A PRELIMINARY INVESTIGATION OF THE FEASIBILITY OF SPENT OIL SHALE AS ROAD CONSTRUCTION MATERIAL

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The use of spent oil shale material as aggregate for flexible pavement construction was investigated, and its suitability for use both in base courses and in bituminous surface courses is discussed. To assess the aggregate and asphalt-aggregate characteristics of spent shale the following tests were performed: the Los Angeles abrasion test, the dry sieve analysis, the specific gravity test, the Atterberg limits test, and the Hveem method of mix design. The results of the testing showed that the spent shale aggregate was well-graded, flat, angular, highly absorbent, friable, and nonplastic, has a rough surface texture, and wears relatively easily. The aggregate mixes tested by the Hveem stabilometer yielded high strength values and were relatively lightweight. The asphalt-spent shale mixtures studied showed very high total resistance (combination of stability and cohesiometer values) values, i.e., more than adequate load-carrying capability. However, the asphalt contents necessary for these very high strengths were also high. Based on the results of the tests performed, it seems apparent that this particular type of spent shale material might perform very well in flexible pavement structures. Although the material showed more than adequate strength and stability, it may not stand up as well to the abrasive action of traffic on high-capacity roads and may be expensive because of the seemingly large amount of asphalt needed. However, this mixture might perform very well as a surface course layer for lower capacity roads (e.g., secondary roads).

•AS SOURCES of proven aggregates are depleted throughout the country, more use will be made of aggregates that do not have a known service record. A new source of aggregate for Colorado may be the by-products of the oil shale industry. One of the major environmental problems facing this rapidly developing industry is the disposal of enormous amounts of spent shale from the oil extraction processes. Most research in this area has focused on revegetation efforts. However, spent shale has no organic material nor nutrients necessary for the growth of plants. Filling small canyons and other natural depressions with spent shale has also been proposed but has received less than favorable support. In view of this, the use of this material as aggregate for flexible pavement construction was investigated. Tests were performed to determine the suitability of one particular type of spent shale aggregate for possible use as base course material or in bituminous surface courses. Successful results could lead to an economic solution for both the processors of the raw oil shale and the users (construction agencies) of the seeming waste, spent shale.

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CHARACTERISTICS AND PROPERTIES OF SPENT SHALE

Generally, raw oil shale is a fine-grained, compact sedimentary marlstone, which may consist of dolomite, quartz, clay, calcite, and other minerals and which contains an organic substance called kerogen. Kerogen is only slightly soluble in known petroleum solvents but is readily converted to shale oil when subjected to temperatures between 700 and 950 F (371 and 510 C). As each particle of kerogen in the raw oil shale reaches its decomposition temperature in the retorting process, it vaporizes, leaving a void area in the shale. As heat continues to penetrate the shale, successive kerogen particles are vaporized and escape from the shale through pore spaces previously occupied with kerogen. The vapors are then condensed to yield the crude shale oil.

The spent shale is the solid waste remaining after retorting and other processing have removed hydrocarbon values and possibly associated mineral values from raw oil shale (1). The spent shale varies in particle size and chemical properties depending on the grade of raw oil shale and type of processing method used (2). Spent shale can range in size from a very fine ash (minus No. 200 sieve) to relatively large chunks (9 in., 229 mm, or more). The chemical analysis of Fischer assay spent shales obtained from raw Colorado oil shale samples is given in Table 1 (3). The constituents of burned shale are as follows:

Constituent	Average Percentage
SiO_2	43.8
Fe_2O_3	4.6
Al_2O_3	12.2
CaO	22.1
MgO	9.3
SO_3	2.2
Na_2O	3.4
K_2O	2.4

Except, perhaps, for its particle size, spent shale from the Fischer assay is generally representative of spent shale from any indirect heated retorting process.

In Table 2, Dinneen (4, 5) has reported the compressive strength of core samples of Green River oil shale before and after retorting. Samples of raw oil shale displayed high compressive strengths whether determined perpendicular or parallel to the bedding planes of the shale. After heating to 950 F (510 C), the lean spent oil shale retained relatively high compressive strength values in both horizontal and vertical planes, indicating a high degree of inorganic cementation between the mineral particles making up each lamina and between adjacent laminae. It is also evident from Table 2 that the compressive strength of rich (high oil yield) spent oil shale is quite low after the organic matter (kerogen) is removed.

