Properties of Asphalt-Oil Shale Ash Bituminous Mixtures under Normal and Freeze-Thaw Conditions

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In the last decade Jordan has given serious consideration to the use of oil shale as an alternative energy source. Such use will be accompanied by the production of a large amount of oil shale ash. Possible areas of application for this byproduct are under investigation, including its possible use with asphalt concrete mixes under different environmental conditions. Many researchers have indicated that it is possible for a mineral filler (fly ash, dust, etc.) to act as an asphalt extender or substitute binder for paving construction. The main objective of this study was to evaluate quantitatively the use of oil shale ash as a partial substitute for the asphalt binder in bituminous paving mixtures under normal conditions as well as under freezing and thawing conditions. Oil shale ash was added to the asphalt concrete mix at optimum asphalt content. Five ash levels, ranging from 0 to 20 percent by volume of asphalt, were used in the study. For each level Marshall and indirect tensile tests were performed under both normal and freeze-thaw conditions. The test results indicated that the substitution of ash up to 10 percent by volume of asphalt would improve the performance of mixtures under both conditions. Such ash inclusion resulted in paving mixtures with higher Marshall stability, greater tensile strength, a larger stiffness modulus, and higher resistance to freeze-thaw damage in comparison with mixtures without ash.

In view of the increasing demand for energy (now dependent mostly on imported oil) and recognizing the fact that Jordan has larger reserves of oil shale rocks, interest has increased in using such rocks as a new source of energy. One of the problems associated with such a practice is the formation of large quantities of oil shale ash. Directing research toward the use of such a byproduct has similarly gained interest. The oil shale ash is considered a low-cost binding material that could be used in many areas, such as building construction, pavements, agriculture, and chemical industries.

The purpose of the present study is to examine the efficiency of using oil shale ash as a substitute or supplement for asphalt cement under both normal and freeze-thaw conditions. The basic engineering properties of the mixture, including Marshall stability, stiffness modulus, and tensile strength, under both conditions will be determined. Limestone and five levels of oil shale ash were used in the study.

LITERATURE REVIEW

Several researchers have investigated the role of different extender/filler materials on the performance of asphalt concrete mixtures. Some of the studies explained the role played by mineral fillers in general (*1–4*), and others investigated the effect on asphalt concrete of specific materials, such as fine dust (*5–7*), fly ash (*8, 9*), and oil shale ash (*10*).

In a study by Puzinauskas (*1*), it was stated that mineral fillers fill the interstices and provide contact points between larger aggregate particles. As a result, such fillers form a high-consistency binder or matrix that cements the aggregate particles together. In addition, Winniford (*2*) concluded that the role of mineral fillers includes the formation of a thick viscous coating that increases the non-Newtonian flow characteristics and lowers the susceptibility to temperature of asphalt. Furthermore, Heithaus and Izatt (*3*) have found that the addition of filler material increases the stability of asphalt mixutre up to a certain point, after which stability is decreased. Similarly, Oglesby (*4*) has indicated that the addition of asphalt binder and mineral filler increases the cohesion resistance and stability of the mixture.

The West Virginia Department of Highways (*5*) conducted a study on the influence of baghouse fines and fine dust on paving mix. It was concluded that, when baghouse fines are used in proper quantity, they can improve the stability and cohesion of the mix. In the same study, the finer sizes of dust were found to act as asphalt extender when the proper amount and size are used. Similar conclusions on fine dust were also reached by Anderson and Tarris (*7*).

The effect of fly ash on the properties of asphalt concrete mixtures has been also investigated (*8, 9*). In a study by Tons et al. (*8*), it was found that replacing up to 30 percent of asphalt with fly ash would increase the Marshall stability and the tensile strength and improve the resistance of asphalt concrete to freeze-thaw damage. Likewise, Bolk et al. (*9*) concluded that fly ash fillers give larger creep stiffness than limestone fillers.

Finally, while several studies have been made on the different fillers as just stated, limited information is available on the role played by oil shale ash on asphalt concrete. In a preliminary study by Khedaywi (*10*), it was stated that substituting 10 percent of asphalt with ash can increase the stability and cohesion of the mixture.

