATE DEPARTMENTS OF TRANSPORTATION ABOUT USE OF ASPHALT ADDITIVES IN HMAC, 1990

Location	Perception of the User About Additives	Plans for Future Use
Alabama	Additives appear to have promise.	The DOT will not buy polymer additives for HMAC in the near future, but will in the distant future.
California	DOT likes to use polymer additives because they can use softer asphalts which age at a slower rate in the desert climates and crack less in the cold climates.	The DOT will regularly use modified asphalt.
Colorado	The DOT believes the rubber-type polymers are cost effective in reducing rutting, cracking, and stupping.	The DOT uses significant amounts of rubber-type polymers in their routine asphalt pairing operations and will like increase the use of these additives.
Iowa	DOT believes that most polymers appear to make significant changes in asphalt material properties but are not cost effective regarding pavement performance.	DOT will probably not buy polymer additives for general use.
Indiana	The DOT believes fibers are cost effective in reducing reflection cracking over cracked and seated concrete pavement. Fibers reduce the thickness of the overlay.	The DOT will continue to use fibers and is developing thickness equivalency factors.
Louisiana	Latex modified mixes are showing increased benefits over the conventional mixes in controlling reflective cracking. The DOT believes polymers and latex are cost-effective in some applications. The ability of the polymers to enhance binder properties at both high and low temperatures should provide for longer service life in dense and open-graded mixtures.	The DOT will specify polymers and latex address special problems or situations where it is anticipated that cracking or rutting would occur in conventional mixe
Maine	DOT believes additives improve pavement performance but not to an extent that they are cost effective.	DOT does not anticipate using large quantities of polymer-type asphalt additives in the near future.
Minnesota	The DOT considers additives, such as carbon black and sulphur, to be experimental.	The DOT will continue to test new additives as they appear on the market. will likely be several years before they purchase large quantities of additives for routine use.
Montana	The DOT has a genuine interest in the cost effectiveness of various asphalt additives.	The DOT will continue to monitor performance of test pavements in an attempt to develop correlations between pavement performance and various binder properties.
New Jersey	The DOT considers additives and other modifications to be experimental.	They do not plan to use additives routinely in the near future.
Texas	DOT is observing several experimental pavements, uses additives occasionally to address special problems.	Will continue to monitor performance and specify additives in special situations.
Utah	Utah uses latex and hydrated lime routinely in their hot mix work. They have been using latex since the 1960's. Latex is used in areas of high volume or stop and go traffic, places where early pavement distress is expected. Lime is required for marginal mixes to meet specifications for the AASHIO 1283 moisture treatment procedure	They will continue to specify latex.
Vermont	The DOT believes additives offer improvements in asphalt pavement performance but that they are not usually cost effective.	The DOT does not anticipate the routine use of asphalt additives in the near future.
Virginia	Additives are too expensive to use routinely on long stretches of interstate highways.	The DOT will probably use additives for special situations where early distress may be expected due to high volume, heav traffic or intersections.

erroneous results. Some agencies go a step further and periodically recover the bituminous binder from the extraction solvent to determine its physical properties. Properties of polymer-modified binders recovered after the extraction process are questionable because, even if all the modified binder is recovered, the additive and bitumen have been intimately and unrealistically blended in the procedure, which, in all probability, significantly changed the rheological properties.

Tests to verify the quantity of polymer in bitumen are not difficult but are time-consuming and expensive. Fourier transform infrared (FTIR) analysis, with careful calibration, using mixtures of known quantities of bitumen and polymer, can readily be used to determine the polymer content of a modified binder (21,22). However, for reasons discussed previously, the difficulty factor rises sharply if the modified binder must be first extracted from an aggregate mixture.

Heat stability of polymer-modified binders has been evaluated in an attempt to predict problems that might occur during prolonged hot storage (23). After exposure to 325°F for 24 hr while protected from oxidation, SBR and SBS products exhibited a significant decrease in viscosity. The drop in

viscosity is apparently due to a breakdown of the molecular structure of the polymer. Similar findings have been reported from the field after prolonged hot storage in a tank. In one case, the user agency rejected the modified binder because it no longer met viscosity specifications. In another case, the damaged binder was used, but significant mixture tenderness was noted during construction. One highway district in the Texas Department of Transportation specifies that SBR latex be added to the asphalt mixture in the mixing plant to avoid hot storage and possible damage to the modified asphalt.

BENEFIT-COSTS FOR ADDITIVES

Costs of the most widely used polymer additives are influenced by the cost of crude oil, as is the cost of asphalt cement. Currently, the price for the commonly used polymers ranges from \$0.80/lb to \$1.00/lb. For the carbon black, the price is about \$0.50/lb. This translates into a cost increase of about \$4.00/ton to \$9.00/ton of hot mixed asphalt concrete, depending on the dosage of the additive. On the basis of an in-

- 1. Polymers
 - a. Styrene Butadiene Rubber (SBR) (Latex)
 - b. Block Copolymers
 - i. Triblock Styrene-Butadiene-Styrene (SBS)
 - ii. Radial Block SBS
 - iii. Vulcanized (SBR)
 - iv. Styrene-Isoprene-Styrene (SIS)
 - v. Styrene-Ethylene-Butylene-Styrene (SEBS)
 - vi. Styrene-Ethylene-Propylene-Styrene (SEPS)
 - c. Polyethylene
 - d. Ethylene Vinyl Acetate (EVA)
 - e. Polypropylene
 - f. Crumb Tire Rubber
 - g. Polychloroprene latex
 - h. Polychloroprene solids
 - i. Natural Polyisoprene
 - j. Synthetic Polyisoprene
 - k. Ethylene Propylene-Diene-Monomer (EPDM)
 - 1. Polyisobutylene
- 2. Extenders
 - a. Sulfur
 - b. Fillers
- 3. Mineral Fillers
 - a. Carbon Black
 - b. Hydrated Lime
 - c. Flyash
 - d. Silica Fines
 - e. Baghouse Fines
- 4. Natural Asphalts
 - a. Trinidad
 - b. Gilsonite
- 5. Antistripping Agents
 - a. Amidoamines
 - b. Imidazolines
 - c. Polyamines
 - d. Hydrated Lime
 - e. Organo-metallics
- 6. Antioxidants
 - a. Diethyldithio Carbamates
 - b. Viscosity Modifiers
 - i. Lead
 - ii. Zinc b. Carbon Black
 - c. Hydrated Lime
 - d. Phenols
- 7. Hydrocarbons
 - a. Tall Oil
 - b. Aromatics
 - c. Naphthenics
 - d. Paraffinics/Wax
 - e. Vacuum Gas Oil
 - f. Petroleum/Plastic Resins
 - g. Asphaltenes
- 8. Fibers
 - a. Polypropylene
 - b. Polyesterc. Natural
 - d. Glass
- 9. Others
 - a. Gelling Agents
 - b. Viscosity Modifiers

FIGURE 1 Bitumen additives currently being used or tested in pavements.

place cost of \$32.00/ton of hot mixed asphalt concrete, the additives would increase the paving cost by about 12 to 28 percent. Therefore, assuming an average overlay life of 13 years, an additive would need to increase pavement life by less than 2 to 4 years to be cost-effective or decrease maintenance costs accordingly, or both. On the basis of laboratory test results and findings from the older field tests in the United

States and Europe, certain polymer and microfiller additives properly applied can reasonably be expected to provide cost-effective pavement performance. Indiscriminate use of asphalt additives will not permit cost-effectiveness.

OUTLOOK FOR ADDITIVES

Vehicle weights, traffic volume, and tire pressures are steadily increasing and demanding more and more from pavement structures. Engineers are faced with serious problems regarding quality of paving material. Often materials are shipped long distances at high cost because local material supplies of high quality have been depleted. As a result, bituminous binder additives have been widely accepted by the paving industry for the present time. The concept of additives is logical, and results from laboratory testing look positive. Even though field test results using many additives are incomplete, many of those responsible for pavement quality are willing to gamble because the odds appear to be in their favor.

The bituminous binder additive industry and associated technology are advancing at a rapid rate. By the time results from the field are available for the additives being currently marketed, it is reasonable to assume that a whole new generation of bitumen additives will be on the market. It is, therefore, surmised that the outlook for additives in asphalt paving materials is excellent.

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REFERENCES

- 1. R. Reese. Evaluation of Three Types of Fiber and Carbon Black as an AC Additive To Resist Abrasion. Construction report. Office of Transportation Laboratory, California Department of Transportation, 1986.
- N. Predoehl. Modified Asphalt Binders for Stability Improvement. Construction evaluation research report. Office of Transportation Laboratory, California Department of Transportation, 1988.
- S. L. Cumbaa and H. R. Paul. Latex Modified Asphalt and Experimental Joint Treatments on Asphaltic Concrete Overlays. Report FHWA/LA-88/211. Louisiana Transportation Research Center, Louisiana Department of Transportation and Development, 1988.
- 4. H. R. Paul. Personal communication, 1991.
- T. Ravn, R. Cassellius, and R. Olson. Methods and Materials for Reducing Crack Reflectance. Report FHWA/MN/RD/05. Minnesota Department of Transportation, St. Paul, 1987.
- M. F. Lynch. Big Timber-Springdale Experimental Project. Performance Evaluation Report 5. Montana Department of Highways, Helena, 1989.
- R. I. Frascoia. Experimental Use of Resin Modified Asphalt Pavement on Montpelier State Highway. Report DTFH71-84-4503-VT-07. Vermont Agency of Transportation, Montpelier, 1987.
- 8. P. C. Winters. *Ultrapave Latex Modified Pavement on Vt. 12 Worcester*. Report 87-9. Materials and Research Division, Vermont Agency of Transportation, Montpelier, 1987.
- G. W. Maupin, Jr. Installation Report—Evaluation of Asphalt Additives. Report FHWA/VA-87/29. Virginia Transportation

- Research Council, Virginia Department of Transportation, Richmond, 1987.
- G. W. Maupin, Jr. Evaluation of Asphalt Additives To Resist Permanent Pavement Deformation. Virginia Transportation Research Council, Virginia Department of Transportation, Richmond, 1989.
- G. N. King, H. W. King, O. Harders, and P. Chaverot. Low Temperature Benefits of Polymer Modified Asphalt. *Proc.*, Canadian Technical Asphalt Association, Vol. 33, Calgary, Alberta, Canada, 1988.
- J. W. Button. Asphalt Additives in Thick Hot Mixed Asphalt Concrete Pavements. Research Report 187-18. Texas Transportation Institute, Texas A&M University, College Station, 1991.
- D. N. Little, J. W. Button, R. M. White, E. K. Ensley, Y. Kim, and S. J. Ahmed. *Investigation of Asphalt Additives*. Research Report FHWA/RD-87/001. Texas Transportation Institute, Texas A&M University, College Station, 1986.
- J. W. Button. Asphalt Additives in Highway Construction. Research Report FHWA/TX-87-187-14. Texas Transportation Institute, Texas A&M University, College Station, 1988.
- F. L. Roberts, R. L. Lytton, and D. Hoyt. Criteria for Asphalt-Rubber Concrete in Civil Airport Pavements (Vols. 1 and 2). Research Report DOT/FAA/PM-86/39. Texas Transportation Institute, Texas A&M University, College Station, 1986.
- S. Shuler. Specification Requirements for Asphalt-Rubber. In Transportation Research Record 843, TRB, National Research Council, Washington, D.C., 1982, pp. 1-4.
- D. Hoyt, R. L. Lytton, and F. L. Roberts. Performance Prediction and Cost-Effectiveness of Asphalt-Rubber Concrete in Airport Pavements. In *Transportation Research Record 1207*, TRB, National Research Council, Washington, D.C., 1988.

- D. S. Decker, D. F. Griffin, and J. P. Nielsen. An Evaluation of Asphalt Rubber Mixtures for Use in Pavement Systems. Report CEEDO-TR-79-02. Civil Engineering Research Facility, University of New Mexico, Albuquerque, 1979.
- 19. R. G. McKeen. Development of Criteria for Use of Asphalt-Rubber Mixtures in the Design, Construction and Maintenance of Airport Pavements. Air Force Contract F29601-84-C-0080. Civil Engineering Research Facility, University of New Mexico, Albuquerque, 1985.
- T. W. Kennedy, F. L. Roberts, and J. N. Anagnes. Texas Boiling Test for Evaluating Moisture Susceptibility of Asphalt Mixtures. Research Report 253-5. Center for Transportation Research, University of Texas, Austin, 1984.
- J. W. Button and L. Y. He. FTIR and Gravimetric Methods To Determine Polyethylene Content of Modified Asphalt. Research Report 0393-1F. Texas Transportation Institute, Texas A&M University, College Station, 1989.
- 22. A. E. Malek. The Determination of SBS Block Co-Polymer Concentration in Bitumen/Polymer Blends in Infra-Red Spectroscopy. Research Report AIR286-10. Australian Road Research Board, Vermont South, Victoria, Australia, 1984.
- J. W. Button and D. N. Little. Asphalt Additives for Increased Pavement Flexibility. Research Report FHWA/TX-87/471-2F. Texas Transportation Institute, Texas A&M University, College Station, 1987.

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Relating Asphalt Absorption to Properties of Asphalt Cement and Aggregate

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Mineral aggregates used in hot mix asphalt (HMA) mixtures have some porosity and tend to absorb some amount of asphalt cement. Several indirect and direct methods for estimating or determining asphalt absorption have been researched. However, there is a need to study the asphalt absorption phenomenon as related to the physical properties of the mineral aggregate and asphalt cement binder. Eight mineral aggregates of different absorptive characteristics, geologic origin, and mineral compositions were selected from the Strategic Highway Research Program Materials Reference Library (SHRP MRL). Four asphalt cements ranging from AC-5 to AC-30 grades were also obtained from SHRP MRL. A total of 96 HMA mixtures were prepared and tested for asphalt absorption. Physical properties of aggregates (including pore characteristics) and asphalt cements were determined. Generally, the asphalt absorption decreased with increase in viscosity (at the mixing temperature) of the asphalt cement. A high percentage of maltene (oil) fraction in asphalt cement is likely to increase the total asphalt absorption, possibly because of selective absorption. There appears to be a threshold pore diameter of 0.05 μm in the aggregate below which no appreciable asphalt absorption takes place. The most important pore size range affecting the asphalt absorption appears to be 0.05 to 0.1 µm.

Mineral aggregates used in hot mix asphalt (HMA) mixtures have some porosity and tend to absorb some amount of asphalt cement. Although some absorption may lead to improved strength in a compacted mixture through particle interlocking, the portion of the asphalt that is absorbed is no longer available as binder (1). The situation is further aggravated because of the time-dependent nature of the absorption phenomenon. Moreover, the asphalt available in thin films serving as a binder may have different physical, chemical, and rheological properties if selective absorption takes place (2). The absorption of asphalt in mineral aggregates may cause the following, which may lead to premature failure of the asphalt paving mixtures (1):

- 1. Incorrect computation of percent air voids, voids in mineral aggregate, or voids filled with asphalt (since one or more of these are generally used as criteria for asphalt mixture design, incorrect calculations may lead to mixtures lacking durability or stability);
- 2. Not having enough effective binder, which may lead to raveling, cracking, or stripping;
- 3. Possible premature age hardening and low temperature cracking as a result of changes in asphalt properties due to selective absorption; and
- 4. Construction problems such as segregation and tender mixes.

In view of these consequences, the absorption of asphalt by mineral aggregates needs to be studied. Often aggregates having relatively high asphalt absorption may have to be used either because high-quality aggregates become depleted at an ever-increasing rate or because geographic proximity predicates the use of such aggregates.

