**Evaluation of Oil Shale Bitumen as a
Pavement Asphalt Additive to Reduce
Moisture Damage Susceptibility**

Topical Report

**R.E. Robertson
P.M. Harnsberger
J.M. Wolf**

January 1991

Work Performed Under Contract No.: DE-FC21-86MC11076

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Western Research Institute
Laramie, Wyoming

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TABLE OF CONTENTS

	<u>Page</u>
LIST OF TABLES	iii
SUMMARY	iv
INTRODUCTION	1
EXPERIMENTAL	2
Materials	2
Procedures	2
RESULTS AND DISCUSSIONS	3
CONCLUSIONS	4
ACKNOWLEDGEMENTS	5
DISCLAIMER	5
REFERENCES	6

LIST OF TABLES

<u>Table</u>	<u>Page</u>
1. Water Sensitivity Test (WST) Results for Neat and Modified Wyoming Sour Asphalt in Combination with Two Aggregates	3

SUMMARY

An unrefined shale bitumen was evaluated as an agent to reduce moisture damage susceptibility of asphalt aggregate mixtures. Some activity was observed but less than might have been expected based on the molecular weight and nitrogen content of the bitumen. The counter effects of free carboxylic acids, which are known to be variable in asphalt and which are also present in the unrefined bitumen, appear to diminish the activity of the bitumen to inhibit moisture damage.

INTRODUCTION

Moisture damage in asphalt-concrete (compacted asphalt-graded aggregate mixtures) used in roadways is a major cause of failure in pavement and has received much attention for many years. The presence of moisture on and in roads is, of course, unavoidable. One of the more effective chemical methods used with asphalt to reduce the effects of moisture has been to modify asphalt with basic nitrogen components isolated from shale oil.

The objective of this study is to examine the utility of an unrefined oil shale-derived product as an additive to reduce moisture damage susceptibility of asphalt-aggregate pavement mixtures. The effectiveness of the pyridinic portions of shale oil as antistripping additives has been demonstrated by Plancher and Petersen (1982, 1984) using close-graded aggregates in a water sensitivity test (WST) also developed and described by Plancher et al. (1980). This method reduces the aggregate interlocking interference commonly observed when fully graded aggregates are used and, thereby, allows good comparison among test samples of differing composition. In the previous studies, partially refined shale oil products were employed. The more specific objective in this study was to conduct a very limited (preliminary) evaluation of the effectiveness of an unrefined bitumen produced from oil shale as an additive to reduce moisture susceptibility in WST mixtures. The possible advantages are (1) the cost of the additive is lower because there is no refining, (2) shale bitumen is typically rich in basic nitrogen, and (3) the generally higher molecular weight of bitumen versus shale oil may be advantageous. The latter has been demonstrated repeatedly in numerous and varied studies of interfacial phenomena. The possible disadvantage to using unrefined shale bitumen is that materials typically removed during refining may interfere with the effectiveness of the high molecular weight, nitrogen-rich components.

The shale bitumen chosen was prepared previously at Western Research Institute (WRI) as described by McKay et al. (1983). Several extractions of the bitumen were combined to provide sufficient material for this study, but none was refined. Generally, McKay's procedure is supercritical fluid extraction of oil shale with methanol and water at about 752°F (400°C) for periods of one hour or less. Products are recovered by extraction with organic solvent, which was subsequently removed in vacuo.

Comparisons were made among a neat asphalt, an asphalt modified with two concentrations of shale bitumen, and an asphalt modified with a commercial antistrip agent. Two aggregates were used, and all combinations of neat and modified asphalts were tested in replicate.

EXPERIMENTAL

Materials

Asphalt

The asphalt used in this study was Wyoming Sour, an AC-10-grade asphalt.

Shale Bitumen

The shale bitumen was produced at WRI from Green River Formation oil shale as described by McKay et al. (1983). Several extractions of bitumen were combined to provide a sufficient quantity of starting material. The bitumen was shown to have a high organic acid content and an average molecular weight of about 500 amu (McKay and Chong 1983). The material was used as received.

Commercial Antistrip Additive

The commercial antistrip additive used was Redicote 80S.

Aggregates

The aggregates used in the water susceptibility study were Teton Mountain gravel, a conglomerate from Cheyenne, Wyoming, and Watsonville granite, a granite from Watsonville, California.

