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Development of an Eastern Shale Oil Residue as an Asphalt Additive - Subtask 2.5

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ABSTRACT

An evaluation of eastern shale oil as an asphalt additive to reduce oxidative age hardening and moisture susceptibility is being conducted. An eastern shale oil residue having a viscosity of 1.30 Pa•s at 60°C (140°F) was blended with three different petroleum-derived asphalts that are known to be very susceptible to oxidative aging. In addition, blends of the eastern shale oil residue and the petroleum-derived asphalts are being coated onto three different aggregates that are known to be susceptible to water stripping.

The oxidative age hardening portion of this study is not complete at this time. To date, information has been obtained on the unaged samples and two of the aged petroleum-derived asphalts (AAD-1 and AAK-1). When complete, this data will include rheological data on the unaged, RTFO-aged, and the RTFO/PAV-aged samples and infrared data on the unaged and RTFO/PAV-aged samples. With respect to the rheological data, asphalt AAD-1 meets the specifications of a PG 58 asphalt while asphalt AAK-1 does not. In the latter case this indicates that AAK-1 is more appropriately evaluated at a higher temperature range. The infrared spectroscopic data obtained for the eastern shale oil residue show that it contains appreciable amounts of carbonyl and sulfoxide compound types, 0.22 absorbance units and 0.27 moles/L, respectively. Thus, upon the addition of this residue to the three petroleum-derived asphalts the blends contain increased amounts of these functional groups relative to the petroleum-derived asphalts. This has been observed with other additives and is not considered detrimental.

In addition, the data that has been collected to date indicate that the moisture susceptibility of blends of eastern shale oil residue and asphalt AAM-1 are somewhat improved when coated onto Lithonia granite. However, the addition of eastern shale oil residue to asphalts AAD-1 and AAK-1 did not improve the moisture susceptibility of these petroleum-derived asphalt blends when coated onto Lithonia granite. The results of the petroleum-derived asphalts and blends when coated onto low absorption limestone and silicious Gulf Coast gravel are not complete at this time.

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EXECUTIVE SUMMARY

It has been demonstrated in roadways that a western shale oil-derived asphalt additive when blended with a petroleum-derived asphalt is less susceptible to moisture damage and to oxidative age hardening than the petroleum-derived asphalt when each is laid as a pavement in the same vicinity. Based upon the experience and information that is available for this additive produced from western shale oil, an evaluation of eastern shale oil as another source of an asphalt additive is being conducted. A stabilized vacuum residue prepared from an eastern shale oil produced by the KENTORT II process is being evaluated as an asphalt additive in this study. The residue was blended with three different petroleum-derived asphalts that are known to be very susceptible to oxidative aging. In addition, blends of the eastern shale oil residue and the petroleum-derived asphalts are being coated onto three different aggregates that are known to be susceptible to water stripping.

The results of this evaluation will provide information regarding the ability of the eastern shale oil residue, when used as an additive in petroleum-derived asphalts, to reduce oxidative age hardening and moisture susceptibility. The specification methods established by the Strategic Highway Research Program (SHRP) and approved by the American Association of State Highway and Transportation Officials (AASHTO) are being utilized in this study. In general, the SHRP/AASHTO methodology differs from the older methods as follows. The SHRP/AASHTO method requires determination of the temperatures at which specified physical properties are met as opposed to the older methods which required determination of certain physical properties at specified temperatures. The SHRP/AASHTO method also requires examination of physical properties over the full service temperature range, whereas the practical lower limit of the older methods was 25° C (77° F). All of the information that is being obtained for the blends is being compared with that obtained for the petroleum-derived asphalts.

The oxidative age hardening portion of this study is not complete at this time. To date, information has been obtained on the unaged samples and two of the aged petroleum-derived asphalts (AAD-1 and AAK-1). When complete, this data will include rheological data on the unaged, RTFO-aged, and the RTFO/PAV-aged samples and infrared data on the unaged and RTFO/PAV-aged samples. With respect to the rheological data, asphalt AAD-1 meets the specifications of a PG 58 asphalt, while asphalt AAK-1 does not. In the latter case, this indicates that AAK-1 is more appropriately evaluated at a higher temperature range. The infrared spectroscopic data obtained for the eastern shale oil residue show that it contains appreciable amounts of carbonyl and sulfoxide compound types, 0.22 absorbance units and 0.27 moles/L, respectively. Thus, upon the addition of this residue to the three petroleum-derived asphalts, the blends develop increased amounts of these functional groups relative to the petroleum-derived asphalts. This phenomenon has been observed in other properly functioning systems and is not considered detrimental. Control of age hardening (not oxidation alone) is the desired goal.