Most highway agencies use the Rice method (maximum theoretical specific gravity of voidless paving mixtures) in calculating the amount of asphalt absorbed. Other methods such as bulk impregnated specific gravity are also used. Also, some aggregates have been observed to continue to absorb asphalt with time during construction and the early life of an asphalt pavement. HMA mixtures that appear very rich during production can get leaner with time inducing premature raveling or cracking, or both (R. L. Dunning, personal communication, 1990). There is a need to study the asphalt absorption phenomenon as related to the properties of the mineral aggregate and asphalt cement binder.

REVIEW OF LITERATURE

Many investigators have attempted to evaluate asphalt absorption of aggregates through correlations with absorption using other liquids, of which kerosene has been the móst common. Hyeem (3) devised the centrifuge kerosene equivalent (CKE) test in 1942. The CKE is defined as the quantity of kerosene absorbed by 100 g of aggregate under specified conditions of soaking and centrifuging. It is a function of the surface area and absorptive capacity of the aggregate and has been used as a part of the design of bituminous mixtures by Hyeem method. Lohn (4) used a similar approach and correlated asphalt absorption of an aggregate with kerosene absorption. He studied the effects of factors such as saturation time, centrifuge force, and centrifuge time as well and finally adopted 10 min of saturation and 8 min of centrifuging at a force of 400 times gravity. Donaldson et al. (5) further proposed some modifications to the Hveem CKE method by increasing the soaking time to 30 min and by testing a nonabsorptive aggregate of the same gradation for comparison. Since kerosene has wetting properties similar to those of asphalt, it was believed to give a better representation as an absorption agent than water (5,6). A modified CKE procedure was developed by Cechetini (7), which can determine the surface areas and evaluate absorptive behavior of crushed aggregate mixes. Together with this information and the data characterizing the asphalt to be used, the amount of asphalt needed by an aggregate can be determined from a nomograph. Other investigators (8,9) have tried to use oils to evaluate the Kandhal and Khatri 77

absorptive capacity of coarse aggregates. However, because of the differences in wetting properties and viscosities of these liquids and asphalts, only approximate estimates could be made of the asphalt absorption of the aggregates.

In 1936, a procedure was described by Reagel (10) for determination of relative absorption of water and liquid bituminous material by the coarse aggregate using a water displacement method. It was recommended that absorption of liquid bituminous material be estimated at 75 percent of the water absorption.

In 1942, Goshorn and Williams (11) developed the immersion method. The aggregate is in contact with an unlimited supply of asphalt at relatively low viscosity for an extended time in this method. Therefore, the absorption is much higher than would be expected in actual HMA mixtures (12).

Rice (13,14) proposed a vacuum procedure to determine the maximum specific gravity of the voidless HMA mixture. In this method, the absorption of asphalt by aggregates is calculated from the maximum specific gravity of the mixture, the asphalt content, and the bulk specific gravity of the aggregate used in the HMA mixture. This method is now standardized as ASTM D2041.

A different approach was used by Larsen (15), who conducted a high pressure test instead of vacuum saturation to determine the effective specific gravity of the aggregate. Asphalt absorption was calculated as percent of the volume of aggregate and ranged from 26 to 88 percent of the water absorption.

The U.S. Army Corps of Engineers (16,17) developed and has used the bulk impregnated specific gravity in the design and control of bituminous mixtures. It is a function of the ratio of asphalt to water absorption, which varies widely but follows a definite pattern with different types of aggregates. The asphalt absorption can be found knowing the bulk impregnated specific gravity of the mixture, the bulk specific gravity of the aggregate, and the specific gravity of the asphalt used in the HMA mixture.

Absorption of asphalt by aggregates has also been determined by colorimetric analysis with photometer (12). The basic principle is that the amount of light absorbed by a given solution is proportional to the intensity of the incident light and to the concentration of the absorbing species in the path of the light beam. However, only solutions of light concentration can be used, and the mechanism of absorption of asphalt in solution is likely to be different from that in an HMA mixture.

Cross-sectional measurements have also been used to evaluate asphalt absorption (12). A compacted specimen of the HMA mixture is cut in halves by a diamond saw to expose the inner surface of the aggregates. The sample is then placed in an opaque projector and from its image projected on a screen, tracings are made of the external contours of the rock particles and lines of deepest asphalt penetration. The total area of a particle and area penetrated by the asphalt are measured by a planimeter, and the percentage of asphalt absorption is calculated. Limitations of this method are selective absorption of asphalt and different sizes of aggregate in a mixture.

More recently the methylene blue test has been used in Europe to indirectly measure the absorption/adsorption, surface area, cationic exchange capacity, soundness, and overall clay characteristics of aggregates. The test, first developed in France (18), uses methylene blue to quantify the absorption/ adsorption of an aggregate. The method has serious limitations: (a) a powdered aggregate is used, and (b) no asphalt cement is used.

Franco and Lee (19) have recently evaluated the viability of using an air meter for determining the maximum specific gravity of HMA mixtures. The air meter normally has been used for determining the percent of air entrained in portland cement concrete.

Thus, many methods of estimating or determining the amount of asphalt cement absorbed by the mineral aggregate have been researched. However, very few studies (20,21) have been conducted to determine the effect of the properties of mineral aggregate and asphalt cement binder on asphalt absorption.

OBJECTIVES

This research project was undertaken as a part of SHRP A-003B to study asphalt absorption as related to the physical properties of mineral aggregate and asphalt cement binder.

MATERIALS USED

All the materials used in this study were obtained from the SHRP MRL.

Aggregates

SHRP MRL has 11 different aggregates, 8 of which were used in this study. The aggregates used were RB, Watsonville granite; RC, McAdam limestone; RD, Frederick limestone; RF, glacial gravel; RG, commercial sandstone; RJ, Wyoming gravel; RK, Blue Mountain basalt; and RL, Texas Gulf Coast gravel. These aggregates were selected to include all SHRP core aggregates, to encompass the complete range of water absorption, and to include aggregates of different geologic origins and mineralogical composition. Only one size aggregate (i.e., passing ½-in. sieve and retained on ¾-in. sieve) was used. The bulk specific gravity and water absorption of the aggregates, as determined by ASTM C127, along with their code names are reported in Table 1. The core aggregates are also identified.

Asphalt Cements

The asphalt cements used in this study were AAB-2, Wyoming sour (AC-5); AAK-2, Boscan (AC-10); AAM-1, West Texas (AC-20); and AAK-1, Boscan (AC-30). Various properties of these four asphalt cements, as obtained from SHRP MRL, and their code names are given in Table 2. These asphalt cements were selected to encompass all viscosity graded asphalt cements commonly used for paving. All four were SHRP core asphalt cements.

TABLE 1 PROPERTIES OF AGGREGATES USED

Aggrega	ate Source	Bulk Specific Gravity	Water Absorption (% Wt. Agg.)
RB	Watsonville Granite	2.692	1.68
RC *	McAdam Limestone	2.485	2.88
RD *	Frederick Limestone	2.713	0.38
RF	Glacial Gravel	2.700	1.39
RG	Commercial Sandstone	2.660	0.49
RJ *	Wyoming Gravel	2.653	0.56
RK	Blue Mountain Basalt	2.828	1.73
RL *	Texas Gulf Coast Gravel	2.590	0.72

^{*} Core aggregate.

TABLE 2 PROPERTIES OF ASPHALT CEMENTS USED

		Asphal	t Cement	
Property	AAB-2	AAK-2	AAH-1	AAK-1
Grade	AC-5	AC-10	AC-20	AC-30
Original Asphalt Cement				
Specific Gravity	1.014	1.027	0.993	1.035
Viscosity at				
140 F, poise	403	996	1992	3256
275 F, cSt	193	320	569	562
Penetration, 0.1 mm (77 F, 100 g, 5 sec)	166	154	64	70
Ductility, cm (39.2 F, 1 cm/min)	81.0	150+	4.6	27.8
Softening Point (R&B), F	115	108	125	121
Component Analysis				
Asphaltenes (n-heptane)	16.7	20.5	3.9	21.1
Poler Aromatics	35.7	39.4	50.3	41.8
Naphthene Aromatics	36.5	30.6	41.9	30.0
Saturates	10.8	7.5	1.9	5.1
Element Analysis				
Nitrogen, %	0.50	0.70	0.50	0.80
Sulfur, %	5.40	6.90	2.40	6.60
Vanadium, ppm	163	1165	60	1427
Nickel, ppm	36	117	29	128
Thin Film Oven Test				
Mass Change, %	-0.0149	-1.2305	+0.0516	-0.5483
Viscosity of TFOT Residue at				
140 F, poise	1073	3098	3947	9708
275 F, cSt	263	533	744	930
Viscosity Ratio (140 F)	2.66	3.11	1.98	2.98

^{**} Determined by ASTM C127.

TESTING PLAN AND PROCEDURES

The study was divided into two phases. For Phase 1, a design consisting of 8 aggregates \times 4 asphalt cements (8 \times 4) was employed with 3 replicates for each treatment combination, giving a total of 96 tests. The replicates were considered as blocks, and the design was a completely randomized blocked design.

Phase 2 involved the determination of various properties of the eight aggregates and four asphalt cements. The various properties determined were viscosity (maltene viscosity) for asphalt cements and pore volume, pore diameter, pore size distribution, particle shape and texture, and water absorption for aggregates. Separation of asphalt cements into maltene and asphaltene fractions was done using ASTM D4124. Viscosity of the maltene fraction of asphalts was then determined using ASTM D2171. Pore volumes, pore diameters, and pore size distributions were determined through mercury porosimetry. Index of particle shape and texture was determined using ASTM D3398, and water absorption was determined using ASTM C127. In addition to the standard procedure, a variation was done in the determination of water absorption by subjecting the aggregates to a residual pressure of 30 mm of Hg under water for 10 min before soaking for 24 hr. During mercury porosimetry, mercury was injected in the pores of aggregate at increasing pressures, and the change in volume was measured. The pore radius, r, related to any pressure, p, based on cylindrical pores, was computed using the Washburn (22) equation:

$$r = -\frac{2\sigma\cos\theta}{p}$$

where σ is the surface tension of mercury and θ is the contact angle between aggregate and mercury. The data were then used to determine the cumulative porosity of the aggregate up to each pore diameter and its pore size distribution. Pores are not necessarily cylindrical in shape, but this assumption has to be made for simplicity.

No statistical experiment design was used for this phase.

DISCUSSION OF RESULTS

During Phase 1 of the study, the Rice method with 4 hr of aging in the oven at 290°F was used to determine asphalt absorption values for all the combinations of the eight SHRP MRL aggregates and four SHRP MRL asphalt cements in the study. Four hr aging was established by the authors in a previous SHRP study (23) to ensure that all potential asphalt absorption takes place before testing. Various asphalt cement and aggregate properties were also determined during Phase 2 of the study for developing correlations with asphalt absorption values.

The data obtained from the study are reported in Tables 3 and 4. Table 3 gives the asphalt absorption values as percent by weight of aggregate for the aggregate and asphalt combinations used during the study. Table 4 gives a summary of

the properties of aggregates used in the study. The properties reported include the bulk specific gravity and absorption data, results from mercury porosimetry, and the particle shape and texture index data as obtained using ASTM D3398. The 24hr soaked values were determined using ASTM C127. In addition, the aggregates were subjected to a residual pressure of 30 mm Hg under water for 10 min before running ASTM C127. These values are referred to as vacuum + 24-hr soaking in Table 4. The bulk specific gravity of the aggregates used was also determined using mercury porosimetry at atmospheric pressure. Water absorption values shown under mercury porosimetry were backcalculated using the cumulative porosity of aggregates and their bulk specific gravity as determined by mercury porosimetry. An analysis of variance (ANOVA) was conducted for the Phase 1 data presented in Table 3 and is reported in Table 5. It can be seen that both asphalt cements (A) and aggregates (B) significantly affect asphalt absorption at $\alpha = 0.05$. The interaction A \times B (asphalt cements × aggregates) is also found to be significant at $\alpha = 0.05$.

The variation of asphalt absorption values with the viscosity (of the original asphalt cements) at 290°F is shown in Figures 1 and 2. The asphalt absorption, in general, decreases with increase in viscosity of the asphalt cement used. However, the absorption for AAM-1 (AC-20) is somewhat higher than that for AAK-1 (AC-30), although viscosity (at 290°F) of AAM-1 is more than that of AAK-1.

It was suspected that the viscosity of the maltene (oil) fraction of AAM-1 might be lower than that of AAK-1, giving rise to selective absorption. Both of these asphalt cements were thus separated into asphaltene and maltene fractions using ASTM D4124. The maltene fractions thus obtained were tested for viscosity at 140°F. Three samples were tested for each asphalt cement. The results obtained are given in Table 6.

Table 6 indicates that the viscosity of the maltene fraction of AAM-1 is also higher than that of AAK-1 and hence does not explain why AAM-1 would be absorbed more than AAK-1. However, the maltene fraction of AAM-1 (96.1 percent) is much higher than that of AAK-1 (78.9 percent) as can be calculated from data reported in Table 2. This means that the maltene content of an asphalt cement may affect the amount of asphalt cement absorbed by the aggregate.

The asphalt absorption is also plotted against water absorption, as shown in Figure 3. There is a definite general relationship between the water and asphalt absorption values. There are some exceptions to the general trend, however. Aggregates RB and RK have almost the same water absorption; however, RK absorbs considerably less asphalt cement than RB. This can be explained by looking at the pore size distribution curves (Figures 4 and 5) for the two aggregates. Aggregate RB is seen to have a considerable amount of pores of large diameter, whereas RK has much smaller pores.