MATERIALS USED

- Asphalt: One asphalt cement (80/100) that is widely used in pavement construction in Jordan was used in the study. Table 1 gives the properties of the asphalt cement used.

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TABLE 1 PROPERTIES OF ASPHALT CEMENT USED IN THE STUDY

Properties	ASTM Test Designation	Test Result
Specific gravity at 77°F	D 70	1.010
Penetration ,(0.1mm) 77°F,100 gm,5sec	D 5	83
Softening Point (°F) Ring and Ball	D 36	125
Ductility (Cm) at 77°F	D 113	130
Kinematic Viscosity (Cst) 275°F	D 2170	310

Note: 1mm = 0.0394 in.

TABLE 2 AGGREGATE GRADATION USED IN THE STUDY

Sieve Size	% Passing	Specification[a]
3/4 in.	100	100
1/2 in.	95	90-100
3/8 in.	85	75-90
# 4	60	45-70
# 8	45	33-53
# 30	25	15-33
# 50	15	10-20
# 100	7	4-16
# 200	5	2-9

Note: 1 in. = 2.54 cm
[a]Jordan Specification Limits.

- Aggregate: One crushed limestone aggregate was used in the study. Its gradation conformed to the Ministry of Public-Works specifications in Jordan. The aggregate gradation and its properties are given in Tables 2 and 3, respectively.
- Oil shale ash: The ash used in the study was obtained by burning oil shale rock at about 600°C. It was supplied by the Natural Resources Authority, Jordan. The mineral composition of oil shale ash is given in Table 4, and Table 5 shows the ash gradation by hydrometer analysis on selected ash samples.

TEST PROCEDURE AND PHASES OF INVESTIGATION

A Marshall test (ASTM D1559) and an indirect tensile test were performed on the laboratory-made specimens with 50 blows on each face. The specimen was 4 in. in diameter and 2.5 in. high. There were three phases of investigation.

Phase 1: Marshall Test Using ASTM D1559

The objective of this phase was to determine the optimum asphalt content by weight of the total mix (conventional bituminous mix). The only variable in this phase was the asphalt content by total weight of the mix. Five levels of asphalt content were tried: 4.5, 5, 5.5, 6, 6.5, and 7 percent. The aggregate weight was 1,200 g. The procedure used in preparing and testing the bituminous mixture Marshall specimens is that outlined in Asphalt Institute Manual MS-2 (*11*). Table 6 and Figures 1–5 show the results of this phase.

Phase 2: Freeze-Thaw-Induced Damage, Using Marshall Statibility Test

The specific objective of this phase was to evaluate quantitatively the effect of ash on the behavior of bituminous mixes using the Marshall test, under normal dry conditions and under wet conditions after 10 cycles of freezing and thawing.

During the course of this study, it was found that the replacement of more than 20 percent (by volume) of optimum asphalt (determined in Phase 1) by ash produces an unworkable mix. The variables in this part of the study were

1. The Percent Ash by Volume of Binder

(1) 0 (2) 5 (3) 10 (4) 15 (5) 20

The volume of asphalt plus the volume of ash were kept constant and were equal to the volume of optimum asphalt content (in Phase 1).

2. The curing condition, where the prepared specimens were set into two groups:

(a) The first group of specimens was cured in air at room temperature for 10 days.

(b) The second group of specimens was subjected to 10 cycles of freezing and thawing. They were put in plastic freezing bags, and about 10 ml of water was added to each bag (*8*); they were kept in a freezer at −18°C for 16 hr followed by 8 hr of thawing at 25°C. The indicated freeze-thaw periods were adopted to minimize the work and for practical performance in the locality.

Preparation of Ash-Asphalt Binder

The ash was heated in a stainless steel beaker at 145–150°C. Asphalt cement was heated in an oven at 145°C. The required amount of asphalt and ash was weighed and mixed at 145°C. The laboratory mixer was used to attain a homogeneous binder mix; mixing continued for 5 min at 1,600 rpm. Directly after

TABLE 3 PROPERTIES OF AGGREGATE USED IN THE STUDY

Type of Aggregate	ASTM Test Designation	Bulk Specific Gravity	Apparent Specific Gravity	Absorption (%)
Limestone Coarse Aggregate	C-127	2.42	2.68	3.10
Limestone Fine Aggregate	C-128	2.45	2.71	3.65
Limestone Mineral Filler	C-128	2.55	2.79	4.00
Oil Shale Ash	C-188	----[a]	2.35	----[a]

[a]Data Not Applicable.