In addition, the data that has been collected to date indicate that the moisture susceptibility of blends of eastern shale oil residue and asphalt AAM-1 are somewhat improved when coated onto Lithonia granite. However, the addition of eastern shale oil residue to asphalts AAD-1 and AAK-1 did not improve the moisture susceptibility of these petroleum-derived asphalt blends when coated onto Lithonia granite. The results of the petroleum-derived asphalts and blends when coated onto low absorption limestone and silicious Gulf Coast gravel are not complete at this time.

INTRODUCTION

Purpose

The primary purpose of this study is to conduct an evaluation of another natural resource that currently is not used for the production of commercial products. The justification for conducting this study is based upon previous work on western shale oil. An intensive investigation of (Green River) western shale oil and the discovery that a residue produced from it reduced the moisture susceptibility of petroleum-derived asphalt pavements eventually led to the granting of an exclusive patent in 1982.

Since that time, 13 test strips that incorporate the shale oil-derived additive have been constructed in five states (Lukens and Plummer 1988, Harnsberger and Robertson 1994). The most recent is 6 miles of Interstate 90 just west of Sundance, Wyoming, which was reconstructed in the summer of 1994 using this western shale oil-derived additive.

Based on this body of work and our experience in shale oil chemistry, it appeared that a vacuum residue produced from eastern shale oil may also possess unique properties–for different reasons than those ascribed to western shale oil–that might improve the performance of conventional asphalts. In the case of eastern shale oil, it was suspected that the improvement would appear to be directed at reducing the propensity for moisture damage and oxidative aging of asphalts. Moisture damage and age hardening, which lead to embrittlement and subsequent cracking of asphalt pavements, have proven to be two of the major modes of failure of asphalt highways.

The objective of this task is to use a stabilized eastern shale oil residue to develop an additive for petroleum-derived paving asphalts. If the results of this study demonstrate that the preliminary indications are correct, then Western Research Institute (WRI) will pursue a patent on the concept and thus extend our capability to license asphalt-modification materials. In addition, WRI will also evaluate other recent technical developments and, if appropriate, apply for patent coverage.

Background

Since the discovery, patenting, and licensing of the technology to the New Paraho Corporation of the concept and process for the production of an asphalt modifier that can be produced from western shale oil, 13 test strips have been constructed in five states in the United States (Lukens and Plummer 1988, Harnsberger and Robertson 1994). These states include Wyoming, Colorado, Utah, Texas, and Michigan. The environment of the shale oil-modified asphalt (SOMAT) test strips in these states include, for example, hot, dry climates (Utah and Texas) and cold, wet climates (Colorado and Michigan). One of the test strips located in Wyoming is west of Rawlins in the westbound driving lane of Interstate 80. The environment at this location is cold and relatively dry but, in addition to the weather, the pavement is also subjected to a very high volume of interstate truck traffic.

One of the more recent test strips was laid in early to mid September of 1993. This test strip is 2500 feet long and is located in the northbound lane of U.S. Highway 89 north of Jackson, Wyoming. This test strip, which is located in Grand Teton National Park, will be evaluated both for pavement performance and environmental impact by personnel from WRI for the next 5 years and represents an evaluation of shale oil-modified asphalt (SOMAT) under extreme climatic conditions. In addition, 6 miles of the westbound driving lane of Interstate 90 just west of Sundance, Wyoming was reconstructed in the summer of 1994 using this shale oil-derived additive.

In the construction of these test strips 10 to 15% of the shale oil modifier (SOM) was blended with conventional petroleum-derived asphalt, then the highway was built using conventional construction methods. To date, the results of field evaluations of test strips constructed of SOMAT indicate that the test strips are less susceptible to moisture damage (stripping) than the conventional petroleum-derived asphalts that are laid in the immediate vicinity of the SOMAT test strips. These SOMAT pavements also age more slowly than conventional pavements, which prolongs the time to embrittlement cracking.