The particle shape and texture data as reported in Table 4 were also correlated with asphalt absorption values, but no relationships were noticed. Further investigation into correlations between material properties and asphalt absorption was conducted using mercury porosimetry data obtained for all the eight aggregates used in the study. These data are reported in summary form in Table 4. The asphalt absorption data were also transformed to percent by volume of aggre-

TABLE 3 ABSORPTION VALUES FROM PHASE 1 EXPERIMENTS (PERCENT BY WEIGHT OF AGGREGATES)

Asphalt Cement	AAB-2 Wyoming Sour (AC-5)	AAK-2 Boscan (AC-10)	AAM-1 West Texas (AC-20)	AAK-1 Boscer (AC-30)
Aggregate				
RB - Watsonville Granite	0.92	0.81	0.95	0.70
Water Abs. = 1.68%	0.89	0.71	0.84	0.6
Bulk S.G. = 2.692	0.69	0.66	0.81	0.7
Avg. =	0.83	0.73	0.87	0.7
RC - McAdam Limestone	1.16	0.68	0.95	0.63
Water Abs. = 2.88%	1.07	0.85	0.87	0.67
Bulk S.G. = 2.485	1.06	0.76	1.00	0.70
Avg. =	1.10	0.76	0.94	0.67
RD - Frederick Limestone	0.17	0.21	0.24	0.15
Water Abs. = 0.38%	0.19	0.21	0.20	0.12
Bulk S.G. = 2.713	0.15	0.11	0.15	0.18
Avg. =	0.17	0.18	0.20	0.15
RF - Glacial Gravel	0.37	0.70	0.37	0.43
Water Abs. = 1.39%	0.52	0.54	0.38	0.40
Bulk S.G. = 2.700	0.43	0.49	0.38	0.42
Avg. =	0.44	0.58	0.38	0.42
RG - Commercial Limestone	0.20	0.09	0.19	0.12
Water Abs. = 0.49%	0.13	0.09	0.12	0.11
Bulk S.G. = 2.660	0.13	0.06	0.08	0.09
Avg. =	0.15	0.08	0.13	0.11
RJ - Wyoming Gravel	0.18	0.14	0.18	0.03
Water Abs. = 0.56%	0.11	0.22	0.10	0.14
Bulk S.G. = 2.653	0.27	0.15	0.17	0.35
Avg. =	0.19	0.17	0.15	0.17
RK - Blue Mountain Basalt	0.04	0.09	0.07	0.04
Water Abs. = 1.73%	0.07	0.11	0.05	0.02
Bulk S.G. = 2.828	0.09	0.06	0.05	0.01
Avg. =	0.07	0.09	0.06	0.02
RL - Tx. G. Coast Gravel	0.23	0.10	0.13	0.21
Water Abs. = 0.72%	0.16	0.31	0.25	0.15
Bulk S.G. = 2.590	0.20	0.33	0.23	0.20
Avg. =	0.20	0.25	0.20	0.19

TABLE 4 AGGREGATE PROPERTIES FROM PHASE 2 EXPERIMENTS

		Bulk	Specific Gr	avity	Water Abs	corption (%	Wt. Agg.)	Median Pore D	ia. (Micron)	Index of
Agg.	Source	24-hr Soaking	Vacuum+ 24-hr Soaking	Mercury Porosimetry	24-hr Soaking	Vacuum+ 24-hr Soaking	Mercury Porosimetry	Based on Volume	Based on Surface Area	Particle Shape and Texture
RB	Watsonville Granite	2.692	2.709	2.759	1.68	1.58	0.76	0.054	0.006	14.6
RC	McAdam Limestone	2.485	2.494	2.467	2.88	2.95	3.40	0.178	0.015	13.2
RD	Frederick Limestone	2.713	2.722	2.899	0.38	0.28	0.29	0.013	0.006	15.2
RF	Glacial Gravel	2.700	2.707	2.638	1.39	1.46	1.87	0.119	0.007	12.9
RG	Commercial Limestone	2.660	2.659	2.662	0.49	0.49	0.31	0.010	0.006	17.8
RJ	Wyoming Gravel	2.653	2.650	2.640	0.56	0.61	0.71	0.026	0.005	12.7
RK	Blue Mountain Basalt	2.828	2.821	2.831	1.73	1.80	0.18	0.006	0.005	14.1
RL	Texas Gulf Coast Gravel	2.590	2.589	2.589	0.72	0.80	0.45	0.008	0.005	11.9

TABLE 5	ANOVA FOR	ABSORPTION	DATA	FROM:	PHASE 2
EXPERIM	ENTS				

Source	df	SS	MS	Fo	F _{Crit}
Total	95	57.784	-	-	-
Aggregates (A)	7	52.751	7.5358	265.4	2.2
Asphalt Cements (B)	3	0.757	0.2522	8.9	2.8
AxB	21	2.506	0.1193	4.2	1.8 *
Blocks (Replicates)	2	0.011	-	-	-
Error	62	1.760	0.0284	-	_

* significant at $\alpha = 0.05$.

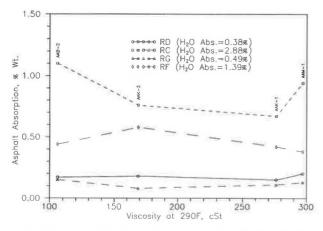


FIGURE 1 Asphalt absorption versus viscosity of asphalt cement (RD, RC, RG, and RF).

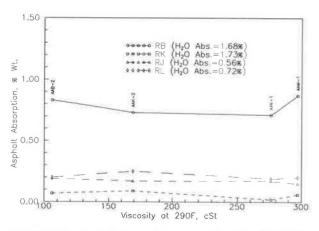


FIGURE 2 Asphalt absorption versus viscosity of asphalt cement (RB, RK, RJ, and RL).

TABLE 6 VISCOSITY OF MALTENE FRACTION

Asphalt Cement	AAK-1	AAM-1
Grade	AC-30	AC-20
Absolute Viscosity	91	1,218
of maltene fraction at	58	2,034
140 F, Poise	110	1,501
Average	86	1,584

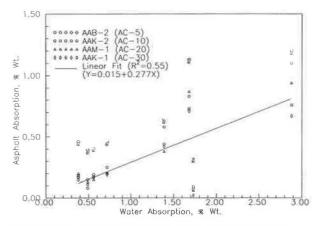


FIGURE 3 Asphalt absorption versus water absorption.

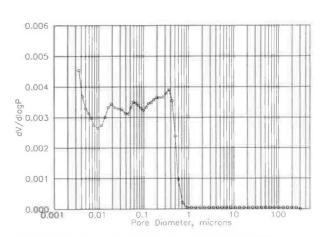


FIGURE 4 Pore size distribution for RB using Hg porosimetry.

gates. The mercury porosimetry data were further analyzed to obtain

- 1. Forward cumulative porosities (from start of intrusion) up to the pore sizes of 100, 50, 25, 10, 5, 2, 1, 0.5, 0.2, 0.1, 0.05, 0.01, and 0.0035 μm ;
- 2. Backward cumulative porosities (increasing pore size) up to the pore sizes of 0.01, 0.05, 0.1, 0.2, 0.5, 1, 2, 5, 10, 25, 50 and 100 μ m. Backward cumulatives were obtained to give an insight into a threshold pore size above which the

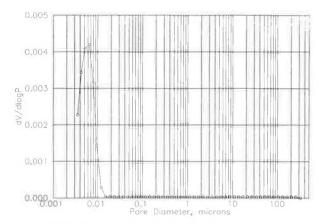


FIGURE 5 Pore size distribution for RK using Hg porosimetry.

value of R^2 suddenly jumps from a lower value to a substantially higher value; and

3. Porosities between the pore size ranges of 100 to 50, 50 to 25, 25 to 10, 10 to 5, 5 to 2, 2 to 1, 1 to 0.5, 0.5 to 0.2, 0.2 to 0.1, 0.1 to 0.05, 0.05 to 0.01, and 0.01 to 0.0035 μm . This would give information as to which pore size range is

more important in determining the amount of asphalt cement absorbed into aggregate pores.

The data are shown in Table 7. Correlations were run between these data and asphalt absorption values for all four asphalt cements used in the study to get a best fit model. It was found that a power relationship would fit the data the best. Results of correlations based on the power relationship are reported in Table 8. The following observations are made:

- 1. For forward cumulatives, the maximum values of coefficient of determination (R^2) were obtained for cumulative porosities up to the pore sizes of 0.01 μ m for all asphalt cements except AAK-2, for which it was obtained up to the pore size of 0.05 μ m. The values of R^2 ranged from 0.74 to 0.83
- 2. For backward cumulatives, the maximum values of R^2 were obtained for cumulative porosities up to the pore size of 0.5 μ m for all asphalt cements except AAK-1, for which it was obtained up to the pore size of 0.05 μ m. The values of R^2 ranged from 0.70 to 0.79. However, the jump in the value of R^2 for all asphalt cements occurred at a pore diameter of 0.05 μ m, indicating that this might be the threshold pore

TABLE 7 CUMULATIVE POROSITY AND POROSITY BETWEEN INDIVIDUAL PORE SIZE RANGES (PERCENT BY VOLUME OF AGGREGATE)

				P	ore Diamet	er (micron	s)						
Aggregate	100	50	25	10	5	2	1	0.5	0.2	0.1	0.05	0.01	0.0035
RB	0.0070	0.0111	0.0152	0.0203	0.0244	0.0300	0.0342	0.1104	0.5168	0.8014	1.0827	1.6890	2.1050
RC	0.0379	0.0583	0.0786	0.1059	0.1265	0.1537	0.3690	1.3336	3.9051	5.7957	6.9680	8.0500	8.3860
													0.851
RD	0.0010	0.0010	0.0020	0.0020	0.0030	0.0037	0.0040	0.0040	0.0050	0.0345	0.1218	0.5057	
RF	0.0301	0.0463	0.0630	0.0846	0.1012	0.1570	0.2957	0.5642	1.7511	2.6560	3.4259	4.3728	4.931
RG	0.0040	0.0060	0.0081	0.0111	0.0132	0.0162	0.0183	0.0210	0.0240	0.0264	0.0362	0.4257	0.830
RJ	0.1068	0.1301	0.1482	0.1961	0.2324	0.2878	0.3370	0.4048	0.5241	0.6273	0.7647	1.3031	1.876
RK	0.0052	0.0083	0.0113	0.0154	0.0174	0.0215	0.0245	0.0276	0.0316	0.0346	0.0377	0.0825	0.514
RL	0.0633	0.0964	0.1297	0.1737	0.2060	0.2500	0.2833	0.3166	0.3674	0.4056	0.4448	0.5514	1.171
b. Backward	Cumulativ	es (0.0035	micron up	to size i	ndicated)								
				F	ore Diamet	er (micror	ns)						
Aggregate	0.01	0.05	0.1	0.2	0.5	1	2	5	10	25	50	100	
RB	0.4160	1.0223	1.3036	1.5882	1.9946	2.0708	2.0750	2.0806	2.0847	2.0898	2.0939	2.0980	
	0.3360	1.4180	2.5903	4.4809	7.0524	8.0170		8.2595					
RC							8.2323		8.2801	8.3074		8.3481	
RD	0.3453	0.7292	0.8165	0.8460	0.8470	0.8470	0.8473	0.8480	0.8490	0.8490		0.8500	
RF	0.5582	1.5051	2.2750	3.1799	4.3668	4.6353	4.7740	4.8298	4.8464	4.8680		4.9009	
RG	0.4043	0.7938	0.8036	0.8060	0.8090	0.8117	0.8138	0.8168	0.8189	0.8219		0.8260	
RJ	0.5729	1.1113	1.2487	1.3519	1.4712	1.5390	1.5882	1.6436	1.6799	1.7278	1.7459	1.7692	
RK	0.4315	0.4763	0.4794	0.4824	0.4864	0.4895	0.4925	0.4966	0.4986	0.5027	0.5057	0.5088	
RL	0.6196	0.7262	0.7654	0.8036	0.8544	0.8877	0.9210	0.9650	0.9973	1.0413	1.0746	1.1077	
. Pore Vol	ume betwee	n Individu	al Sizes										
				Р	ore Diamet	er (micron	s)						
lggregate	100-50	50-25	25-10	10-5	5-2	2-1	1-0.5	0.5-0.2	0.2-0.1	0.1-0.05	0.05-0.010.0	1-0.0035	
RB	0.0041	0.0041	0.0051	0.0041	0.0056	0.00/3	0.0762	0.707	0.38/4	0 2017	0.4047	0 /160	
						0.0042		0.4064	0.2846	0.2813	0.6063	0.4160	
RC	0.0204	0.0203	0.0273	0.0206	0.0272	0.2153	0.9646	2.5715	1.8906	1.1723	1.0820	0.3360	
RD	0.0000	0.0010	0.0000	0.0010	0.0007	0.0003	0.0000	0.0010	0.0295	0.0873	0.3839	0.3453	
RF	0.0162	0.0167	0.0216	0.0166	0.0558	0.1387	0.2685	1.1869	0.9049	0.7699	0.9469	0.5582	
RG	0.0020	0.0021	0.0030	0.0021	0.0030	0.0021	0.0027	0.0030	0.0024	0.0098	0.3895	0.4043	
RJ	0.0233	0.0181	0.0479	0.0363	0.0554	0.0492	0.0678	0.1193	0.1032	0.1374	0.5384	0.5729	
RK	0.0031	0.0030	0.0041	0.0020	0.0041	0.0030	0.0031	0.0040	0.0030	0.0031	0.0448	0.4315	
RL	0.0331	0.0333	0.0440	0.0323	0.0440	0.0333	0.0333	0.0508	0.0382	0.0392		0.6196	

TABLE 8 CORRELATION OF ASPHALT ABSORPTION WITH MERCURY POROSIMETRY DATA

a. Forward Cumulatives													
Pore Dia (microns)	100	50	25	10	5	2	1	0.5	0.2	0.1	0.05	0.01	0.0035
. Asphalt Cement AAB-													
Intercept	1.388	1.322			1.256	1.208	1.235	1.279	1.115	1.061	0.969	0.709	0.443
Slope	0.161	0.163	0.185	0.175	0.187	0.189	0.217	0.278	0.294	0.366	0.407	0.576	0.851
R Sq.	0.078	0.086	0.094	0.092	0.098	0.102	0.165	0.354	0.537	0.681	0.743	0.813	0.762
. Asphalt Cement AAK-	2 (AC-10)												
Intercept	1.459	1.368	1.427	1.287	1.293	1.249	1.236	1.221	1.055	1.008	0.918	0.668	0.431
Slope	0.186	0.186	0.215	0.200	0.214	0.220	0.239	0.284	0.295	0.372	0.411	0.519	0.794
R Sq.	0.113	0.122	0.138	0.132	0.141	0.151	0.217	0.400	0.590	0.764	0.828	0.716	0.723
. Asphalt Cement AAM-	(AC-20)												
Intercept	1.105	1.069	1.110	1.034	1.047	1.016	1.056	1.133	1.020	0.987	0.911	0.672	0.429
Slope	0.120	0.122	0.144	0.134	0.146	0.148	0.178	0.247	0.271	0.350	0.396	0.560	0.811
R Sq.	0.042	0.047			0.058	0.061	0.107	0.270	0.443	0.601	0.680	0.742	0.670
. Asphalt Cement AAK-							01.0.	0.2.0	0.445	0.001	0.000	01142	0.0.0
Intercept	1.300	1.156	1.209	1.068	1.091	1.041	1.016	1.009	0.859	0.825	0.750	0.520	0.304
Slope	0.218	0.208			0.242	0.245	0.262	0.313	0.327	0.421	0.478	0.698	0.966
R Sq.	0.100	0.098			0.115	0.121	0.168	0.314	0.466	0.629	0.719	0.834	0.688
к эч.	0.100	0.098	0.111	0.103	0.115	0.121	0.100	0.314	0.400	0.029	0.719	0.034	0.000
. Backward Cumulative	5												
Pore Dia (microns)	0.01	0.05	0.1	0.	2	0.5	1	2	5	10	25	50	100
. Asphalt Cement AAB-													
Intercept	0.345	0.830	0.601	0.52		0.484	0.475	0.470	0.464	0.460	0.456	0.452	0.449
Slope	-0.876	1.964	1.396	1.07	6	0.893	0.857	0.848	0.848	0.849	0.850	0.852	0.852
R Sq.	0.049	0.671	0.754	0.77	4	0.789	0.786	0.781	0.777	0.776	0.773	0.772	
. Asphalt Cement AAK-	2 (AC-10)												
Intercept	0.568	0.769	0.575	0.50	18	0.470	0.462	0.457	0.452	0.448	0.443	0.440	0.437
Slope	-0.181	1.745	1.269	0.98	15	0.823	0.789	0.783	0.785	0.787	0.790	0.792	
R Sq.	0.002	0.577	0.678	0.70	16	0.729	0.727	0.725	0.725	0.726	0.726	0.727	0.727
. Asphalt Cement AAM-	1 (AC-20)												
Intercept	0.297	0.780	0.574	0.50	4	0.466	0.458	0.454	0.448	0.445	0.441	0.438	0.435
Slope	-0.996	1.861	1.333	1.02		0.854	0.819	0.809	0.809	0.810	0.811	0.812	
R Sq.	0.061	0.583	0.665	0.68		0.698	0.695	0.689	0.685	0.683	0.680	0.679	0.678
. Asphalt Cement AAK-			******		****				01002	0.000	0.000	010.7	0.070
Intercept	0.397	0.634	0.428	0.36	.0	0.338	0.331	0.327	0.323	0.319	0.315	0.313	0.310
Slope	-0.309	2.453	1.643	1.22		1.001	0.957	0.949	0.952	0.955	0.959	0.961	0.963
R Sq.	0.004	0.733	0.731	0.70		0.693	0.687	0.685	0.686	0.687	0.688	0.688	0.689
c. Pore Volume between	Individ	ual Sizes											
Pore diameter (microns	3) 100-50	50-25	25-10) 10	-5	5-2	2-1	1-0.5	0.5-0.2	0.2-0.1	0.1-0.05	0.05-0.0	0.01-0.003
1. Asphalt Cement AAB	2 (AC-5)												
Intercept	1.543					1.620	1.714	1.628	1.479	1.717	1.840	1.39	
Slope	0.152					0.187	0.203	0.220	0.261	0.334	0.401	0.66	-0.87
R Sq.	0.100					0.109	0.256	0.493	0.687	0.784	0.795	0.62	0.04
2. Asphalt Cement AAK						45.5-5					520 5.5		
Intercept	0.796		1.29	7 2.5	01	1.834	1.691	1.501	1.393	1.642	1.681	1.13	0.56
Slope	0.047					0.227	0.213	0.214	0.260	0.339	0.387	0.52	
R Sq.	0.011					0.175	0.307	0.507	0.742	0.879	0.808	0.41	
3. Asphalt Cement AAM			. 0.10	. 0.1	. ,	0.175	0.301	0.501	0.142	0.019	0.000	0.410	. 0.00
	1.213		7 1.036	5 1.8	17	1.278	1.415	1.425	1.346	1.609	1.738	1.29	0.29
Intercept													
Slope	0.116					0.146	0.172	0.199	0.247	0.330	0.399	0.647	
R Sq.	0.056		0.040	0.0	AD	0.064	0.177	0.391	0.593	0.739	0.763	0.570	0.06
4. Asphalt Cement AAK													
Intercept	1.238					1.550	1.392	1.265	1.177	1.468	1.679	1.22	
Slope	0.170	0.341	0.14	0.3	35	0.246	0.227	0.236	0.291	0.393	0.492	0.847	7 -0.30
R Sq.	0.087		0.07		20.00	0.133	0.224	0.397	0.596	0.758	0.841	0.70	0.00