TABLE 4 CHEMICAL PROPERTIES OF OIL SHALE ASH[a]

Silica	SiO_2	32.43
Calcium	CaO	41.96
Alumina	AL_2O_3	7.19
Iron	Fe_2O_3	2.83
Magnesium	MgO	1.89
Phosphate	P_2O_5	5.04
Potassium	K_2O	0.58
Sodium	Na_2O	0.3
Titanium	TiO_2	6.7
Sulfate	SO_3	4.49

[a]Department of Natural Resources.

TABLE 5 OIL SHALE ASH GRADATION IN THE STUDY[a]

Size , Micron	Percent Finer
75	100
56	97
21	90
20	80
8	75
3	25
1	10

Note : 1 μm=0.0394X10^{-3} in.

[a]Hydrometer Analysis

mixing, the binder was added to the heated aggregate, and the specimens were prepared according to MS-2.

For each percentage, three specimens were prepared. Therefore 15 specimens for each group were made.

Marshall Stability

All specimens were cured at a constant temperature of 25°C for 24 hr, after which a Marshall test was conducted at 60°C. Table 7 gives the weights and volumes of asphalt and oil shale ash of the specimens used in Phase 2, and their results are presented in Table 8.

Phase 3: Freeze-Thaw-Induced Damage, Using Indirect Tensile Test

The specific objective of this phase was to evaluate quantitatively the effect of ash on the behavior of bituminous mixes using an indirect tensile test, under normal conditions and under wet conditions after 10 cycles of freezing and thawing.

The indirect tensile test involved loading a cylindrical specimen with compressive loads that were set parallel to and along the vertical diametrical plane (*12*). Marshall specimens measuring 4 in. in diameter and 2.5 in. high were used in the study. To distribute the load and maintain a constant loading area, the compressive load was applied through a 0.5-in.-wide,

TABLE 6 MARSHALL PROPERTIES OF MIXTURES WITHOUT THE OIL SHALE ASH (RESULTS OF PHASE I)

Asphalt Content[a] %	Marshall Stability (lb),60°c	Unit Weight (PCF)	Flow (0.01 in.)	V.T.M[b] %	V.F.B[c] %
4.5	1960	132.10	12	9.10	45
5.0	2520	133.95	12.75	7.10	57
5.5	2780	134.80	13.8	4.65	72
6.0	2750	134.41	15.2	3.40	80
6.5	2590	133.52	17.4	2.00	88
7.0	2190	132.55	19.0	1.32	92

[a]Asphalt Content, Percent by total weight of the mix.
[b]V.T.M: Air Voids in the mix, Percent by total Volume .
[c]V.F.B: Percent of Voids Filled with bitumen.

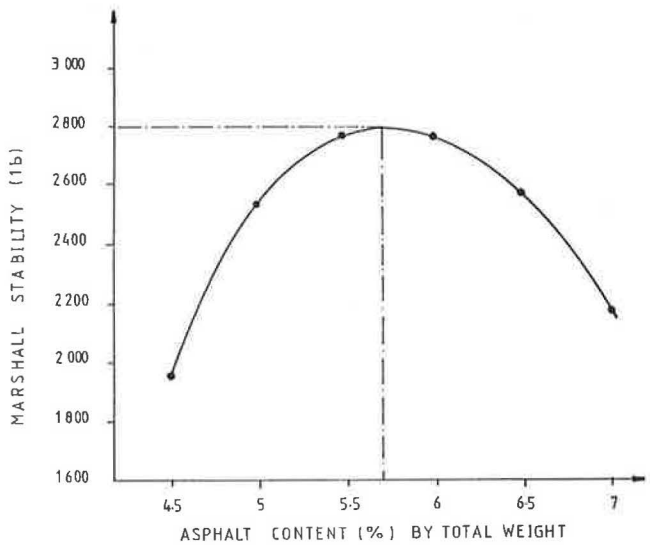

FIGURE 1 Relationship between asphalt content and Marshall stability.