It is known that highly aromatic components, such as those common in eastern shale oil, are as good a dispersant as are the pyridinic components in western shale oil. These are the types of materials that are needed to improve the compatibility of the components in paving asphalts to reduce aging. A reduction in aging reduces the probability of embrittlement that contributes to fatigue cracking (alligator cracks), that leads to reduced pavement strength. A reduction in cracking also reduces the potential for the invasion of water into and under the pavement, which can eventually lead to freeze-thaw deterioration of the pavement. Fatigue cracking constitutes one of the three major forms of asphalt pavement failure, the others being thermal cracking and permanent deformation (rutting and shoving). Fatigue and permanent deformation are greatly aggravated by moisture damage.

During the last few years, WRI has conducted some experiments in cooperation with the University of Kentucky, Center for Applied Energy Research (CAER), to examine residua from eastern shale oil for the same purposes as have been achieved with (Green River) western shale oil (Harnsberger and Robertson 1990; Mahboub et al. 1992). While some of the results were encouraging, there was a stability problem with the eastern shale oil residua which were evaluated.

Based on the results discussed above, additional discussions were held with the CAER regarding working jointly on a project to produce, stabilize, and evaluate a residue from eastern shale oil. Consequently, the CAER produced and stabilized an eastern shale oil under a separate U.S. Department of Energy (DOE) contract. The residue was then shipped to WRI where it is currently being evaluated as an asphalt additive. This latter work at WRI is being conducted using DOE funding associated with Cooperative Agreement DE-FC21-93MC30126.

METHODOLOGY

Upon arrival at WRI, the stability of the eastern shale oil residue, which was produced from the Cleveland member of the Ohio shale by the KENTORT II process (Carter et al. 1990, 1994), was evaluated by measuring its viscosity using a Brookfield viscometer, ASTM D 4402 (ASTM 1992). The viscosity of the residue was then measured again one month later and the data compared. In addition, the flash point of the residue was determined using ASTM D 92, and the distillate range was determined using a gas chromatographic procedure that relies upon the use of an external standard for quantification (Thomas et al. 1987).

Since the viscosity of the residue was too low, it was vacuum distilled to produce a material that had a higher viscosity. The vacuum distillation procedure is described by Thomas et al. (1994). The viscosity of the resultant residue was measured and considered to be satisfactory. Consequently, mixtures containing 10 and 15 wt% of this eastern shale oil residue were prepared using three conventional petroleum-derived asphalts. The three petroleum-derived asphalts and the six blends are currently being evaluated. Specifically, each material is being evaluated using the Strategic Highway Research Program (SHRP) developed and American Association of State Highway and Transportation Officials (AASHTO) approved specification methods which examine the rheology (viscoelastic properties) of new and artificially-aged materials (AASHTO 1994). The results will be compared with AASHTO specification values. All materials are being aged artificially to simulate both hot-mix plant aging and pavement aging using the rolling thin-film oven (RTFO) and the pressure aging vessel (PAV) methods, T240 and PP1, respectively. The rheological properties of these aged and unaged materials will be compared. Controls will be employed to demonstrate any advantages of the eastern shale oil residue as an asphalt additive. In addition, the WRI-developed test for moisture susceptibility (Plancher et al. 1980) is being applied to all material combinations (including controls) to demonstrate any advantages that the eastern shale oil residue may have to reduce moisture damage in asphalt-aggregate mixtures.

RESULTS AND DISCUSSION

The eastern shale oil residue that is being evaluated in this study was produced by CAER using the fluidized-bed, KENTORT II process. Stabilization of the shale oil produced by this process was accomplished by distillation of the oil to an initial boiling point of about 204° C (400° F). The stabilized vacuum residue was received at WRI and the evaluation of the residue as an asphalt additive was initiated. In order to insure that the residue was indeed stabilized, the viscosity of a sample of the residue was determined shortly after arrival at WRI and then again one month later. The initial viscosity of the residue was determined to be 1.29 Pa•s at 60° C (140° F), and its viscosity one month later was determined to be 1.30 Pa•s at 60° C (140° F).