diameter in determining whether asphalt cement will be absorbed into the pores of the aggregate or not.

3. For porosities in individual pore size ranges, the values of R^2 for the above asphalt cements peaked for porosities between 0.1 and 0.05 μm in general. For Asphalt Cement AAK-2, however, the maximum value of R^2 was obtained for the pore size range 0.2 to 0.1 μm . The range of R^2 values was from 0.76 to 0.88. The inferences drawn from the individual pore size ranges are in line with those obtained from the backward cumulative porosities. That is, there seems to be a threshold pore diameter of 0.05 μm below which no appreciable asphalt absorption takes place.

The aggregate particle shape and texture index data alone, as expected, do not have any correlation with the asphalt absorption data.

CONCLUSIONS

The main conclusion drawn from this study is that no single model can be developed using asphalt absorption data obtained on a limited number of asphalt cements and aggregates. Every asphalt cement has to be treated differently. However, some general trends are apparent.

- 1. Generally, the asphalt absorption decreases with increase in viscosity (at the mixing temperature) of the asphalt cement used. However, there is some indication that a high percentage of maltene (oil) fraction in an asphalt cement can increase its total absorption, possibly because of the phenomenon of selective absorption.
- 2. A definite general relationship between the water and asphalt absorption values was observed. Aggregates that absorb more water are likely to absorb more asphalt cement. However, an exception was noted in case of an aggregate that has much smaller pores and, therefore, absorbed significantly less asphalt cement compared with water.
- 3. Cumulative aggregate porosity up to $0.01~\mu m$ pore diameter has a good correlation with asphalt absorption. The R^2 values for different asphalt cements using a power fit range from 0.74 to 0.83.
- 4. There appears to be a threshold pore diameter of $0.05\,\mu m$ in the aggregate below which no appreciable asphalt absorption takes place.
- 5. The most important pore size range in the aggregate affecting the asphalt absorption appears to be 0.05 to 0.1 μm .

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REFERENCES

- D. Y. Lee, J. A. Guin, P. S. Kandhal, and R. L. Dunning. A Literature Review: Absorption of Asphalt into Porous Aggregates. Prepared for Strategic Highway Research Program Contract A-003B, 1990.
- P. S. Kandhal. Asphalt Absorption as Related to Pore Characteristics of Aggregates. Master's thesis. Iowa State University, Ames. 1969.
- F. N. Hveem. Use of Centrifuge Kerosene Equivalent as Applied To Determine the Required Oil Content for Dense Graded Bituminous Mixtures. *Proc. AAPT*, Vol. 13, 1942.
- R. N. Lohn. A Method to Determine Aggregate Absorption and Control of Bituminous-Aggregate Properties. *Proc. AAPT*, Vol. 16, 1947.
- J. A. Donaldson, R. J. Loomis, and L. C. Krchma. The Measurement of Aggregate Absorption. *Proc. AAPT*, Vol. 16, 1947.
- L. C. Krchma and H. G. Nevitt. Absorption of Liquid Bituminous Cement by Aggregates. Proc. AAPT, Vol. 13, 1942.

- J. A. Cechetini. Modified CKE [Centrifuge Kerosene Equivalent] Test. Proc. AAPT, Vol. 40, 1971.
- P. Kraemer. Doe Optimale Bindemitteldosierung der Bituminosen Meneralmassen im HeiBeinbau. Bitumen-Teeree-Asphalt-Peche und Verwandte Stoffe 2, 1966.
- L. A. Lattier, D. F. Fink, N. B. Wilson, and F. F. Fraley. Mechanism of Absorption of Bituminous Material by Aggregates. *Proc. AAPT*, Vol. 18, 1949.
- F. V. Reagel. Absorption of Liquid Bituminous Material by Coarse Aggregate. Highway Research Abstracts, No. 40, HRB, 1937.
- 11. J. H. Goshorn and F. M. Williams. Absorption of Bituminous Materials by Aggregates. *Proc. AAPT*, Vol. 13, 1942.
- 12. D. Y. Lee. Study of Absorptive Aggregates in Asphalt Paving Mixtures. Research Report HR-127. Iowa State University Engineering Research Institute, 1968.
- J. M. Rice. Maximum Specific Gravity of Bituminous Mixtures by Vacuum Saturation Procedure. ASTM Special Technical Publication 191, 1956.
- J. M. Rice. New Test Method for Direct Measurement of Maximum Density of Bituminous Mixtures. Crushed Stone Journal, Sept. 1953.
- O. Larsen. Development and Use of High Pressure Apparatus for Determining Voids in Compacted Bituminous Concrete Mixtures. Proc. AAPT, Vol. 26, 1957.
- W. C. Ricketts, J. C. Sprague, D. D. Tabb, and J. L. McRae. An Evaluation of the Specific Gravity of Aggregates for Use in Bituminous Mixtures. *Proc. ASTM*, Vol. 54, 1954.
- Investigation of the Penetration of Asphalt into Porous Aggregates as Related to and Affecting the Specific Gravity of the Aggregate. U.S. Army Corps of Engineers Waterways Experiment Station, Corps of Engineers Misc. Paper 4-88, 1954.
- 18. N. L. Tran. The Methylene Blue Test. Bulletin de Liaison des Ponts et Chaussees, 107, 1980.
- C. A. Franco and K. W. Lee. Development of a Pressure Method To Determine Theoretical Specific Gravity of Bituminous Paving Mixtures. In *Transportation Research Record* 1269, TRB, National Research Council, Washington, D.C., 1990.
- D. Y. Lee. The Relationship Between Physical and Chemical Properties of Aggregates and Their Asphalt Absorption. Proc. AAPT, Vol. 38, 1969.
- P. S. Kandhal and D. Y. Lee. Asphalt Absorption as Related to Pore Characteristics of Aggregates. In *Highway Research Record* 404, HRB, National Research Council, Washington, D.C., 1972.
- E. W. Washburn. Note on a Method of Determining the Distribution of Pore Size in a Porous Material. *Proc.*, *National Academy* of Sciences, Vol. 7, 1921.
- P. S. Kandhal and M. A. Khatri. Evaluation of Asphalt Absorption by Mineral Aggregates. *Proc. AAPT*, Vol. 60, 1991.

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Study of the Effectiveness of Styrene-Butadiene Rubber Latex in Hot Mix Asphalt Mixes

E. R. Brown, Frazier Parker, Jr., and Michael R. Smith

Many benefits are attributed to the use of styrene-butadiene rubber (SBR) latex in asphalt concrete pavements. These include decreased temperature susceptibility, increased rut resistance, and increased resistance to stripping. Potential benefits of SBR latex in hot mix asphalt mixtures were evaluated, and the results of the first 1½ years of a 5-year study are reported. Six existing test sites were identified and selected for evaluation. The test sites were located throughout Alabama, and each contained a control mixture and SBR latex modified mixture. Condition surveys were performed at each site to compare performance parameters such as rutting, transverse cracking, raveling, and bleeding. The Alabama Highway Department's pavement management data base provided additional data. The data base was investigated to compare performance of pavements with the department's 416 (control) and 417 (SBR latex modified) surface mixes. The parameters analyzed included mean (rut depth/sqrt ESAL), present serviceability index, friction number, condition rating, and transverse cracking. On the basis of preliminary results, no significant long term benefits can be attributed to the use of SBR latex in dense graded asphalt mixtures. However, further testing is required to verify the results.

Synthetic latexes have been used in asphalt pavements for a number of years. Evidence indicates that the use of synthetic latexes in surface treatments improves chip retention and results in improved performance. Synthetic latex has also been used with success on open-graded friction course projects to improve the adhesion of the asphalt cement to aggregate and reduce raveling. More recently, synthetic latex has been used in hot mix applications, and the reports to date indicate mixed performance. Sometimes the latex improves the performance of the mixture and sometimes it does not. Thus, it is not clear whether the increased cost of this additive is justifiable.

OBJECTIVES, SCOPE, AND PLAN

This study, the first of a two-phase research effort, was conducted to evaluate potential benefits from the use of styrene-butadiene rubber (SBR) latex in hot mix asphalt (HMA) mixtures. The cost-effectiveness of the use of SBR latex in Alabama will be determined at the end of Phase 2 and the completion of the study.

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Test plans and procedures used in this project were chosen to evaluate potential benefits from the use of SBR latex in HMA pavements. Condition surveys of six existing pavements modified with SBR latex were evaluated and their performance compared with that of appropriate control sections. Cores were taken from control and SBR latex—modified sections so that material and mixture properties could be characterized and evaluated on a rational basis. A laboratory investigation was performed to evaluate the effect of SBR latex on binder and mixture properties as well as design methodology. Finally, performance was assessed by comparing the pavement condition of the department's 417 (SBR latex—modified) mixes with that of 416 (control) surface mixes.

Condition surveys were performed at six test sites located throughout Alabama. The sites were evaluated for rutting, cracking, bleeding, and raveling. At Sites 1 through 4, 500-ft pavement test sections with control and SBR latex-modified mixes were selected for condition surveys. At Sites 5 and 6, 1,000-ft test sections from each mixture were selected for evaluation.

The Alabama Highway Department's pavement management data base was investigated to compare field performance of the department's 416 (control) and 417 (SBR latex—modified) mixes. The performance parameters analyzed included mean (rut depth/sqrt ESAL), present serviceability index, condition rating, friction resistance, and transverse cracking.

REVIEW OF LITERATURE

A variety of SBR latexes are in use today. They are distributed under a number of trade names. The Alabama Highway Department has had most experience with an anionic (negatively charged) latex developed for use in asphalt cements and asphalt emulsions.

Performance

Rutting, cracking, and stripping are principal concerns in asphalt pavement performance.

Lee and Demirel (1) compared the rut resistance of laboratory-prepared samples modified with various additives. For their comparison they used results from static uniaxial compression creep tests and the Shell pavement thickness design procedure (2) for a pavement structure. They found

that the addition of SBR latex to laboratory-prepared samples had no significant effect on rut resistance.

Epps et al. (3) analyzed the behavior of polyolefin and SBR latex-modified mixtures. They found that the addition of 3 percent latex improved the high temperature resilient modulus of laboratory-prepared specimens. Their conclusion, on the basis of this increase, was that SBR latex had the potential to reduce pavement rutting.

Button and Little (4) showed that blends of AC-5 with SBR latex respond with a creep compliance curve at 40°F, higher than an AC-20 control mixture. The more compliant nature of the SBR latex blend indicates a mixture better suited to resist thermal cracking. They used the modified Lottman moisture treatment to compare damage of specimens prepared with AC-5 and AR-1000 asphalts and a local river gravel. They found that the addition of SBR latex had little effect on moisture susceptibility.

Lee and Demirel (*I*) evaluated the effect of SBR latex on the resistance to moisture-induced damage by Marshall immersion (24 hr at 140°F) of asphalt concrete mixtures. In their analysis they used AC-5 and AC-20 grade binders (3 percent SBR latex and control) with gravel and limestone aggregate. They found that SBR latex slightly reduced the moisture resistance for gravel and limestone mixtures using the AC-5 binder, had no effect for the AC-20/gravel, and produced a slight improvement for the AC-20/limestone mixture.

CONSTRUCTION

The addition of SBR latex to asphalt cement increases viscosity. An increase in high temperature viscosity requires mixing at temperatures 30°F to 50°F above that for conventional HMA to achieve proper mixing and compaction. The higher mixing temperature may result in more emission problems than lower temperatures.

Blending of SBR latex and asphalt cement should be more efficient and consistent at the refinery than at the mixing plant. Latex blended with the asphalt cement at the refinery will prevent interruption of normal mixing operations at the plant. However, the heated storage life of the blend is somewhat limited since the enhanced properties of the modified binder may degrade with time (5).

A compatible blend of asphalt cement and SBR latex can be defined as a homogeneous mixture that neither separates during storage nor is altered chemically thereby diminishing the enhanced properties of the modified binder. If the blend is compatible, refinery blending ensures that the proper amount of SBR latex has been added to the asphalt cement and thoroughly dispersed in the blend.

SBR latex can be blended in-line with hot asphalt cement before injection into a drum mix plant. This method uses a cavity pump that continuously feeds and blends latex and asphalt cement in the proper proportions. The addition process is not entirely automated, and rates of flow of SBR latex must be changed to compensate for changes in plant production.

For batch plants, a double diaphragm pump is commonly used to inject the SBR latex directly into the pug mill. The addition of latex may alter required mixing time and temperature to achieve proper aggregate coating. The supplier

should be contacted for guidance in selecting dry and wet mixing times.

Compatibility of SBR Latex and Asphalt Cement

As described earlier, a compatible blend of asphalt cement and SBR latex is one that is homogeneous and neither separates nor is altered by chemical interaction during storage. If the SBR latex is preblended with the asphalt cement, as in the refinery blending process, compatibility of the blend must be verified.