A study (6) conducted by the Denver Research Institute (DRI) in 1966 discussed the disposal and uses of spent shale ash (material passing No. 200 sieve). A significant result of this study was the development of cohesion in the shale ash, when it was subjected to burning temperatures from 1,150 to 1,700 F (521 to 927 C) and burning times from ½ sec to 2 hours, and the resulting high compressive strengths of the shale ash test specimens. However, most processing methods are conducted with burning temperatures ranging from 700 to 950 F (371 to 510 C), resulting in spent shale that is nonplastic and noncohesive, e.g., the material investigated by the author. Another DRI study (7) on spent oil shale ash showed that this material can possibly be used as fines in asphaltic concrete, as a soil-cement additive for road base and subbase material, and as lightweight aggregate for highway use.

Other researches (8, 9, 10) have obtained data on spent shale (particle size ranging from plus No. 4 through minus No. 200 sieves, with the majority passing the No. 200

sieve) in the form of compaction characteristics, Atterberg limits, consolidation, and triaxial characteristics. Their conclusions (10) are given below.

1. The spent shale material loses strength significantly upon saturation. For partially saturated spent shales (moisture content between 5 and 20 percent), the strength increases with an increase in dry densities. However, upon saturation, the strength seems to be independent of magnitude of dry densities in the range tested.
2. The angle of internal friction of saturated spent shales is on the order of 20 deg; that for the partially saturated spent shales was 35 deg.

These relatively high angles of internal friction usually indicate a material that may exhibit a relatively high frictional component of shear strength (a characteristic that is very desirable in flexible pavement structures).

It is readily apparent from this discussion that much of the research reported to data has concentrated on so-called spent shale ash material. This research has shown that this material does possess some of the desirable characteristics of the more traditional forms of aggregate in construction use. However, the use of large quantities of material with such small particle sizes and limited particle size range is not very practical for large construction operations, e.g., earth embankments, and flexible pavements. Little if any research has been conducted on larger sizes and a wider range of sizes of spent shale. Consequently, the remainder of this paper is devoted to an evaluation of a limited number of physical characteristics of relatively coarser material (with only 10 percent of it, classified as ash, passing the No. 200 sieve).

EVALUATION AND DISCUSSION OF SPENT SHALE AGGREGATE AND MIXES

Methodology

The spent shale investigated in this study was obtained from the U.S. Bureau of Mines, Laramie Energy Research Center (LERC), in Wyoming. The raw oil shale in use at LERC originated from the Green River formation in which the Colorado shale field is located (11). The retorting process in use at LERC is the Nevada-Texas-Utah (NTU) process, which uses very large pieces of raw oil shale. Approximately 20 percent of the raw oil shale used is larger than 20 in. (508 mm) in size. Some typical chemical properties of the spent shale from the NTU retort at LERC are given in Table 3 (12).

To evaluate the suitability of this material for use in road construction, the following tests were performed with the cooperation of the Materials Division of the Colorado Division of Highways (CDH): the Los Angeles abrasion test, dry sieve analysis, specific gravity, Atterberg limits, and the Hveem method of mix design. These tests were used to determine the following aggregate characteristics: size and grading, toughness, particle shape, surface texture, absorption, and affinity for asphalt. These basic aggregate characteristics combine to yield the desirable properties that asphalt-aggregate and aggregate mixes must possess to perform satisfactorily as pavement mixtures. The desirable properties of pavement mixtures (13) include stability, durability, flexibility, fatigue resistance, skid resistance, impermeability, and workability.

Size, Grading, and Atterberg Limits

The material received from LERC ranged in size from approximately 9 in. (229 mm) to less than 0.0029 in. (0.07 mm, minus No. 200 sieve); the majority was less than 1 in. (25 mm). For convenience in handling, the material was passed through a small rock crusher set at a maximum size of ¾ in. (19 mm) before the dry sieve analysis was performed. The gradation curve shown in Figure 1 conforms to the Asphalt Institute's type 4-B (14) and CDH's E grading for asphalt-concrete surface course mixtures. This

Table 1. Chemical analyses of spent shales from Fischer assays conducted on Colorado oil shale samples.

Constituents	Grade of Original Oil Shale Sample 17.8	19.5	22.3	29.8	36.6	38.0	51.8
Carbon (total)	7.26	7.62	8.56	8.12	10.27	10.07	10.32
Carbon (organic)	1.81	1.69	2.49	2.57	3.53	4.20	4.55
Hydrogen	0.26	0.22	0.15	0.24	0.27	0.22	0.26
Nitrogen	0.11	0.10	0.18	0.21	0.24	0.27	0.33
Sulfur	0.37	0.29	0.50	0.61	0.47	0.66	1.06

Table 2. Compressive strengths of raw and thermally treated oil shales (in psi).