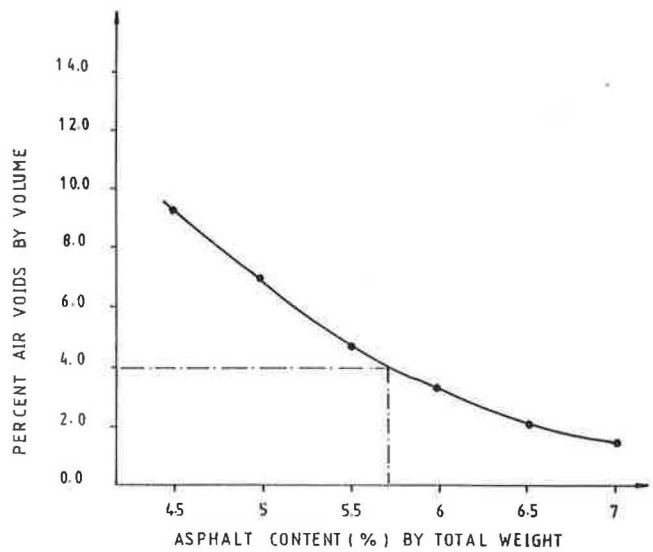

FIGURE 3 Relationship between asphalt content and air voids.

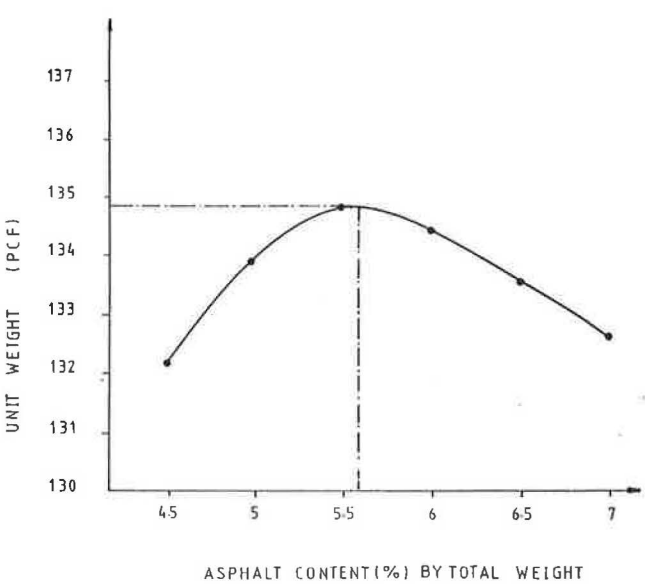

FIGURE 2 Relationship between asphalt content and unit weight.

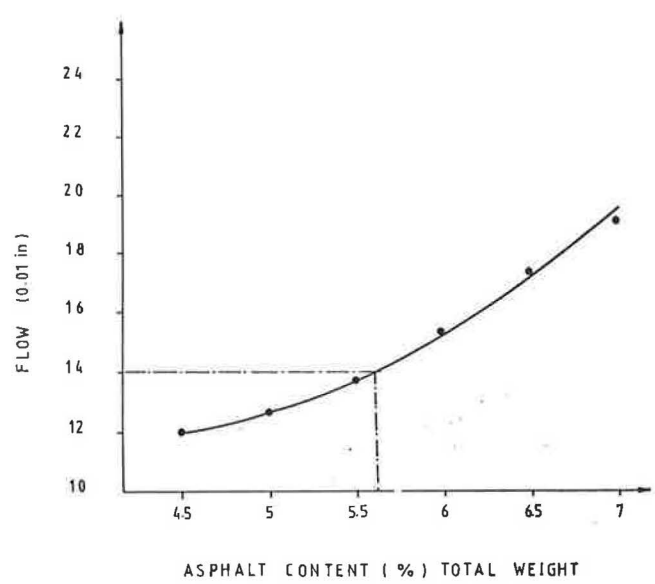

FIGURE 4 Relationship between asphalt content and flow.