Hazlett (6) showed that degradation of the physical properties of SBR latex-modified asphalt can result from prolonged storage at high temperatures. Bass (7) also found that compatibility of asphalt cement and SBR latex depends on asphalt source. Button and Little (4) found that prolonged hot storage of SBS/SB rubber-modified asphalt resulted in a significant decrease in viscosity, which later produced tender mixtures. Their laboratory data indicated that this could also occur with SBR latex.

Recycling Old Asphalt Pavements

With the depletion of the nation's basic road building materials, recycling asphalt pavements has proven to be cost-effective and environmentally sound. However, the use of rubber in asphalt pavements may present several design and recycling challenges.

Potential problem areas are in milling, stockpiling, mixture design, plant operations, laydown, and compaction. In the milling operation, the rubber may cause binding of the milling blades, and this could slow production. In the stockpiling, handling the material could potentially be more difficult if the rubber binds the fines together.

Currently, few laboratory data or construction records have been published regarding the recyclability of old asphalt pavements containing SBR latex. Because of the lack of information, a nationwide survey of highway departments was initiated to determine their experiences in recycling old asphalt pavements containing additives.

The survey was sent to the state highway departments and 44 responded. Of these, 41 had not tried to recycle old asphalt pavements with reclaimed mix modified with latex. A summary of the responses from the three states follows.

In the first state, a project containing 3 percent SBR latex additive in a friction course was milled and recycled in 1989. No problems were encountered during milling or production of the recycled mix. The very small amount of latex in the RAP did not appear to affect the rejuvenation of the RAP or cause any pollution problems.

In the second state, a section of pavement containing latex was milled but was not recycled. No problems were encountered during the milling operation.

In the third state, a section of pavement containing latex was milled and recycled. No problems were experienced during the milling or recycling.

Costs

The Alabama Highway Department experienced an approximate 14 percent increase in mix cost for construction of SBR

latex mixes in 1990. This implies that pavement performance must be increased by at least 14 percent (potentially greater because of inflation) for the additive to be cost-effective. Currently, no data are available concerning the cost-effectiveness of SBR latex-modified pavements.

FIELD INVESTIGATION

Six control and SBR latex-modified test sites, located throughout the state, were evaluated for rutting, transverse cracking, bleeding, and raveling to characterize the in-service performance of the Alabama Highway Department's 416 (control) and 417 (SBR latex-modified) surface mixes. All field measurements and observations were made in 1990.

Site Location Details

Each site was selected so that control and SBR latex-modified mixes were located end-to-end and placed at approximately the same time using the same materials. Figure 1 shows the location of these sites. Table 1 gives details for each site.

At Sites 1 through 4, 500-ft-long representative sections from the control and SBR latex-modified mixes were selected for condition surveys. At Sites 5 and 6, 1,000-ft-long sections were rated. Cores were taken from these last two sites so that a more detailed evaluation could be made of the material and mixture properties. Material and mixture properties determined from tests at Site 6 indicated that the control and SBR

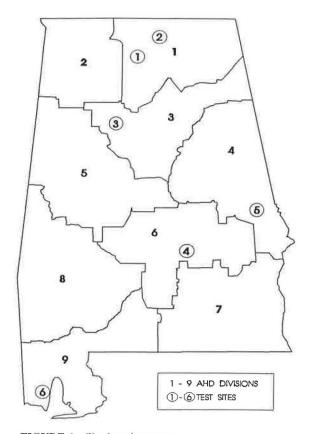


FIGURE 1 Site location map.

TABLE 1 SITE LOCATION DETAILS

Site	Route	Division	County	Mix' Type	Date Placed	Milepost Location	AADT
1	US-31	1	Limestone	416 417	7/89 9/88	366.02 364.00	10,870 12,850
2	US-231	1	Madison	416 417	7/89 5/88	311.00 313.00	41,070 45,700
3	AL-69	3	Walker	416 417	9/85 7/85	209.15 212.80	3,980 3,980
4	US-82	6	Autauga	416 417	8/89 8/89	141.05 142.00	8,500 8,500
5	267	4	Lee	416 417	7/87 1/87	2.34 1.95	5,112 5,112
6	AL-163	9	Mobile	416 417	6/85 6/85	27.00 25.00	8,730 8,730

¹⁴¹⁶ mix does not contain latex.

latex-modified mixtures at this site were not comparable, and data obtained from this site were not included in the analysis.

Rutting

Maximum rut depth measurements were made with a 12-ft straightedge in both inside and outside wheelpaths at 50-ft intervals. The average rut depths for each site are given in Tables 2 and 3.

A statistical analysis of the average rut depth is given in Table 4. In this analysis, the *t*-test was used to test the significance of the difference between rut depth means for each site at 95 percent confidence. The *F*-test was used to test the significance of the variance difference. If the sample variances were found to be significantly different, an alternate form of the *t*-test was used. The analysis indicates a statistically significant difference between rut depth in control and latex sections at Sites 1 through 3 but no difference at Sites 4 and 5. A complete description of the statistical procedure can be found elsewhere (8).

The average rut depths given in Tables 3 and 4 indicate that more rutting is occurring in three of the five control sections and in two of the five SBR latex-modified sections. Rutting in two of the control sections is significantly larger than rutting in the SBR latex-modified mixes. At Site 2 the rutting in the SBR latex-modified section is statistically greater than rutting in the control section. At Sites 4 and 5 there is no statistical difference in rutting of control and SBR latex-modified mixes.

Cracking

Cracking was quantified by counting the number of half- and full-width transverse cracks for each test section. The total number of full-width transverse cracks at each site is given in Table 5. For Site 1 slightly more transverse cracks were observed in the control section. For Site 3 many more transverse cracks were observed in the control section. No transverse cracks were observed at Sites 2, 4, or 5 in either section.

Minimal raveling was observed on the condition survey sites. However, the raveling that was surveyed was usually located at longitudinal joints. The amount of raveling noted between the control and SBR latex-modified pavements was not visually different.

TABLE 2 AVERAGE RUT DEPTHS FOR CONTROL SECTIONS

Site #	Number of Measurements	Inside Wheel Path	Outside Wheel Path	Average
1	11	0.12	0.20	0.16
2	11	0.06	0.02	0.04
3	11	0.14	0.14	0.14
4	11	0.05	0.05	0.05
5	21	0.11	0.09	0.10

TABLE 3 AVERAGE RUT DEPTHS FOR SBR LATEX-MODIFIED SECTIONS

Site #	Number of Measurements	Inside Wheel Path	Outside Wheel Path	Average
1	11	0.03	0.10	0.06
2	11	0.13	0.11	0.12
3	11	0.01	0.06	0.04
4	11	0.08	0.08	0.08
5	21	0.14	0.04	0.09

TABLE 4 STATISTICAL ANALYSIS OF DIFFERENCE IN MEAN RUT DEPTHS FOR CONDITION SURVEY SITES 1 THROUGH 5

		Significant	
Site #	_t_	_ltes l1_	Difference
1	-7.155	2.179	Yes
2	5.852	2.086	Yes
3	-5,632	2.086	Yes
4	1.860	2.086	No
5	-0.582	2.021	No

 $^{1}a = 0.05$

TABLE 5 NUMBER OF FULL-WIDTH TRANSVERSE CRACKS FOR CONDITION SURVEY TEST SITES 1 THROUGH 5

Site #	Control	Latex Modified
1	37.0	35.5
2	0.0	0.0
3	62.5	11.0
4	0.0	0.0
5	0.0	0.0

TABLE 6 AGGREGATE GRADATION DETERMINED FROM EXTRACTION OF CONTROL SECTIONS 1 THROUGH 5

	Site Number, Percent Passing									
Sieve Size	1	2	3	4	5					
11	100.0	100.0	100.0	100.0	100.0					
3/4"	100.0	100.0	100.0	100.0	100.0					
1/2"	94.4	92,7	98.1	94.3	96.2					
3/8"	89.9	84.5	90.0	82.2	85.7					
4	64.1	59.6	60.9	59.3	65,6					
8	48.1	42.3	45.4	43,4	48.8					
16	39.4	31,7	40.3	32.2	39.8					
30	30.8	25.5	36,9	23,2	28.7					
50	15.6	17.4	21.8	13.4	15,3					
100	9.1	10.3	9.1	7,8	8.0					
200	7,0	7.0	6,6	5,0	5,3					
Binder, %	5,69	5.87	5.31	4.91	5,51					

Mix Composition

Two extractions (ASTM D2172) were performed on samples from each of the control and SBR latex-modified sections for Sites 1 through 5. The gradations and binder contents are given in Tables 6 and 7.

Inspection of Tables 6 and 7 indicates more material passing the #100 and #200 sieves on all the control sections. This difference is probably caused by the rubber binding the smaller sized material and preventing removal during the extraction process. Considerable differences in binder content were also observed in the mixes from Sites 1, 2, and 4.

PAVEMENT MANAGEMENT DATA BASE ANALYSIS

In Alabama, distress measurements are made every 2 years in a statewide pavement condition survey on approximately 11,000 mi of pavement. These data have been compiled into a pavement management data base (PMD) by the Alabama Highway Department. The purpose of this section is to analyze and compare performance parameters surveyed for the department's 416 (control) and 417 (SBR latex-modified) surface mixes. The parameters investigated include mean (rut depth/sqrt ESAL), present serviceability index (PSI), condition rating, friction number, and transverse cracking. Much more 416 mix has been placed than 417 mix, and the sample sizes for the 417 mix may, in some cases, not be large enough to support definite conclusions.

In the PMD, a representative 200-ft sample section is selected within each lane mile of pavement. Since more rutting occurs in the outside lane, only those measurements in the outer lane were included in this analysis. Because of the extent of the data involved and the lack of 417 mixes on Interstate routes, only state route pavements rated in the 1988 data base (latest data in data base at the time the analysis was performed) were investigated.

Many variables influence the performance parameters investigated, such as amount of traffic, age of pavement, location of pavement within the state, and so forth. Therefore,

TABLE 7 AGGREGATE GRADATION DETERMINED FROM EXTRACTION OF SBR LATEX-MODIFIED MIXES, SECTIONS 1 THROUGH 5

_	Site Number, Percent Passing									
Sieve Size	1	2	3	4	5					
1*	100.0	100.0	100.0	100.0	100.0					
3/4"	100.0	100.0	100,0	100.0	100.0					
1/2"	91,2	93.6	96.8	95,4	98.5					
3/8"	82.7	85.7	88.0	81,0	0.88					
4	62,1	64,5	61.3	61.5	64.7					
8	47.6	74.5	47.2	45.3	56.3					
16	37.8	37.8	42.5	33.3	48.8					
30	26.1	29.8	39.5	23.8	35.2					
50	10.6	14.7	24.6	13.8	16.7					
100	5.3	6.2	8,0	7.7	7.4					
200	3.1	3,6	5.0	4.7	4.4					
Binder, %	6.24	5.62	5.21	5.24	5.57					

every effort was made to analyze the data so that a reasonable comparison could be made of the control and SBR latexmodified mixes. In this analysis the 416 and 417 mix performance data obtained from the state routes were sorted by division and year placed. A summary of performance data is given in Table 8, and the results of the statistical analysis of the data are given in Table 9.

In the PMD eight rut depth measurements are made in each lane within each 200-ft test section. Four of these measurements are from the inner wheelpath and four from the outer wheelpath. In this analysis the measurements from the outer wheelpath were averaged to yield the average rut depth. The average rut depth was then divided by the square root of the number of ESALs for the section of pavement being rated.

For mixes placed in 1985, the SBR latex-modified mixes statistically show an improvement in rut resistance in Divisions 2, 5, 6, and 7. However, in Divisions 3 and 4 the control mixes appeared to perform better. In Division 8 there was no statistical difference in mean (rut depth/sqrt ESAL).

For the mixes placed in 1986, there is a statistical difference in the mean (rut depth/sqrt ESAL) in Division 2, which showed

TABLE 8 SUMMARY OF PERFORMANCE PARAMETERS FOR CONTROL AND SBR LATEX-MODIFIED MIXES FOR DIVISIONS 1 THROUGH 9 AND OVERALL

Division	Year Placed	Mix Type	Rut Depth Sqrt ESAL (ln.)	PSI	Condition Rating	Transverse Cracking (No./200 ft.)
2	1985	Control Latex	2.2 E-04 1.1 E-04	3.70 3,44	83.76 72.85	1.96 14.15
3	1985	Control Latex	2.0 E-04 3.1 E-04	3.52 2.85	76.72 73.77	3.01 6.93
4	1985	Control Latex	2.5 E-04 5.0 E-04	3.41 3.74	71,34 76.61	10,40 3.33
5	1985	Control Latex	1.4 E-04 7.3 E-05	3.64 3.83	81.61 75.67	4.80 12.28
6	1985	Control Latex	2.7 E-04 2.3 E-04	3.72 3.86	82.66 84.33	2.71 1,08
7	1985	Control Latex	2.3 E-04 1.4 E-04	3.59 3.58	76.95 78.00	6.62 0.95
В	1985	Control Latex	2.9 E-04 3.3 E-04	3.62 3.31	81.26 78.42	0.81 4.61
9	1985	Control Latex	2.4 E-04 5.1 E-04	3.27 3.06	78.41 72.25	2.95 0.58
Overall	1985	Control Latex	2.3 E-04 2.5 E-04	3.55 3.44	78.00 76.35	5.09 6,56
2	1986	Control Latex	2,5 E-04 1,5 E-04	4.02 3.66	87.20 86.55	0.51 0.05
Overall	1986	Control Latex	2.4 E-04 1.5 E-04	3.60 3.66	80.93 86.55	3.31 0.05
1	1987	Control Latex	2.8 E-04 1.5 E-04	3.59 3.43	84.50 85.15	2.11 0.22
2	1987	Control Latex	3.0 E-04 2.3 E-04	3.64 3.44	86.36 84.94	0.02 0.67
4	1987	Control Latex	3.1 E-04 2.5 E-04	3.63 3.40	81.33 84.64	3.74 0.00
Overall	1987	Control Latex	3.0 E-04 1.9 E-04	3.52 3.44	82.15 85.02	2.54 0.48
1	1988	Control Latex	2.1 E-04 4.1 E-04	3.65 3.50	87.58 86.98	0.00 0.15
2	1988	Control Latex	1.8 E-03 7.6 E-04	3.08 3.56	80.50 85.67	1.50 0.00
3	1988	Control Latex	2.5 E-05 3.0 E-04	4.00 4.00	87.72 84.86	0.50 1.63
Overall	1988	Control Latex	4.3 E-04 5.3 E-04	3.78 3.64	86.68 84.94	0.66 0.73

NOTE: Divisions that did not place latex sections in a given year are not shown in the table.

TABLE 9 STATISTICAL ANALYSIS OF PERFORMANCE PARAMETERS FOR CONTROL AND SBR LATEX-MODIFIED MIXES

	Rutting	PSI	Condition Rating	Friction Number	Transverse Cracking
Division 1 1985 1986 1987 1988	* * L	* * C N	* * *	* N L	* * L N
Division 2 1985 1986 1987 1988	Z 	CCCL	0201	LXXC	CLCX
Division 3 1985 1986 1987 1988	C • • z	C * * N	C * + C	C • N	C * * C
Division 4 1985 1986 1987 1988	C • Z •	L · C ·	L L	C * L	
Division 5 1985 1986 1987 1988	L *	L * *	C *	C * *	c
Division 6 1985 1986 1987 1988	L *	L.	L •		:
Division 7 1985 1986 1987 1988	L * *	N •	N	N	:
Division 8 1985 1986 1987 1988	z • • • z	C •	c.	c •	C
Division 9 1985 1986 1987 1988	C···	C •	C • • •	c •	L * *
Overall 1985 1986 1987 1988	0 2	0200	0 0	C C C Z	C L L N

C - Control mixes performing better.

the SBR latex-modified mixes were performing better. For the mixes placed in 1987 the SBR latex-modified mixes were performing better than the control mixes in Divisions 1 and 2. No statistical difference was found in the mean (rut depth/ sqrt ESAL) for Division 4 in 1987. For the mixes placed in 1988, the control mixes were performing better in Division 1; however, in Divisions 2 and 3 the means were not statistically different.