Based on these results, it was concluded that the residue was indeed stable. In addition, the flash point and the initial boiling point of the residue were determined to be 191.5° C (377° F) and 197° C (386° F), respectively. However, the viscosity of the residue was considered to be too low for use as an additive in petroleum-derived asphalts. Consequently, the residue was vacuum distilled to produce a material that had an initial boiling point of about 243° C (470° F). The viscosity of this residue was determined to be 23.9 Pa•s at 60°C (140°F). Thus, if this residue were graded according to ASTM D 3381, it meets the low-temperature, viscosity requirement of an AC-2.5 asphalt. This residue, which is the result of the vacuum distillation procedure, is the material that was used to prepare the eastern shale oil residue / petroleum-derived asphalt blends that are the subject of this report.

Three petroleum-derived asphalts that exhibit high levels of oxidative aging were selected for this study. They are designated in the SHRP nomenclature as AAD-1, AAK-1, and AAM-1. According to ASTM D 3381, these three asphalts are graded as an AR-4000, an AC-30, and an AC-20, respectively. The viscosity of these asphalts at 60° C (140° F) is 10.6, 3.26, and 1.99 Pa•s, respectively. These three asphalts are also different from one another in several other important respects as regards this study. AAD-1 and AAK-1 have high propensities for aging. However, while AAK-1 is considered a good asphalt, AAD-1 is only considered marginal for pavement applications in hot climates. Both asphalts contain high concentrations of asphaltenes and sulfur, moderate amounts of nitrogen, and have relatively low molecular weights. However, AAK-1 is more aromatic than AAD-1.

AAM-1 is rather unique in that it is an aromatic asphalt that contains low amounts of nitrogen, sulfur, and asphaltenes, has a high molecular weight, has a moderate propensity for aging, but is considered a good asphalt where it has been used in Texas (a hot, aging prone climate). The eastern shale oil residue was mixed with these three conventional asphalts at two concentrations, 10 and 15 wt%. The sample matrix is shown in Table 1.

Table 1. Sample Matrix

Petroleum-Derived Asphalt	AAD-1	AAK-1	AAM-1
Eastern Shale Oil Residue - 0%	x	x	x
Eastern Shale Oil Residue - 10%	x	x	x
Eastern Shale Oil Residue - 15%	x	x	x

The rheological properties are being determined for various aged and unaged samples. The results of aging in the RTFO will provide information on the extent of aging that can be expected to occur in the hot-mix plant. An evaluation of the data obtained for samples aged in the RTFO and PAV will give an indication of the extent of aging that will occur in the pavement. By plotting the viscoelastic data collected at 13 and 25°C (55 and 77°F), the minimum temperature asphalt grade can be determined for the various blends. A performance grade that can be used in Kentucky is PG 58. The temperatures of 13 and 25°C (55 and 77°F) are the extremes for the mid-range temperature rheological evaluation of the PAV-aged asphalt for fatigue cracking. These temperatures correspond to PG 58-16 and PG 58-40 grades of asphalt, respectively. A temperature of 58°C (136°F) is the average 7-day maximum pavement design temperature, and -16 and -40°C (3 and -40°F) are the limits for the minimum pavement design temperature.

From the above information, the relative degree of aging of the petroleum-derived asphalts and the blends can be ascertained. The data that have been obtained to date are shown in Table 2. In this table and the following tables, the dashed lines indicate data that still need to be collected.

The G*/sinδ value for the unaged asphalts and blends is shown in the table. This parameter is a measure of a binder's ability to resist rutting. The value obtained must be greater than the minimum of 1.00 kPa at 58° C (136° F), and in all cases the value for the petroleum-derived asphalts and the blends exceeds 1.00 kPa. The reason that the values for the three petroleum-derived asphalts are significantly different from one another is that they are viscosity-graded (according to ASTM D 3381) as AR-4000, AC-30, and AC-20 asphalts.