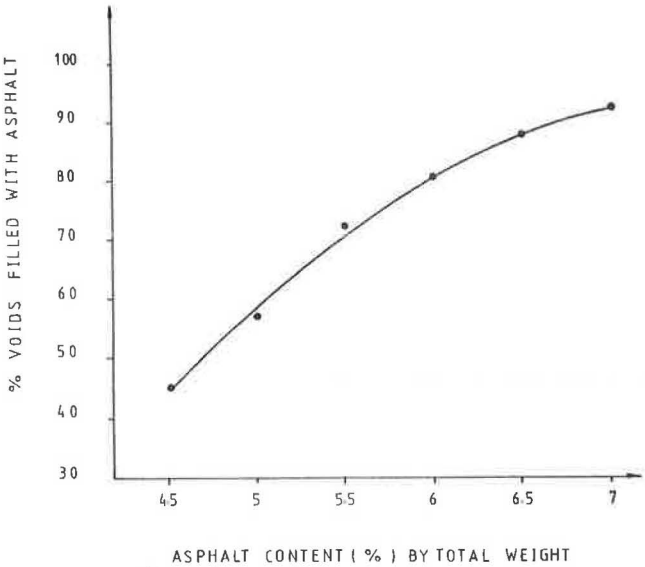

FIGURE 5 Relationship between asphalt content and percent voids filled with asphalt.

steel loading strip that was curved at the interface with the specimen and had a radius equal to that of the specimen.

The theoretical relationships used in calculating the specimen tensile strength (ST), Poisson ratio (ν), and stiffness modulus (SF) are as follows (*12*):

$$\text{tensile strength } (ST), \text{ psi} = 0.156\frac{P_{\text{fail}}}{h} \quad (1)$$

$$\text{Poisson's ratio } (\nu) = \frac{0.0673DR - 0.8954}{-0.2494DR - 0.0156} \quad (2)$$

$$\text{stiffness modulus } (SF), \text{ psi} = \frac{SH}{h}(0.9976\,\text{v} + 0.2692) \quad (3)$$

where

P_{fail} = total load at failure in pounds;
h = height of specimen in inches;
DR = deformation ratio YT/XT = the slope of the line of best fit between vertical deformation YT and the corresponding horizontal deformation XT in the linear portion only; and

TABLE 7 WEIGHTS AND VOLUMES OF ASPHALT AND OIL SHALE ASH FOR EACH MARSHALL SPECIMEN (DATA FOR PHASE II)

Ash Content[a] %	Binder Volume[b] (cm³)	Ash Volume (cm³)	Asphalt Volume (cm³)	Ash Weight (gm)	Asphalt Weight (gm.)	Total Weight of mix (gm.)
0	71.8	0.00	71.80	0.0	72.50	1272.50
5	71.8	3.59	68.21	8.44	68.89	1277.33
10	71.8	7.18	64.62	16.87	65.27	1282.14
15	71.8	10.77	61.03	25.31	61.64	1286.95
20	71.8	14.36	57.44	33.75	58.01	1291.76

[a]Ash content ,Percent by total Volume of Binder

[b]Binder Volume Kept Constant = Ash Volume + Asphalt Volume.

TABLE 8 STABILITY, AIR VOIDS, FLOW, AND UNIT WEIGHT FOR ASH-ASPHALT CONCRETE MIXES (RESULTS OF PHASE II)

Ash Content[a] %	V.T.M[b] %	Flow (.01 in.)	Unit Weight (PCF)	Stability (lb) Dry	Stability (lb) Wet[c]	Index of Retained Marshall Stability (Wet/Dry)
0	4.0	14.2	134.5	2770	2240	0.81
5	3.8	13.5	135.4	3185	2880	0.904
10	3.9	13.0	136.0	3500	3240	0.926
15	4.2	12.6	135.6	3450	3160	0.916
20	5.10	11.5	134.9	3210	2825	0.88

[a]Ash Content,Percent by total Volume of binder.

[b]Voids in the total mix

[c]Stability of Specimens after 10 cycles of Freezing and Thawing

SH = horizontal tangent modulus P/XT = the slope of the line of best fit between P and XT in the linear portion only.

The variables, preparation of ash-asphalt binder, preparation of specimens, groups of specimens, number of specimens, and curing method were the same as in Phase 2. All specimens were kept at a constant temperature at 25°C for 24 hr before the indirect tensile test, which was performed at 25°C. Table 9 presents the calculated data for this phase.