The overall mean (rut depth/sqrt ESAL) for mixes placed between 1985 and 1988 indicates that for mixes placed in 1985 the overall mean is not statistically different between the control and the SBR latex-modified mixes. However, for the mixes placed in 1986 and 1987 the overall mean (rut depth/ sqrt ESAL) is statistically greater for the control mixes. For mixes placed in 1988 the overall mean (rut depth/sqrt ESAL) is statistically greater for the SBR latex-modified mixes.

 ⁻ SBR latex modified mixes performing better.
 N - Neither mix performing better than the other.
 * - SBR latex modified mixes not placed during the indicated year.

PSI

For pavements placed in 1985 the control mixes indicate higher PSI in Divisions 2, 3, 8, and 9. However, the SBR latex—modified mixes performed better in Divisions 4, 5, and 6. No significant difference was found for the mean PSI in Division 7. For those mixes placed in 1986, the control mixes show statistically better performance than the SBR latex—modified mixes in Division 2. For the mixes placed in 1987, the control mixes were performing better in Divisions 1, 2, and 4. For the mixes placed in 1988, the SBR latex—modified mixes were performing better in Division 2, whereas no significant difference was determined for the mean PSI in Divisions 1 and 3.

For mixes placed in 1985 the overall mean PSI is significantly greater for the control mixes. However, for mixes placed in 1986, no statistical difference was determined for the overall mean PSI. For the mixes placed in 1987 and 1988 the control mixes have a greater PSI than the SBR latex—modified mixes. Pavement PSI as used in the Alabama PMD system is a function only of ridability. The lower PSI values for latex-modified mixes may be due to increased placement and compaction difficulty.

Condition Rating

The pavement condition rating used by the Alabama Highway Department is primarily a function of distress variables with some influence from PSI. Higher numbers indicate better condition.

For mixes placed in 1985 the control mixes had a higher condition rating for Divisions 2, 3, 5, 8, and 9. The SBR latex-modified mixes had a higher condition rating in Divisions 4 and 6 for the mixes placed in 1985, whereas no statistical differences were determined for the mean condition rating in Division 7. For mixes placed in 1986, Division 2 shows no statistical difference in the mean condition rating. For mixes placed in 1987, the control mix was performing better in Division 2, whereas the SBR latex-modified mix was performing better in Division 4. No statistical difference was determined for the mean condition rating in Division 1 for 1987. For mixes placed in 1988, the control mixes had a higher condition rating for Division 3, whereas the SBR latexmodified mix had a higher condition rating in Division 2. No statistical difference in the mean condition rating was indicated in Division 1 for 1988.

For mixes placed in 1985 and 1988 the control mixes had a mean overall condition rating higher than the SBR latex—modified mixes. However, for mixes placed in 1986 and 1987 the overall condition ratings for the SBR latex—modified mixes were higher.

Friction Number

One of the primary functions of a flexible pavement is to provide good friction and steering qualities for the vehicle. The frictional qualities of a flexible pavement depend on the asphalt content and type of aggregate used in the mix. Friction

can be quantified by a friction number measured with a locked wheel skid trailer (ASTM E249).

Control mixes placed in 1985, 1986, and 1987 had an overall mean friction number higher than that for the SBR modified. However, for mixes placed in 1988 no statistical difference was determined in the mean friction numbers.

Transverse Cracking

In the PMD transverse cracking is divided into four severity levels beginning with hairline cracking. In this analysis the number of transverse cracks were totaled and then averaged per division.

For mixes placed in 1985 the control mixes had lower mean transverse cracking in Divisions 2, 3, 5, and 8. The SBR latex—modified mixes had a lower amount of transverse cracking in Divisions 4, 6, 7, and 9. For the mixes placed in 1986, in Division 2, the SBR latex—modified mixes had a lower average transverse cracking compared with the control mixes. For the mixes placed in 1987, the SBR latex—modified mixes had a lower mean transverse cracking count in Divisions 1 and 4. However, the control mixes had a lower mean in Division 2. For the mixes placed in 1988, the control mixes had a lower mean transverse cracking in Division 3, whereas there were no significant differences in Divisions 1 and 2.

For mixes placed in 1985 the mean overall transverse cracking is less for the control mixes. The SBR latex-modified mixes show less transverse cracking for 1986 and 1987. For mixes placed in 1988 there is no significant difference in the mean overall transverse cracking.

For the mixes placed in 1985, the control mixes appeared to perform better than the SBR latex-modified mixes (Table 8). For the mixes placed in 1986 and 1987 the SBR latex-modified mixes seemed to perform better overall. For the mixes placed in 1988, the control mixes performed better than the SBR latex-modified mixes. On the basis of these results the presence of SBR latex does not appear to significantly enhance the long-term performance of the pavements evaluated in this study.

CONCLUSIONS

The literature review indicated mixed performance in the use of SBR latex. Sometimes the use of this additive appears beneficial and sometimes it does not. The use of SBR latex increases the in-place mix cost. Alabama has experienced about a 14 percent increase in mix cost over that of a conventional mix.

The review of literature indicated that the compatibility of asphalt cement and SBR latex depends on the asphalt source. However, compatibility of the blend is more important if the SBR latex is added at the refinery. Rubber particles may settle out of the mixture during transportation to the job site if the blend is incompatible.

On the basis of results from the survey of state highway departments, there appears to be little experience with recycling old asphalt pavements that contain SBR latex. The effect on the recyclability of old asphalt pavements containing SBR latex is unclear at this time, but no significant problems have been identified.

The condition surveys indicate that for the three sites where the difference in rutting between the control and SBR latex—modified mixes was statistically significant, more rutting was occurring on two of the control sections and on one of the SBR latex—modified sections. The condition surveys indicated that more transverse cracking had occurred on the control sections at two sites and that no cracking had occurred in either section at the other three sites.

Analysis of the Alabama Highway Department's pavement management data base indicates mixed performance for the department's 416 (control) and 417 (SBR latex-modified) surface mixes. The use of SBR latex does not appear to consistently enhance mix performance, and in some cases it may adversely affect performance.

For the data analyzed thus far, the presence of SBR latex does not appear to enhance the long-term field performance of dense graded mixtures in Alabama. However, further study is required to establish these findings.

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REFERENCES

- D. Y. Lee and T. Demirel. Beneficial Effects of Selected Additives on Asphalt Cement Mixes. Iowa Department of Transportation, 1987.
- Shell Pavement Design Manual. Shell International Company Limited, London, 1978.
- 3. J. Epps et al. Factors Affecting the Behavior of Polyolefin and Latex Modified Asphalt Concrete Mixtures. (No date).
- J. W. Button and D. N. Little. Asphalt Additives for Increased Pavement Flexibility. Research Report 471-2F. Texas Transportation Institute.
- W. Siegel et al. Properties of Latex Modified Asphalt Concrete Mixtures. Presented at 67th Annual Meeting of the Transportation Research Board, Washington, D.C., 1988.
- D. G. Hazlett. Experimental Projects: Evaluation of Asphalt Latex Rubber Blends. Texas State Department of Highways and Public Transportation, 1983.
- D. A. Bass. Experiences with Latex in HMAC. Texas State Department of Highways and Public Transportation, 1985.
- 8. L. Blank. Statistical Procedures for Engineering, Management, and Science. McGraw-Hill, New York, 1980.

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Stability of Straight and Polymer-Modified Asphalts

JIM H. COLLINS AND MARK G. BOULDIN

Both short- and long-term aging can have a significant influence on the performance of hot mix asphalt pavements. This is true for polymer-modified asphalts as well as for conventional asphalt cements. The latter can undergo significant age hardening during storage, mixing, and placement in the field. This long-term aging can be dramatic and lead to premature pavement failure. The effects of aging can be exacerbated in the case of permeable pavements or in regions with extreme climatic conditions. Polymer modification is a way of overcoming these potential deficiencies by providing binders with well-balanced property sets. However, unsaturated polymers are susceptible to thermal and oxidative degradation. Thus, it is imperative to avoid prolonged storage and excessive temperatures during processing. Improved thermal and oxidative stability can be achieved by modifying asphalts with saturated polymers.

In recent years various studies have shown that polymer modification can be successful in obtaining asphalt binder systems with improved property sets. Improvements were noted in the binder properties, the properties of the hot mix asphalt (HMA), and in actual pavements in the field. In the case of permanent deformation Valkering et al. (1) found that in wheel tracking experiments the rutting rate at 40°C and 50°C could be significantly reduced by polymer modification. These results have been substantiated by Bouldin and Collins (2) with the TRRL wheel tracker for temperatures up to 60°C. Both repetitive and static creep experiments on HMA cores appear to correlate with these findings (1.3-5). Reports by Reese et al. (6) and Fleckenstein et al. (7) for example show that on heavily trafficked pavements dramatic improvements have been observed. In the case of thermal and fatigue cracking both Collins et al. (8) and Goodrich (9) have observed enhanced performance.

Such results have lead to widespread commercial use of polymer-modified asphalts. In some states, for example Nevada, polymer modification of asphalt is currently being specified for wearing courses on all major thruways.

How the long- and short-term stability of both conventional and polymer-modified asphalt influence their performance is discussed. The issues addressed are

- Asphalt cement aging,
- Polymer stability,
- Polymer-asphalt phase stability and microstructure, and
- How these influence the binder properties as well as the actual field performance.

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The way these materials are handled in the field (i.e., from initial polymer-asphalt blending to laydown and compaction) can have a significant impact on the previously mentioned issues. The first section therefore primarily addresses short-term aging encountered in these operations. In the second part, we discuss how long-term field aging can affect the pavement performance.

EXPERIMENTAL

Sample preparation, microscopy, rheological characterization, and traditional binder tests are described in detail elsewhere (10).

Gel Permeation Chromatography

A gel permeation chromatography (GPC) technique was used similar to that described by Portfolio and Fensel (II) to determine mean molecular weight (M_w) and molecular weight distribution. For the evaluation of field samples, the binders were extracted directly from the HMA with tetra-hydrofuran (THF). The effective amount of polymer is defined as

$$C_{\text{eff}} = \left[\frac{C_{\text{HM,aged}} \times 100}{(C_{\text{HM,aged}} + C_{\text{LM,aged}})} \right] \div \left[\frac{C_{\text{HM}}}{(C_{\text{HM}} + C_{\text{L,M}})} \right]$$

The unaged samples have therefore by definition an effective polymer concentration of 100 percent.

Materials

Tables 1 and 2 show the asphalts and polymers used in this study.

Low Temperature Thermal Cracking Test

We have devised a method to determine the critical cracking temperature (T_{cr}) of binder systems that is very similar to Hills' method (12). It is basically a visual method where T_{cr} is defined as the temperature at which the first crack in the asphalt is observed. A detailed description of the method is given by Collins et al. (8).

TABLE 1 ASPHALTS USED IN STUDY

Code	Asphalt Source	Asphalt Grade	Location
TX	W. Tx Intermediate	AC-10	TX HWY 287
WY	Wyoming	AC-10	WY I-80
NV06	California Valley	AR 1000	NV I-15
NV01	California Valley	AR 4000	NV RT 95
CA-N1	California Valley	AR 1000	CA I-40
CA-N2	California Valley	AR 4000	CA I-40
CA-C1	California Valley	AR 1000	CA RT 395
CA-C2	California Valley	AR 2000	CA RT 395
CA-S1	California Valley	AR 1000	CA Sacramento Intersection of Mack Rd. and Stockton Blvd.
CA-S2	California Valley	AR 8000	CA Sacramento Intersection of Mack Rd. and Stockton Blvd.
CV	California Valley	AR 4000	-
ET-1	East Texas	AC-5	:=:
ET-2	East Texas	AC-20	-
VEN-1	Venezuela	AC-7	-
VEN-2	Venezuela	AC-20	

Repetitive Creep Experiments

To determine the ability of a certain mix to resist permanent deformation, repetitive creep experiments were carried out. Contrary to some earlier work done by other groups (I), we did not apply a square wave but used a Haversine wave. The reasons for this are as follows:

- 1. It is easier to control this form of force application. The initial slope dF/dt is very small, and therefore significant force overshoots do not occur.
- 2. This pressure profile tends to mimic much more closely the pressures that a pavement will experience in the field (13).

The experiment is described elsewhere (14).

Short-Term Aging

In this section we would like to focus our interest on how the following processes may affect the performance of both straight and polymer-modified asphalts. These process steps are

- Blending of the polymer with asphalt,
- Storage of straight and polymer-modified asphalts, and
- Processing of the binder in a hot mix plant.

Asphalt Age Hardening

During the processing and storage of straight unmodified asphalt, one usually observes a so-called age hardening of the asphalt (15). Oxidation leads to more structured asphalts. In general this will result in both a higher asphaltene content and a higher average molecular weight (16–18). An example of age hardening is shown in Figure 1. The asphalt CA-S1 was stored over a period of 5 days at a temperature of 180°C (low shear agitation). As reflected in the penetration at 25°C and the penetration at 4°C the material becomes significantly harder with time. This effect is even more pronounced in crudes that are more susceptible to aging such as, for example, heavy Venezuelan crudes (9).

The most significant asphalt age hardening is normally observed when the asphalt is mixed with the aggregate in the

TABLE 2 POLYMERS USED IN STUDY

Code	Polymer Type	Oil Content	Trade Name and Grade
A	SBS	- 2	Shell KRATON® Rubber D1101
В	SBS	29%	D4141
С	SBS	50%	D4460X
D	(SB) _x	4	D1184
E	SEBS	960	G1657
F	SEBS		RP6904
G	SBR	70% solids (no Oil)	Goodyear Ultrapave® 70
Н	PE	97	Dow PE 2045

hot mix plant. Whereas exact temperatures are not available, the surface temperature of the aggregate can reach temperatures in excess of 260°C. Because the AC forms a thin layer on the aggregate, oxidation can be very significant.

Rolling thin film oven (RTFO) is a laboratory aging procedure (ASTM D2872) that is supposed to simulate the aging commonly observed in a hot mix plant (19). Some Western states have adopted this technique and grade all their asphalts according to the viscosity of the aged residue (AR grading system, ASTM D3381 Table III).

Polymer Stability

Under normal conditions, no significant changes in $M_{\scriptscriptstyle w}$ are observed during the blending process for SBS polymers. This has been established by comparing the molecular weight distribution of the neat polymer with the polymer in modified asphalt. The drop in $C_{\rm eff}$ due to mechanical and thermal conditions in the blending process is generally less than 4 to 5

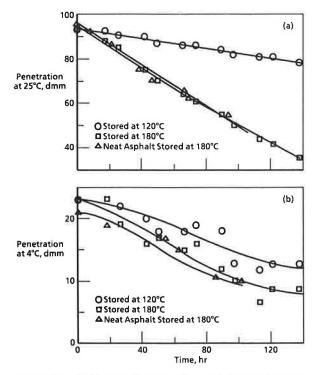


FIGURE 1 Influence of storage time and temperature on penetration (a, 25°C; b, 4°C) for 4 percent weight SBS (Polymer D) in CA-S1.