The limited rheological data currently available regarding the aged samples is also shown in Table 2. The G*/sinδ value for two of the RTFO-aged petroleum-derived asphalts exceeds the minimum value of 2.20 kPa at 58° C (136° F). For binders subjected to aging using both the RTFO and the PAV, the G*sinδ value for the aged binders must not exceed a maximum value of 5000 kPa at the test temperature, in this case 13 and 25° C (55 and 77° F). The G*sinδ value is a measure of an aged binder's ability to resist fatigue cracking.

Table 2. Rheological Characteristics of Unaged and Various Aged Samples

	G*/sinδ kPa, 58°C 10 rad/s		G*sinδ kPa,10 rad/s RTFO/PAV-Aged		Relative Aging Index, RTFO-Aged
Sample	Unaged	RTFO-Aged	13° C	25° C	
AAD-1	1.65	6.60	12407	2684	4.0
AAD-1/ESO 10%	1.27	--	--	--	--
AAD-1/ESO 15%	1.22	--	--	--	--
AAK-1	5.16	14.84	22805	5595	2.9
AAK-1/ESO 10%	3.07	--	--	--	--
AAK-1/ESO 15%	2.54	--	--	--	--
AAM-1	3.02	--	--	--	--
AAM-1/ESO 10%	1.75	--	--	--	--
AAM-1/ESO 15%	1.41	--	--	--	--
Limit Values, kPa					
Minimum	1.00	2.20	NA	NA	NA
Maximum	NA	NA	5000	5000	NA

NA means not appropriate.

For the two petroleum-derived asphalts tested to date, this maximum value was exceeded in all but one case–the G*sinδ at 25°C (77°F) for asphalt AAD-1. This indicates that asphalt AAK-1 does not meet the specifications for this performance grade and is more appropriately evaluated at a higher temperature range. Remember that asphalt AAK-1 is viscosity-graded as an AC-30 asphalt; thus, it is stiffer than the other two asphalts, which are graded as AR-4000 (AAD-1) and AC-20 (AAM-1) asphalts.

Infrared analyses is also being conducted on these blends before and after RTFO/PAV aging to determine changes in the concentrations of the oxygen-containing functional groups (i.e., carbonyls and sulfoxides) which are present in the asphalt samples. The objective of this phase of the study is to determine the ability of the eastern shale oil residue to reduce the propensity for these conventional petroleum-derived asphalts to exhibit aging at any given level of oxidation. The infrared spectroscopic data for the unaged asphalts is shown in Table 3.

Table 3. Infrared Spectroscopic Analysis of Carbonyls (absorbance units) and Sulfoxides (moles/L) in Unaged and RTFO/PAV-Aged Samples

Sample	Carbonyls / Sulfoxides	
	Unaged	Aged
AAD-1	0.02/0.02	--/--
AAD-1/ESO 10%	0.04/0.05	--/--
AAD-1/ESO 15%	0.04/0.05	--/--
AAK-1	0.02/0.02	--/--
AAK-1/ESO 10%	0.03/0.06	--/--
AAK-1/ESO 15%	0.04/0.05	--/--
AAM-1	0.04/0.02	--/--
AAM-1/ESO 10%	--/--	--/--
AAM-1/ESO 15%	--/--	--/--
Eastern shale oil residue (ESO)	0.22/0.27	--/--

At this time, the only conclusion that can be drawn is that the addition of the eastern shale oil residue to the petroleum-derived asphalts results in an increase in the amounts of carbonyl and sulfoxide compound types. This, however, is not unusual considering that the shale oil, which was produced by a thermal process, contains appreciable amounts of carbonyl and sulfoxide compound types as is shown in the table. Upon completion of this study, consideration of the data shown in Tables 2 and 3 will make it possible to determine the impact that blending an eastern shale oil residue with these petroleum-derived asphalts has on the extent of aging of these blends at high oxidation levels.

Asphalt-aggregate briquets were prepared in duplicate using the petroleum-derived asphalts and the blends. Three different aggregates are being tested. These include Lithonia granite (RA), a low absorption limestone (RD), and a silicious Gulf Coast gravel (RL). The abbreviations, RA, RD, and RL, are the SHRP nomenclature for these aggregates. The briquets are being subjected to repeated freeze-thaw cycling until breakage. The objective of this phase of the study is to evaluate the moisture susceptibility of the prepared asphalt-aggregate samples. The briquets represent mixtures that may be used in a pavement constructed of the different binder and aggregate materials. A summary of the data that have been collected to date is shown in Table 4.