Oil Yield (liters/ metric ton)	Raw Oil Shales		Oil Shale Heated to 510 C		Oil Shale Heated to 816 C	
	A	B	A	B	A	B
4.2	22,168	19,382	19,979	19,084	15,489	14,892
27.1	31,177	28,377	28,178	26,175	17,080	13,585
56.3	26,573	23,375	13,286	6,196	10,487	3,695
104.3	18,388	18,587	412	384	171	156
125.2	9,890	10,586	355	284	114	114
164.8	11,496	13,770	71	114	85	114
244.1	12,988	2,889	28	28	57	28

Note: A = compressive strength perpendicular to the bedding planes; B = compressive strength parallel to the bedding planes. 1 psi = 6900 Pa.

Table 3. Properties of spent shale.

Shale Analysis	Experiment 1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Fischer assay																		
Oil, gal/ton	16.2	7.6	4.92	0.1	0	0.3	0.8	0	0	1.2	1.31	0	0	0	0	0.95	0.3	0
Oil, wt percent	6.2	2.9	1.81	0.05	0	0.1	0.32	0	0	0.47	0	0	0	0	0	0.38	0.1	0
Water, gal/ ton	1.3	1.1	0.63	0.9	0.8	1.9	2.0	0.08	0	1.7	0.38	0.2	1.1	1.4	1.4	0.5	2.2	0.8
Hydrogen, wt percent	1.17	0.56	0.51	0.15	0.14	0.14	0.34	0.11	0.06	0.31	0.24	0.14	0.18	0.22	0.17	0.23	0.20	0.14
Carbon																		
Total wt percent	13.23	8.78	8.04	4.87	4.99	5.53	5.14	4.04	2.85	6.34	5.36	4.00	6.02	7.12	4.33	7.19	2.41	3.62
Mineral wt percent	5.02	4.76	2.99	3.20	3.34	3.46	3.09	2.70	1.13	3.40	2.52	2.67	4.05	3.99	2.86	3.48	1.59	2.19
Organic wt percent	8.21	4.02	5.05	1.67	1.65	2.07	2.05	1.34	1.70	2.94	2.83	1.33	1.97	3.14	1.46	3.71	0.82	1.43
Carbon dioxide, wt percent	18.39	17.46	10.97	11.73	12.27	12.67	11.14	9.88	3.72	12.46	9.26	9.77	14.83	14.61	10.49	12.12	5.84	8.02
Ash, wt percent	71.71	77.46	83.69	86.87	87.30	84.33	85.82	88.77	95.61	83.04	88.34	88.67	82.91	82.29	87.62	83.95	92.82	90.61
Nitrogen, wt percent	0.32	0.17	0.21	0.12	0.12	0.10	0.12	0.09	0.06	0.19	0.13	0.08	0.12	0.14	0.08	0.21	0.08	0.09
Sulfur, wt percent	0.52	0.54	0.97	1.28	0.93	0.56	0.75	0.94	0.86	0.60	0.82	0.66	0.70	0.67	0.73	0.82	0.98	0.74
Mineral carbon dioxide, wt percent	25.08	23.81	14.96	15.99	16.73	17.27	15.19	13.47	4.88	16.99	12.63	13.32	20.22	19.92	14.30	16.49	7.96	10.94
Water soluble carbonates, wt percent	0.23	1.21	4.53	3.43	2.21	0.95	0.99	1.40	3.88	1.23	2.09	0	1.82	0.22	1.31	1.14	1.44	1.40
Water soluble bicarbonates, wt percent	0.32	0.17	0.23	0.07	0.94	0.08	0.07	0.08	0.11	0.07	0.06	1.60	0.08	0.52	0.74	0.07	0.08	0.12
Heat value, Btu/lb (gross)	1,490	610	490	90	150	300	170	40	160	300	380	140	160	410	70	500	70	140

Note: 1 gal/ton = 3800/907 cm^3/kg; 1 Btu/lb = 2326 J/kg.

Figure 1. Gradation curve for spent shale aggregate.

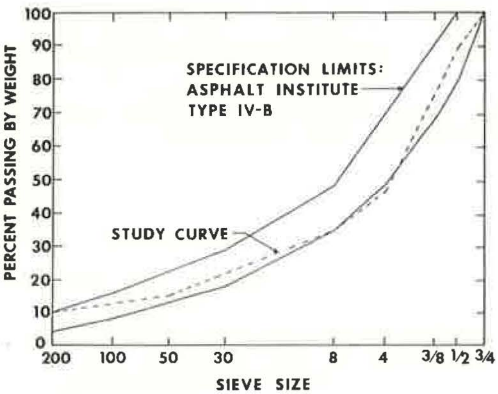

Table 4. Hveem test results.