TABLE 3 GPC RESULTS ON POLYMER-MODIFIED BINDERS

Asphalt	Polymer	Hot Mix Plant Type	Conditioning	Ceff %
TX	3%w A	Drum	Original ²	89.3
1			RFTO	51.0
			loose mix ³	0,0
	6%w A	Drum	Original	70.6
			RTFO	40.1
			loose mix	14,8
WY	6%w C ¹	Drum	Loose mix	~100
			field aged sample	
			(after ~4 years)	87.5
NV01	6%w C1	Drum	Original	100
			RTFO	62,9
- 1			loose mix	89.9
			field aged sample	80.8
			(after ~3.5 years)	
CA-N1	D	Drum	Original	74.9
			CATOD	0.0
CA-S1	4%w F	Drum	Original	95.2
			RTFO	93.4
			CATOD	93.2
			loose mix	96.4

Actual neat polymer content.

Polymer/asphalt blend prior to use at hot mix plant.

Loose mix samples were taken prior to compaction at job site.

percent (compare Table 3). This is valid for both low and high shear mixing. However, sufficient temperature control is necessary to avoid excessive viscous dissipation during high shear mixing.

At the polymer levels used in standard paving applications, virtually only chain scission is observed. Gelation does not occur as shown in Figure 2 (no ultrahigh molecular weight species even after RTFO). A potential exception is when a concentrate is formulated that may contain up to 15 percent weight polymer. In these cases recombination and crosslinking can result in ultrahigh molecular weight species, and the latter may precipitate during solvent extraction.

A more likely source of polymer degradation is hot storage. It is not uncommon in the industry to hold asphalts over

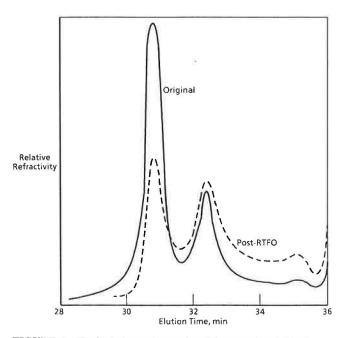


FIGURE 2 Typical chromatographs of 3 percent weight of SBS (Polymer A) in ET-1 before and after RTFO aging.

periods of weeks, and less frequently over months. If this is done with styrene-butadiene (unsaturated) polymers, one should take care to ensure that the storage temperature is well below 135°C to maximize polymer stability. Two laboratory samples were blended both containing 4 percent weight SBS. The first one was held at a temperature of 180°C (356°F), and the second sample was held at a more moderate temperature of 120°C (248°F). In both cases the samples were gently agitated and were exposed to air. As shown in Figure 3 at 120°C, the polymer stability is significantly enhanced relative to storage stability at 180°C. Stability can be further improved by blanketing with nitrogen.

This stability has a potentially significant impact on the resulting rheological properties of the asphalt blend, and thus its relative performance in the field. In general one would

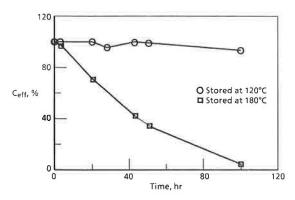


FIGURE 3 Degradation of 4 percent weight SBS (Polymer D) in CA-S1 as a function of storage time and temperature.

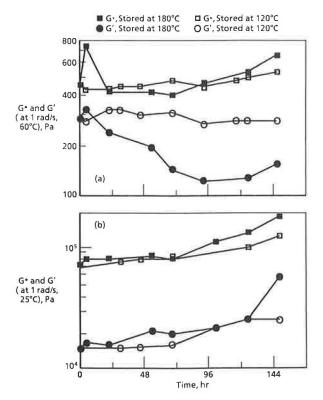


FIGURE 4 Change of storage and complex modulus as a function of storage time and storage temperature for 4 percent weight SBS (Polymer D) in CA-S1.

want a material that exhibits high resistance to deformation at elevated temperatures and that is not prone to exhibit cracking at low temperatures. A measure of the ability of a material to resist deformation is the complex modulus, G^* . The elasticity of a material is measured with the storage modulus, G', which indicates the portion of the energy that is restored elastically. The loss modulus, G'', is an indicator of the amount of energy that dissipates in the form of viscous flow (20). Straight unmodified asphalts are at elevated temperatures virtually only viscous,

G'' >> G'

 $G'' \sim G^*$

whereas polymer-modified asphalt may be even more elastic than viscous at high temperatures (2,10). The ideal binder should have a large value of G^* and G' at 60° C.

On the other hand, to prevent premature fatigue cracking G* at 25°C should be as low as possible (21-23). In Figure 4 the values of G* and G' are plotted as a function of storage time for the two samples of the asphalt CA-S1 containing 4 percent weight SBS (Polymer D). It is interesting to note that the sample stored at 180°C exhibits a rather significant drop of G' over the storage period, whereas the sample stored at 120°C shows a much smaller decrease in elasticity. Fragmentation of the SBS copolymer to lower molecular weight species thus results in a reduction of the material's ability to resist permanent deformation. By the same token the complex mod-

ulus of the material at 25°C increases when stored at 180°C. Hence, polymer-modified asphalt stored at an excessively high temperature of 180°C will be more susceptible to fatigue cracking and thermal cracking due to asphalt age hardening (high G*). Best overall property retention is achieved by storing the samples at temperatures not exceeding 120°C and by minimizing the duration of storage.

As previously mentioned, the RTFO is a standard testing technique to approximate what will happen to the material in the hot mix plant. In Table 3 a series of SBS and SEBS modified asphalts was compared to see whether RTFO is a good indicator for polymer stability. In general we find that the RTFO is more severe to the polymer than the "realworld" hot mix plant. In most cases RTFO significantly overpredicted polymer stability. This appears to be independent of the plant type, continuous or batch. In addition, saturated polymers (Polymers F and H) show virtually no change following RTFO, long-term storage, or hot mix plant processing. Thus we conclude that RTFO aging is probably only an indicator if particular polymer/asphalt systems are potentially unstable.

In the case of the SEBS modified asphalt, RTFO aging was more severe than that observed in the loose mix (compare Table 3, CA-S1 4 percent weight Polymer F). The degradation was, however, for SEBS insignificant and led actually to enhanced high temperature properties of the binder as shown in Table 4 (compare CA-S1 4 percent weight Polymer F). It is interesting to note that only one of the six SBS samples showed a decrease in elasticity at 60°C after RTFO. This reduction of G' is a direct result of polymer fragmentation.

TABLE 4 RHEOLOGICAL PROPERTIES OF BINDERS USED IN STUDY

Asphalt	Polymers	Conditioning	G* (25°C), @ 1 rad/s	G* (60°C), @ 1 rad/s	G' (25°C), @ 1 rad/s	G' (60°C), @ 1 rad/s	Repetitive Creep ² # Cycles to Failure	T _{cr} ,°C
TX	3%w A	Original	67,800	250	22,800	120		-37.2
		RTFO	213,000	460	88,600	77		-34.1
		loose mix	615,000	1,160	250,000	210	1900 ∓ 40	-33.2
	6%w A	Original	88,300	1,160	45,100	840		-47.5
		RTFO	294,400	1,930	176,500	1,130		-43.3
		loose mix	123,900	1,060	62,800	525	1740 + 85	-39.3
NV06	2.5%w D	Original	82,000	330	20,800	30		-39.8
		loose mix	245,200	710	113,800	108	2025 ∓ 240	-37.3
	3.8%w D	Original	100,000	670	29,500	365		-37.3
		loose mix	317,800	1,170	194,800	730	4480 ∓ 320	-37.0
CA-S1	4%w F	Original	114,700	570	24,500	430		-30.7
		RTFO	344,700	920	69,000	515		-25.6
		CATOD	3.8 × 10 ⁶	10,600	600,000	2,880		-23.4
		loose mix	220,000	1,050	54,000	700		-
CA-32	-	Original	697,000	420	21,600	~2		-18.3
(AR 8000)		RTFO	1.5 x 10 ⁶	1,065	35,000	11		-17.2
		CATOD	5 x 10 ⁶	14,300	1.3×10^{6}	1,590		-13.2
		loose mix	495,000	454	33,200	16		-20.4
CV	-	Original	266,400	245	6,100	1		-24.1
(AR 4000)		RTFO	681,000	495	263,000	3		-23.1
ET-2	-	Original	266,500	270	28,000	~1		-25
(AC-20)		RTFO	496,000	463	46,800	5		-24.9
VEN-1	-	Original	34,500	94	15,180	~1		-41
(AC-7)		RTFO	2 3000000					-41.5
VEN-2	-	Original	84,150	190	27,500	11	200	
(AC-20)		RTFO	194,000	571	87,500	40		-34
CA-S1	-	Original	46,300	75	1,140	~0.01		-30.2
(AR 1000)		RTFO	99,200	125	5,070	~0.5		

¹Loose mix samples were taken prior to compaction at job site.

²Repetitive creep on mix; all other tests on binders.

Asphalt age hardening, on the other hand, generally gives an increase in G^* (and a decrease of the penetration). In all the loose mix samples in this study no reduction of G' or G^* was observed, again indicating that RTFO is more severe than the actual processing.

Case Histories—Short-Term Aging

Texas Highway 287

In spring 1989 the Texas State Department of Highways and Public Transportation, District 25, constructed HMA test sections using various polymer-modified asphalts on HWY 287 near Memphis, Texas. Two test sections were put down containing 3 percent weight and 6 percent weight SBS (Polymer A). The base asphalt was given in Table 1, an AC-10 (TX). However, in the case of the binder containing 6 percent weight polymer, an extender oil was added. Hence, the resulting base asphalt was, according to Table 5 (compare TX 6 percent

weight Polymer A 4°C pen), relatively soft and resembled more an AC-5.

Infrared pyrometer measurements on the 3 percent weight Polymer A section taken on the mat directly behind the paver gave excessively high temperatures, ~180°C. Normal mat temperatures should be in the neighborhood of 150°C or lower. The results of the excessively high temperatures can be seen in the material properties. In Table 4 compare the values of both the complex modulus and the storage modulus of the original binder with those of the binder recovered from the loose mix and the more moderate increases due to RTFO. The increase in G* at 25°C is almost an order of magnitude, from 67,800 to 615,000 Pa. This value is actually higher than values typically found for an AC-30 grade asphalt (i.e., the asphalt had dramatically age hardened). As one may expect under these severe conditions the polymer/asphalt blend was largely degraded. No triblock material could be detected by GPC for the blend containing 3 percent weight SBS. For the blend containing 6 percent weight SBS serious stability problems were observed and $C_{\mbox{\scriptsize eff}}$ was reduced to 14.8 percent. In

TABLE 5 PROPERTIES OF BINDERS USED IN STUDY (CLASSICAL TESTS)

Asphalt	Polymers	Conditioning	Penetration, dmm 100g, 5s @25°C	Penetration, dmm 200g, 60s @4°C	Absolute Viscosity Pa.s @ 60°C	Kinematic Viscosity cm ² /s @135°C
TX	3%w A	Original	66	21	840	6.6
		RTFO	32	13	910	8.7
		loose mix	35	13	1,600	9.7
	6%w A	Original	90	31	no flow	12
	0,0 41 11	RTFO	47	24	no flow	12.3
		loose mix	51	25	3,750 @ 0.4s ⁻¹	12.3
NV06	2.5%w D	Original	64	18	391	8.5
14400	2.0 % D	loose mix	44	19	854	7.5
	0.00 70		100	1000	500.07	
	3.8%w D	Original	56 22	18 9	25,840 @ 0.05s ⁻¹	12,1
		loose mix			5,448 @ 0.09s ⁻¹	3.6
CA-S1	-	Original	136	35	55	1.4
(AR 1000)		RTFO	95	25	92	1.7
		CATOD	11	1.7	5,000	-
CA-S2	-	Original	29	5	450	3.5
(AR 8000)		RTFO	19	4	866	4.6
	1	CATOD	5	1	14,000	20
		loose mix	37	12	600	4.2
CV	-	Original	45	13	237	2.8
(AR 4000)		RTFO	31	7	441	3.2
ET-2	-	Original	50	14	209	3.4
		RTFO	28	7	443	5.3
CA-S1	4%w F	original	61	17	6,570	11.1
		RTFO	42	14	13,850	14.5
		CATOD	9	3	17,860	41.9
		loose mix	48	16	6,840	16.5
CA-N2*	=	Original	48	11	172	2.6
(AR 4000)		RTFO	32	10	388	3.65
15-15-5-5-5		CATOD	10	_	9,840	13.4
CA-N1*	D	Original	248	114	185	2.7
	_	RTFO	201	70	212	2.38
		CATOD	40	-	1,000	5.9
	E	Original	126	45	196	_
	"	RTFO	85	28	344	
		CATOD	36	-	1,200	2
CA-C2*	-	Original	75	20	124	1.8
(AR 2000)		RTFO	50	11	226	2.6
(AR 2000)		CATOD	12	-	3,030	7.7
CA-C1*	E					
CM+C1.	-	Original	145	43	143	5.1
		RTFO	103	30	223	6
		CATOD	38	-	910	7.9

¹Loose mix samples were taken prior to compaction at job site.

^{*}Data taken from reference 11.

this case RTFO predicted a significantly higher degree of polymer stability (Table 3). However, note that the absolute amount of degradation is nonetheless unusually high in this asphalt. This implies that certain asphalts lead to less stable polymer/asphalt binder systems. Hence, RTFO coupled with GPC and rheology is a good tool in predicting polymer stability.

As indicated in Table 4, the results for repetitive creep are actually slightly higher for the binder system with less polymer. The reason for this is that polymer content has virtually no influence on the performance because of the high degree of degradation in both instances. Moreover, the base asphalt stiffness is in these cases the overriding factor.

On the other hand, premature rutting could have occurred if the polymer degradation had not been accompanied by significant asphalt age hardening. Therefore it is of particular importance to ensure polymer integrity when modifying soft base asphalts with low levels of polymers.

Nevada I-15

In 1989 the Nevada Department of Transportation (NDOT) placed a series of polymer-modified asphalts on I-15 near Mesquite, Nevada. The department was seeking improvements in resistance to permanent deformation and long-term pavement durability, which are common problems throughout the deserts of the southwestern United States. In these regions the average daily high temperatures of the hottest month exceed 40°C, and pavement temperatures up to 80°C have been measured by NDOT (L. Quilici, personal communication).

The asphalt binder system consisted of a California Valley AR-1000 (soft base; NV06) modified with Polymer D at 2.5 percent weight and 3.8 percent weight SBS. The binders exhibit very high elasticity (G') and resistance to deformation (G*) at high temperatures. In addition, low temperature properties are outstanding. This is reflected in the relatively low values for G* at 25°C (soft material is less susceptible to fatigue cracking) and the very low critical cracking temperatures. This best balance of properties results from using a soft base asphalt and sufficient polymer to establish a polymeric network. The effectivity of this network formation is demonstrated by the exceptionally high numbers of loadings until failure in repetitive creep of the HMA (Table 4). Increasing the polymer concentration from 2.5 percent weight to 3.8 percent weight more than doubles the resistance to permanent deformation of the HMA. A more detailed description of how the rheology of these binders influences their performance is given elsewhere (2,14).

Sacramento Mack Road/Stockton Blvd.

The city of Sacramento was concerned about the severe shoving and rutting at the intersection of Mack Rd. and Stockton Blvd. To evaluate how an SEBS modified asphalt would perform in comparison with a very hard AR-8000 (CA-S2) two pavements were laid down in October 1990.