Procedures

Blends of the Wyoming Sour asphalt and the shale bitumen were prepared with concentrations of 15% and 20% bitumen. A 0.5% blend of Redicote antistrip agent to Wyoming Sour asphalt was also prepared. These mixtures were made by weighing the proper amount of each component into a wide mouth glass jar and flushing the void space of the jar with argon before sealing. The jars were heated in an oven at 275°F (135°C) for one hour, and removed every 20 minutes, and manually shaken for one minute.

Test briquets were prepared using the method described by Plancher et al. (1980). Asphalt-aggregate briquets were prepared using a 5 wt % coating of binder on 20- to 35-mesh aggregate. The asphalt-aggregate mixture was compacted in a laboratory press to form a 1.90 x 4.13 cm diameter cylindrical briquet. Each briquet is submerged in water and subjected to alternating freeze and thaw cycles while suspended on a bevelled plastic support. The freeze and thaw cycles were each 24 hours long, the freeze temperature was 14 to 5°F (-10 to -15°C), and the thaw temperature was 140°F (60°C). The test was concluded when the briquet broke.

The infrared spectrum of the shale bitumen was obtained in carbon disulfide using a Perkin-Elmer 983G infrared spectrometer. The spectrum was obtained using a concentration of 50 mg/mL.

RESULTS AND DISCUSSIONS

Watsonville granite and Teton gravel were chosen as test aggregates because these are commonly used in roadways and both have histories of showing moisture damage problems. The expected service life is improved when antistripping additives are used with either of these aggregates during road construction. The results of the laboratory water sensitivity tests using these two aggregates and the asphalt blends are listed in Table 1.

Table 1. Water Sensitivity Test (WST) Results for Neat and Modified Wyoming Sour Asphalt in Combination with Two Aggregates

Experiment	Freeze-Thaw Cycles to Failure	
	Teton	Watsonville
1. Neat Asphalt	1	3
2. Neat Asphalt	1	3
3. Asphalt + 15% shale bitumen	2	3
4. Asphalt + 15% shale bitumen	3	3
5. Asphalt + 20% shale bitumen	2	3
6. Asphalt + 20% shale bitumen	3	3
7. Asphalt + 0.5% Redicote 80S	2	4
8. Asphalt + 0.5% Redicote 80S	2	5

It is immediately apparent that the commercial antistripping additive was used at a low concentration compared with the shale bitumen. The commercial material is a synthetic product, which must be used sparingly in actual roadway construction because of its relatively high cost. The results show that the shale bitumen did not improve resistance to moisture damage with Watsonville aggregate compared to the control, but Redicote 80S did have a small positive effect. However, the shale bitumen did reduce the moisture susceptibility of the asphalt-Teton mixtures, but this occurred independent of concentration. It appears then that greater than 15% shale bitumen offers no advantage. It may be insignificant that the shale bitumen outperformed the commercial additive with the Teton aggregate with an average of 2.5 versus 2.0 cycles to failure, respectively. Nonetheless, these results do show that the shale bitumen has a positive effect and that this unrefined product does compare well with the synthetic additive.

The results are not altogether encouraging compared with other studies that used shale-derived products; therefore, attention was focused on why the shale bitumen shows only modest activity. McKay and

Chong (1983) reported that significant amounts of carboxylic acids are present in the unrefined bitumen. Carboxylic acids in asphalts are known to have varied effects on moisture sensitivity of asphalt-aggregate mixtures. The infrared spectrum of the shale bitumen was obtained, and it confirmed that this material contained substantial amounts of free carboxylic acid. It appears that the free carboxylic acid probably diminishes the activity of the unrefined shale bitumen to reduce moisture damage susceptibility in pavement mixtures. No attempt was made to investigate the effects of asphalt aggregate mixtures that were aged to simulate several years of pavement service.

CONCLUSIONS

The results show that there is an advantage to using unrefined shale bitumen as an agent to reduce moisture damage susceptibility when Teton gravel is used as an aggregate. However, this was not so for Watsonville granite. Some refinement of the bitumen is needed to achieve the types of activity demonstrated with other shale-derived products.

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DISCLAIMER

Mention of specific brand names or models of equipment is for information only and does not imply endorsement of any particular brand.

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