Percent Bitumen	Specific Gravity of Specimen	Stability Value	Cohesiometer Value	R_t
11	1.831	39	199	98
12	1.851	36	284	100
13[a]	1.896	18	276	86
14	1.884	–	174	–

[a]Maximum specific gravity of 1.950, and voids in compacted specimen of 2.7 percent.

gradation can be described as a relatively dense, well-graded, aggregate blend that should exhibit high strength characteristics.

The Atterberg limits tests (liquid and plastic limits) were performed on a portion of the material passing the No. 40 sieve. The results from these tests showed that the material was nonplastic and thus noncohesive.

Toughness

The Los Angeles abrasion test was conducted to determine the toughness or the abrasion resistance of the spent shale particles. The wear was 48.5 percent. This is slightly higher than the maximum allowable limit (45 percent) reported in the CDH standard specifications. Consequently, this material may not stand up so well as tougher aggregates (such as traprock) to the repeated abrasive action of traffic. Therefore, the spent shale particles should probably be used in the lower pavement layers (binder, base, and subbase courses).

Particle Shape and Surface Texture

Particles retained on the No. 50 sieve are dark gray, friable, flat, angular, and sharp-cornered and have a relatively rough surface texture. Microscopic examination of smaller sizes (minus No. 200) similar to those of this study was reported by Heley and Terrel (8), who found that

1. The particles were crystalline and bulky;
2. Individual particles varied in size, and the larger particles were created by a fusion together of many smaller particles; and
3. The particles are not honeycombed in structure, but large amalgamations do contain many interstices that, because of their fused structure, are possibly isolated from surface fluids.

These characteristics are typical of aggregates that result in relatively high angles (35 to 45 deg) of internal friction. Although a determination of this angle was not performed for the material from LERC, recent research (10) indicates that this angle for spent shale ash can vary from 20 deg for saturated triaxial specimens to 35 deg for partially saturated specimens [moisture contents ranging from 5 to 20 percent and dry densities from 85 to 95 lb/ft^3 (1360 to 1520 kg/m^3)].

Absorption

The centrifuge kerosene equivalent (CKE) test was performed on the spent shale aggregate to determine the estimated oil ratio to use for the subsequent Hveem mixtures. Both the kerosene and oil used to determine the degree of absorption showed a relatively high percentage of porosity; approximately 15 percent of each (kerosene and oil) was retained. The probable reason for this high porosity is the high temperatures used in the retorting process on the raw oil shale as mentioned previously. This characteristic will more than likely necessitate adding an extra amount of asphalt to the paving mix to satisfy the aggregates' seemingly high absorption capacity.

Affinity for Asphalt

Stripping, the separation of asphalt film from the aggregate through the action of water, may make an aggregate unsuitable for asphalt paving mixtures. Immersion-compression tests were performed on asphalt-spent shale mixtures containing 13 percent asphalt. The results were as follows:

Item	Value
Wet strength, psi	359
Dry strength, psi	357
Percentage of absorption by weight	1.36
Percentage of swell by volume	0.00
Index of retained strength	100

These results show a negligible loss of strength, indicating that the spent shale exhibits very high resistance to stripping.

Strength of Spent Shale Aggregate Mixtures

In general, soil strength depends on density, moisture content, and texture of the soil (15). The Hveem stabilometer test was performed to evaluate the shear strength (frictional component) of typically prepared spent shale aggregate specimens.

The stabilometer R-value, expressing the measure of a soil or aggregate's ability to resist the transmission of vertical load in a lateral or horizontal direction, was determined for moist spent shale aggregate specimens and was found to be 85 (average of three tests, all of which were the same value). The associated density and moisture content were 93 lb/ft^3 (1488 kg/m^3) and 18.1 percent. The main reasons for this high strength have been previously discussed. An R-value of 85 significantly exceeds the minimum value of 78 (16) for aggregate base course layers. This indicates that use of spent shale as conventional base course material should be highly recommended. Because of its relatively high strength, more of this material could be used to provide thicker base course layers, probably resulting in thinner asphalt-concrete surface courses for many roads.

In summary, the spent shale is well graded yielding dense mixtures; is flat, angular, and of rough surface texture yielding a relatively high angle of internal friction and therefore high strength in compaction specimens; exhibits very high resistance to stripping; and is a highly absorbent and friable material that appears to wear relatively easily.