Virtually no degradation was observed in the original, the RTFO aged residue and in the extracted binder (Table 3). In general saturated polymers are relatively immune from degradation problems. Therefore, plant operating windows can be much wider. Yet, good handling practices are still imperative to avoid the consequences of serious asphalt age hardening.

With regard to the mechanical properties of the binder systems, the polymer-modified asphalt exhibits superior performance at both high and low temperatures (i.e., higher values of both G* and G' at 60°C and lower values at 25°C). The critical cracking temperature of the SEBS modified material is significantly lower than that of the AR 8000. The pavement is still too new to draw any conclusions.

Long-Term Aging

Following laydown and compaction there has to be concern with the long-term durability of the pavement and how this affects the performance of the HMA. This aging process is exacerbated by the severe desertlike climates that prevail throughout large areas of the southwestern United States.

In the case of straight unmodified asphalts the experience in these areas has been that ACs embrittle within 2 to 4 years. Subsequently the pavements fail because of cracking. In permeable mixes, for example open-graded friction courses or gap-graded mixes, aging may be of concern even in moderate climatic regions. A possible avenue in obtaining long-term aging resistant binder systems is polymer modification. Polymer modification allows the use of soft base asphalts, and, therefore, the bitumen itself will not govern the viscoelastic behavior of the blend. Moreover, the polymeric network is the determining factor. Therefore, polymer stability is of critical concern.

Various attempts have been made to devise laboratory simulations for long-term aging. A detailed evaluation of simulated age hardening of asphalt mixtures has been published by Chollar et al. (17) and Chari et al. (24). Chari reported on the effects of UV and elevated temperatures on the aging rate of a number of straight asphalts. More recently the Strategic Highway Research Program (SHRP) has been evaluating the so-called pressure air vessel (PAV) method (25,26). However, to our knowledge this work has not been extended to include polymer-modified systems. Also further field validation is required.

A simple and straighforward technique to simulate long-term field aging (as it occurs in desert regions) has been developed by Kemp and Predoehl at Caltrans (27). This test uses the standard rolling thin film oven to age the asphalt at a temperature of 111°C for 7 days. The procedure of this so-called California Tilt Oven Durability Test (CATOD) is given in California Test Method 374. Both Kemp and Predoehl (27) and Reese and Predoehl (6) have been successful in correlating field data with CATOD laboratory results. There is insufficient data currently available to determine the general applicability of this test to other climatic regions. At present polymer modified asphalts are being evaluated using this technique.

Polymer Stability

As in the case of short-term aging polymer integrity is to be considered in long-term aging. Here also we find GPC to be

an excellent tool to track the molecular weight and molecular weight distribution of the block copolymers through time. Two samples were taken from roads that were modified with SBS and are located in Nevada (NV-01) and Wyoming (WY). Both pavements were placed in relatively moderate climates. After 3 to 4 years in service we found effective polymer contents of 80.8 percent and 87.5 percent, respectively (Table 3). Thus, one can expect a continued contribution of the polymer to the binder performance over an extended period of time.

We also studied the stability of SEBS (Polymer F), which because of the saturation of the rubber midblock is expected to give superior long-term performance. Using the previously mentioned CATOD, samples of the binders used in the Sacramento job (CA-S1 with 4 percent weight Polymer F and CA-S2) were aged. Virtually no polymer degradation was found (Table 3). In the case of the straight CA-S2 (AR 8000) we found an extreme increase of the moduli (Tables 4 and 5). This implies that after some years of service, this pavement is expected to embrittle and exhibit fatigue and thermal cracking. After CATOD aging, the modified material is significantly less brittle and therefore should not experience fatigue and thermal cracking (in this climatic region). Where extreme long-term stability is required saturated polymers such as Polymer F give better results.

Case Histories-Long-Term Aging

In 1987 Caltrans embarked on a program to find improved binders for desertlike conditions (16). Some examples will be discussed.

California Interstate 40

For this evaluation Caltrans placed a series of test sections near Needles, California, on I-40. Needles has a typical low desert climate with an average daily high during the hottest month of 41.7°C and an average low during the coldest month of 0°C. As a control, an AR 4000 (CA-N2) was placed (Figure 5). Three polymer-modified sections were placed in the west-bound travel lane, which were separated by control sections. The polymers compared were Polymers D, E, and G. The base asphalt was AR 1000 in each case. However, as shown in Table 5, the low temperature pens indicate that varying amounts of extender oils were used.

To simulate the aging the binders would experience over a period of 4 years in the field the samples were CATOD aged and subsequently rheologically characterized. The results are given in Table 6. On the basis of these results, fatigue cracking would be expected to occur with the binders exhibiting the higher moduli at 25°C. On a relative basis one would therefore predict the following resistance to reflective and fatigue cracking:

$CA-N1/D \sim CA-N1/E > CA-N1/G >> CA-N2$

A measure of the binder's ability to mitigate permanent deformation is G^* and G' at 60° C. The largest potential for rutting is directly after placement before the binder has age hardened. Therefore, it is better to look at the moduli of the unaged material. All materials have a value of G^* in the range between 200 and 360 Pa. However, the unmodified asphalt CA-N2 has virtually no elasticity ($G' \sim 2$) at 60° C and, hence, should be much more susceptible to permanent deformation. On a relative basis one would therefore predict the following resistance to permanent deformation:

$$CA-N1/G \sim CA-N1/D \sim CA-N1/E >> CA-N2$$

A summary of the actual field performance is shown in Figure 5. All the control sections are showing reflective and wheelpath cracking. As predicted the controls in the heavily trafficked driving lane are also showing rutting and bleeding (28). All polymer-modified sections have shown excellent resistance to permanent deformation. Only in the CA-N1/G section has reflective cracking been observed. Thermal cracking has not been detected in any of the sections. The field performance confirms the predictions offered on the basis of the rheological findings on the CATOD aged samples.

California U.S. Route 395

A cold climate test section was placed on U.S. Rt. 395 at Crestview, approximately 50 mi north of Bishop, California. This test section does not experience the high temperatures that the Needles test sections receive. The average daily high for the hottest month is 32°C, according to Caltrans. The winters are colder with an average low for the coldest month of -15°C.

As a control an AR 2000 (CA-C2) was chosen. The polymer-modified system was AR 1000 (CA-C1) containing Polymer

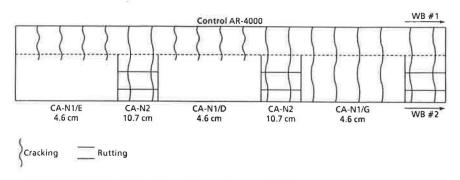


FIGURE 5 Needles test section, I-40 (status April 1991).

TABLE 6	RHEOLOGICAL PROPERTIES OF BINDERS
USED IN	STUDY FOR LONG-TERM AGING

Asphalt	Polymers	Conditioning	G* (25°C), @ 1 rad/s	G* (60°C), @ 1 rad/s	G' (25°C), @ 1 rad/s	G' (60°C), @ 1 rad/s	T _{cr} , °C
CA-N2 (AR 4000)	-	Original CATOD	362,000 3.6 x 10 ⁶	235 13,900	134,000 25 x 10 ⁶	-2 2950	-24.4 -16.5
CA-N1	D	Original	8,300	210	3,260	170	-
	E	CATOD Original	530,000 36,200	2,180 300	360,000 13,900	610 75	-34.6
	G	CATOD Original CATOD	570,000 122,700 960,000	1,600 364 7,180	155,000 36,900 785,000	300 50 2,040	-37.6 - -35.6
CA-C2 (AR 2000)	-	Original CATOD	223,400 3.6 x 10 ⁶	260 6,350	162,000 2.3 × 10 ⁶	~1 530	-29.5 -19.4
CA-C1	E	Original CATOD	27,100 630,000	180 1,210	8,300 300,000	22 140	-36.9 -30.9

E. As shown in Table 5, the system contained a significant amount of extender oils, which is reflected in the very high 4°C pen.

Because of the low traffic volume and the fact that it does not experience excessively high pavement temperatures, fatigue and rutting were not expected. However, thermal cracking should occur in the CA-C2 which, according to Table 6, has a critical cracking temperature of -19.4° C after CATOD aging. The polymer-modified system has a T_{cr} of -30.9° C after CATOD aging and, therefore, should not exhibit thermal cracking.

The field observations agree very well with the laboratory findings. The control has already shown thermal cracking after the exceptionally cold winter of 1990–1991. The polymer-modified test section is not showing distress.

CONCLUSIONS

Both short- and long-term aging can have a significant influence on the performance of HMA. This is true for polymer-modified asphalts as well as for conventional ACs. The latter can undergo significant age hardening during storage, mixing, and in the field. This long-term aging can be dramatic and lead to premature pavement failure. The effects of aging can be exacerbated in the case of permeable pavements or in regions with extreme climatic conditions.

Polymer modification is a viable route in overcoming these potential deficiencies by providing binders with well-balanced property sets. This is generally achieved by modifying soft, compatible asphalts. Improvements are noted in both the binder's resistance to permanent deformation and its ability to mitigate thermal and fatigue cracking. However, unsaturated polymers may exhibit thermal and oxidative instabilities. Thus, it is imperative to avoid prolonged storage and excessive temperatures. This is especially important in hot mix plant operations. GPC and dynamic mechanical analysis are excellent tools in evaluating the stability of polymer (SBS) modified asphalts. Likewise, RTFO can be useful in indicating potentially unstable polymer/asphalt blends. The results presented here indicate that different asphalt chemistries can lead to varying degrees of polymer degradation in the case of unsaturated polymers. In general, we found RTFO aging to be more severe than actual field operations. With regard to longterm aging Caltrans and our findings appear to support the notion that CATOD is a reasonable simulation for field aging in hot desertlike climates. In more moderate climates we observed less than 20 percent reduction of $C_{\rm eff}$ even after 3 to 4 years in the field.

Improved thermal and oxidative stability can be achieved by modifying asphalts with saturated polymers. However, good handling practices are still necessary to preclude the consequences of serious asphalt age hardening. GPC, rheological characterization, and field data demonstrate the superior performance of these blends even in severe climates.

NOMENCLATURE

Symbol	Meaning	Units
A.I.	aging index $[\eta(RTFO)/\eta(original)]$	
AC	asphalt cement	
AR	asphalt residue	
C	concentration	kg/m³ or %w
CATOD	California tilt oven durability test	70 11
G*	complex dynamic shear modulus	Pa
G'	storage modulus	Pa
G"	loss modulus	Pa
HMA	hot mix asphalt	
M_{w}	Weight average mean molecular	g/mol
	weight	
P	pressure	P_a
PE	polyethylene	
P_0	static pressure	Pa
RTFO	rolling thin film oven	
SBR	styrene-butadiene rubber random co-	
	polymer	
SBS	styrene-butadiene-styrene block co-	
	polymer	
SEBS	styrene-ethylene-butylene-styrene	
	block copolymer	
t	time	sec or hr
t_o	duration of loading	sec
T	temperature	°C
T_{cr}	critical cracking temperature	°C
%w	percent weight	_
η	viscosity	Pa

angular velocity rad/sec ω

aged aged polymer eff effective

HM high molecular weight species,

 $(SB)_x$, where $x \ge 2$

LM low molecular weight species

REFERENCES

- 1. C. P. Valkering, D. J. L. Lancon, E. de Hilster, and D. A. Stoker. Proc., Association of Asphalt Paving Technologists, Vol. 59,
- 2. M. G. Bouldin and J. H. Collins. Polymer Modified Asphalt Binders. ASTM STP 1108 (K. R. Wardlaw and S. Shuler, eds.), ASTM, Philadelphia, 1990.
- 3. D. N. Little. Polymer Modified Asphalt Binders. ASTM STP 1108 (K. R. Wardlow and S. Shuler, eds.). ASTM, Philadelphia, Pa., 1990.
- 4. A. A. Tayebali, J. L. Goodrich, J. B. Sousa, and C. L. Monismith. Proc., Association of Asphalt Paving Technologists, Vol. 60, 1991.
- 5. R. J. Salter and M. Y. O. Al-Shakarchi. Transportation Research Record 1228, TRB, National Research Council, Washington, D.C.,
- 6. R. Reese and N. Predoehl. Caltrans Report 65324-633383. Sacramento, Calif., 1989.
- 7. L. J. Fleckenstein, K. Mahboub, and D. L. Allen. Polymer Modified Asphalt Binders. ASTM STP 1108 (K. R. Wardlow and S. Shuler, eds.). ASTM, Philadelphia, Pa., 1990.
- 8. J. H. Collins, M. G. Bouldin, R. Gelles, and A. Berker. Proc., Association of Asphalt Paving Technologists, Vol. 60, 1991.
- J. L. Goodrich. Proc., Association of Asphalt Paving Technologists, Vol. 60, 1991.
- 10. M. G. Bouldin, J. H. Collins and A. Berker. Rheology and Microstructure of Polymer/Asphalt Blends. Rubber Chem. Tech., Vol. 64, No. 4, 1991.
- 11. D. C. Portfolio and F. A. Fensel. Proc., 3rd International Symposium Roofing Techn., Montreal, Canada, 1991.

- 12. J. H. Hills. Inst. Petr. Tech. 74-014, 1974.
- 13. J. B. Sousa, J. Craus, and C. L. Monismith. SHRP-A/1R-91-104. Washington, D.C., 1991.
- 14. M. G. Bouldin, Shell Development Company Technical Bulletin. Houston, Tex., 1991.
- 15. J. C. Petersen. In Transportation Research Record 999, TRB, National Research Council, Washington, D.C., 1984.
- 16. A. S. Noureldin and L. E. Wood. In Transportation Record 1228, TRB, National Research Council, Washington, D.C., 1989.
- 17. B. H. Chollar, J. A. Zenawitz, J. G. Boone, K. T. Tran, and D. T. Anderson. In Transportation Research Record 1228, TRB, National Research Council, Washington, D.C., 1989.
- 18. J. A. S. Pribanic, M. Emmelin and G. N. King. In Transportation Research Record 1228, TRB, National Research Council, Washington, D.C., 1989
- 19. F. N. Hveem, E. Zube, and J. Skog. Proc., Association of Asphalt Paving Technologists, Vol. 32, 1963.
- 20. J. D. Ferry. Viscoelastic Properties of Polymers (third edition). John Wiley & Sons, New York, 1980.
- 21. S. C. S. R. Tangella, J. Craus, J. A. Deacon, and C. L. Monismith. SHRP-A/1R-90-011. Washington, D.C., 1990.
- J. B. Sousa, G. Rowe, and A. A. Tayebali. (Submitted for publication.)
- 23. W. van Dyke and W. Visser. Proc., Association of Asphalt Paving
- Technologists, Vol. 46, 1977, pp. 1-40. 24. C. T. Chari, B. E. Ruth, M. Tia, and G. C. Page. Proc., Association of Asphalt Paving Technologists, Vol. 59, 1990, pp. 176-
- 25. C. J. Glover, R. R. Davison, S. M. Ghoreishi, H. B. Jemison, and J. A. Bullin. In Transportation Research Record 1228, TRB, National Research Council, Washington, D.C., 1989.
- 26. C. A. Bell. SHRP-A/IR-89-004. Washington, D.C., 1989
- 27. G. R. Kemp and N. H. Predoehl. Proc., Association of Asphalt Paving Technologists, Vol. 50, 1981.
- 28. M. G. Bouldin and M. A. Berggren. Shell Development Company Technical Bulletin. Houston, Tex., 1991.

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