The results of the determination of the moisture susceptibility of the petroleum-derived asphalts and the blends when coated onto Lithonia granite (RA) demonstrate that when the residue is mixed with asphalt AAM-1 some improvement is observed. While the AAM-1 asphalt survived only one to two freeze-thaw cycles, the blends survived two to four cycles. However, the addition of the residue to asphalts AAD-1 and AAK-1 did not improve the moisture susceptibility of these petroleum-derived asphalt blends when coated onto Lithonia granite. Regarding the three petroleum-derived asphalts and the blends when coated onto the low absorption limestone (RD) and silicious gulf coast gravel (RL), the former seems to be doing quite well with respect to all asphalts and blends, having survived at least three cycles as of the end of this reporting period. (Regarding the asphalts and blends coated onto RD, the number of cycles completed is greater for some samples than others only because they began the freeze-thaw cycling procedure at an earlier date.) Meanwhile, the latter, at least with respect to asphalt AAD-1 and blends coated onto RL, failed after one cycle.

Briquets of the other two asphalts and blends have been prepared but have not yet been subjected to freeze-thaw cycling.

Table 4. Results of Water Susceptibility Test Conducted on Samples in Duplicate

Aggregates	Binders	Cycles to Failure
Lithonia Granite (RA)	AAD-1	1/1
	AAD-1/ESO 10%	1/1
	AAD-1/ESO 15%	1/1
	AAK-1	1/1
	AAK-1/ESO 10%	1/1
	AAK-1/ESO 15%	1/1
	AAM-1	1/2
	AAM-1/ESO 10%	3/4
	AAM-1/ESO 15%	2/3
Low Absorption Limestone (RD)	AAD-1	>10/>10
	AAD-1/ESO 10%	>10/>10
	AAD-1/ESO 15%	>9/>9
	AAK-1	>9/>9
	AAK-1/ESO 10%	>7/>7
	AAK-1/ESO 15%	>6/>6
	AAM-1	>6/>6
	AAM-1/ESO 10%	>4/>4
	AAM-1/ESO 15%	>3/>3
Silicious Gulf Coast Gravel (RL)	AAD-1	1/1
	AAD-1/ESO 10%	1/1
	AAD-1/ESO 15%	1/1
	AAK-1	--/--
	AAK-1/ESO 10%	--/--
	AAK-1/ESO 15%	--/--
	AAM-1	--/--
	AAM-1/ESO 10%	--/--
	AAM-1/ESO 15%	--/--

CONCLUSIONS

At this time the conclusions that can be made are limited. The oxidative age hardening portion of this study is not complete. To date, information has been obtained on the unaged samples and two of the aged petroleum-derived asphalts (AAD-1 and AAK-1). When complete, this data will include rheological data on the unaged, RTFO-aged, and the RTFO/PAV-aged samples and infrared data on the unaged and RTFO/PAV-aged samples.

With respect to the rheological data, asphalt AAD-1 meets the specifications of a PG 58 asphalt while asphalt AAK-1 does not. In the latter case this indicates that AAK-1 is more appropriately evaluated at a higher temperature range. The infrared spectroscopic data obtained for the eastern shale oil residue show that it contains appreciable amounts of carbonyl and sulfoxide compound types, 0.22 absorbance units and 0.27 moles/L, respectively. Thus, upon the addition of this residue to the three petroleum-derived asphalts, the blends contain increased amounts of these functional groups relative to the petroleum-derived asphalts.

In addition, the data that have been collected to date indicate that the moisture susceptibility of blends of eastern shale oil residue and asphalt AAM-1 are somewhat improved when coated onto Lithonia granite. However, the addition of eastern shale oil residue to asphalts AAD-1 and AAK-1 did not improve the moisture susceptibility of these petroleum-derived asphalt blends when coated onto Lithonia granite. The results of the petroleum-derived asphalts and blends when coated onto low absorption limestone and silicious Gulf Coast gravel are not complete at this time.

DISCLAIMER

The mention of specific brand names or models of equipment is for information only and does not imply endorsement by Western Research Institute or the United States Department of Energy.

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