Hveem Asphalt-Spent Shale Mixtures

The Hveem method of mix design, typically used to determine the probable optimum asphalt content for pavement mixtures, was used to evaluate the suitability of asphalt-spent shale mixtures. The principal features of this method are (13) the CKE test on the aggregates, a stabilometer test, a cohesiometer test, a swell test, and a density-voids analysis on compacted paving mixtures. The CKE test was performed on samples of spent shale (both plus No. 4 and minus No. 4 sieve sizes) to determine the estimated optimum asphalt content, which was 14 percent. Four Hveem samples ranging in asphalt content from 11 to 14 percent were then made. The results given in Table 4 indicate some interesting characteristics. First, it appears that the optimum asphalt content for maximum stability is slightly greater than 11 percent. This at first seems high when compared to a more normal value of 6 percent for most surface course mixes. However, when the specific gravities of spent shale and normal aggregate are compared (1.85 versus 2.35 respectively) the seemingly high asphalt content of 11 percent for the spent shale aggregate is equivalent to 9 percent for normal aggregate. Second, the total resistance value R_t (combination of stability and cohesiometer value) of approximately 100 is surprisingly high. This seems to indicate that the asphalt-spent shale mix possesses very high strength, i.e., more than adequate load-carrying capability.

It is evident from the results shown in Table 4 that the mixtures investigated possess extremely high stability. This is due to dense aggregate gradation, angular shape, and rough surface texture, all of which contribute to a high angle of internal friction and thus to a high frictional component of shear strength, and a relatively high value of cohesion exhibited by the mixture.

Only a partial evaluation can be made at this time of mixture durability, its ability to resist disintegration from weathering and traffic. The results from the Los Angeles abrasion test show that the spent-shale itself may not stand up so well to the repeated abrasive action of traffic. The soundness test, which provides an indication of the resistance to weathering of aggregate, was not performed. Traditionally, however, the shale type of material is unsound because water enters it and, upon freezing, expands and fractures it (14). Hence, based on this limited information the spent shale probably should not be used for surface course paving mixtures.

Because of the limited testing, the property of flexibility cannot be fully assessed. This property is, however, enhanced by high asphalt contents and relatively open-graded aggregates. The asphalt contents tested were high, but the gradation was dense. Because the spent shale is highly absorbent, it is not known how much of the asphalt is absorbed nor how much effectively coats the aggregate. This question might be resolved in a future study.

Fatigue resistance is also enhanced by high asphalt contents and well-graded aggregates. The mixes studied conform to these criteria; consequently, they might exhibit more than adequate fatigue resistance.

The cohesiometer test determines the cohesion properties and the tensile strength of asphalt films. The cohesiometer values obtained in this investigation, near the estimated optimum, are relatively high and substantially exceed the minimum values reported by most design criteria (13). The higher the tensile strength is, the greater are the properties of flexibility and fatigue resistance. Thus, with the relatively high cohesiometer values obtained, it seems likely that the asphalt-spent shale mix might possess more than adequate flexibility and fatigue resistance.

The same factors that govern high stability usually apply to high skid resistance. From the preliminary testing conducted, it seems likely that the spent shale does possess the rough surface texture and angular particle shape necessary for adequate skid resistance.

The results from the immersion-compression test provide an excellent indication of the relative impermeability of the asphalt-spent shale mixture. The absorption of water by weight was slightly greater than 1 percent. This indicates that the mix used is relatively dense graded and impermeable.

Visual examination revealed that the asphalt-spent shale mixtures were difficult to mix. The problem of adequate workability in the field, however, cannot accurately be assessed at this time. This also might be considered in a future investigation.

Although the asphalt-spent shale mixtures possess many of the desirable properties discussed above, its use as a surface layer for high types of roads is questionable. However, these mixtures might perform very well in surface courses for lightly traveled roads (e.g., secondary roads). It seems more likely that these mixtures might be highly beneficial for use in binder layers and also in bituminous base layers.

RECOMMENDATIONS AND CONCLUSIONS

Even though this investigation was very limited in scope, it indicates the suitability of this particular type of spent shale for use in binder layer, bituminous base, conventional base, or subbase layers for flexible pavements. Although the spent shale showed more than adequate strength and stability, its use as aggregate for asphalt concrete surface course mixtures may be dependent on the traffic characteristics of the road involved. Field installations using this material would have to be constructed to evaluate the performance of this material more completely.

Based on the results of the tests performed, the following conclusions are made.

1. Spent shale can be described as bulky, angular, rough, and highly absorptive material capable of producing very high strength, relatively lightweight aggregate mixtures.

2. Asphalt-spent shale mixtures tested by the Hveem method show that very high strengths can be obtained with this material. However, the asphalt contents required to produce these strengths are high.

3. The results of stabilometer tests on aggregate mixtures also indicated very high strengths, pointing to the possible use of this material for base and subbase courses.

4. Based on this limited investigation, this particular type of spent shale material might perform very well in flexible pavement structures.

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