TEST RESULTS AND DISCUSSION

In Phase 1, mixes were made with pure asphalt (without any addition of oil shale ash) to determine the optimum binder content (asphalt). The Marshall test results are shown in Figures 1 to 5 (see also Table 6). The results indicated that the optimum asphalt content is 5.7 percent by total weight of bituminous mix, with 4 percent air voids, a flow of 14, and stability of 2,780 lb.

In Phase 2, mixes were made by replacing part of the asphalt with oil shale ash on the basis of volume. The binder content was kept constant (volume of ash + volume of asphalt = volume of binder = volume of asphalt at optimum asphalt content for pure asphalt mixes in Phase 1). The Marshall test results of the first group under dry conditions and of the second group under wet conditions after 10 cycles of freezing and thawing indicated that the addition of ash resulted in a substantial increase in stability, up to 10 percent by volume of asphalt, and then decreased, as shown in Figure 6. The difference in Marshall stabilities between the first and second groups (dry and wet conditions) is minimum at 10 percent ash and maximum at 0 percent ash. The sample with 10 percent ash tested in wet conditions produced much more stability than the sample with 0 percent ash tested in dry conditions, as shown in Figure 6. Figure 7 indicates (see also Table 8) that the index of retained Marshall stability is maximum at 10 percent ash (93 percent) and minimum at 0 percent ash (81 percent).

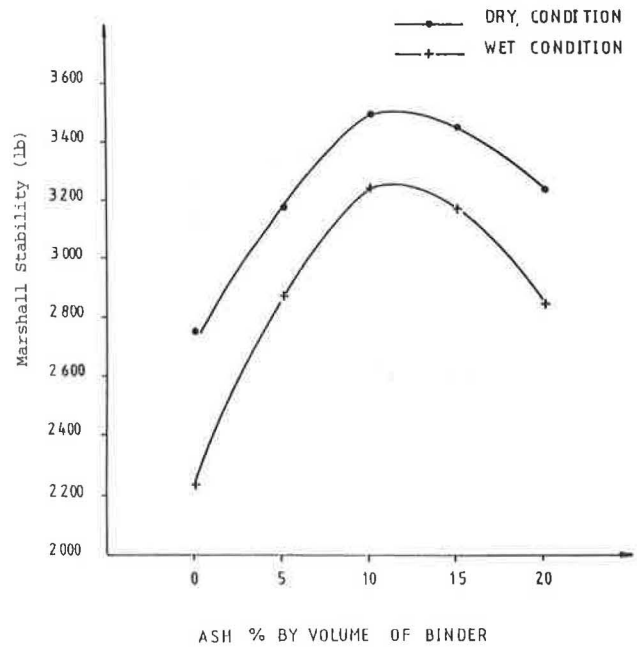

FIGURE 6 Effect of replacing asphalt with oil shale ash on Marshall stability in dry and freeze-thaw conditions.

TABLE 9 INDIRECT TENSILE STRENGTH TEST AT 25°C (RESULTS OF PHASE III)

Ash (%)	Condition	Properties: Load at Failure Fail(lb)	Poisson's Ratio ν	Tensile Strength ST(Psi)	Stiffness Modulus SF,(Psi)	Index of retained: Tensile Strength	Stiffness Modulus
0	Dry[a]	1685.1	0.336	105.15	$3.49*10^4$	0.812	0.911
	Wet[b]	1368.6	0.340	85.40	$3.18*10^4$		
5	Dry	1872.3	0.335	116.83	$3.74*10^4$	0.862	0.912
	Wet	1614.4	0.337	100.74	$3.41*10^4$		
10	Dry	2026.4	0.332	126.45	$3.85*10^4$	0.911	0.956
	Wet	1846.6	0.334	115.23	$3.68*10^4$		
15	Dry	1937.0	0.330	120.87	$3.78*10^4$	0.90	0.942
	Wet	1746.8	0.332	109.00	$3.56*10^4$		
20	Dry	1836.0	0.325	114.57	$3.65*10^4$	0.90	0.926
	Wet	1661.1	0.330	103.65	$3.38*10^4$		

[a] Specimens were cured at 25°c for 11-days and tested at 25°c.
[b] Specimens were subjected to 10-cycles of freeze thaw and tested at 25°c after 24 hours at 25°c.

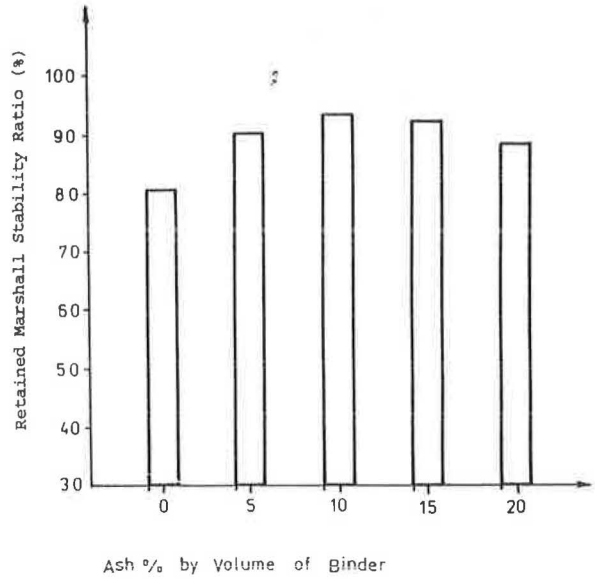

FIGURE 7 Relationship between oil shale ash and retained Marshall stability.

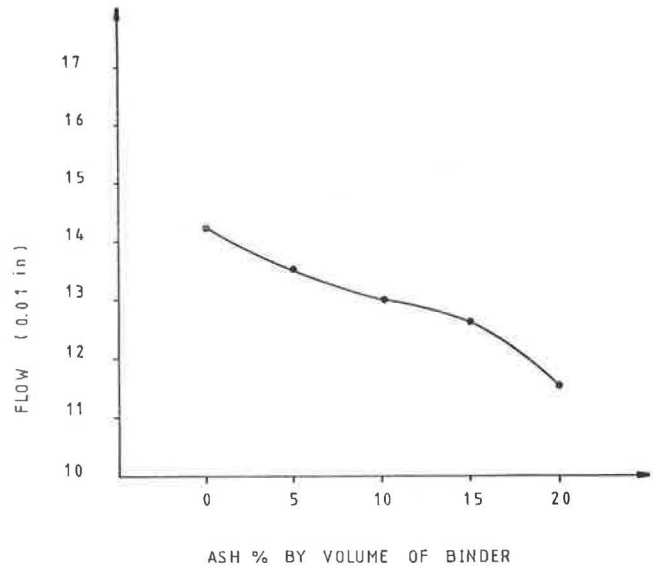

FIGURE 9 Effect of replacing asphalt with oil shale ash on Marshall flow.

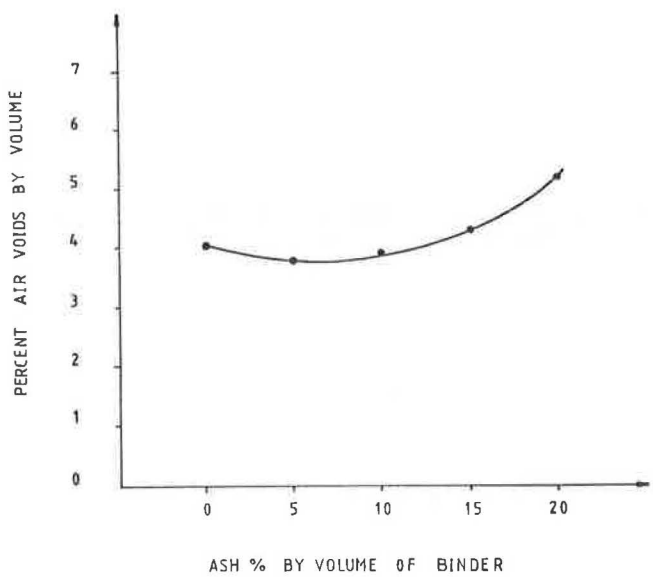

FIGURE 8 Effect of replacing asphalt with oil shale ash on air voids.

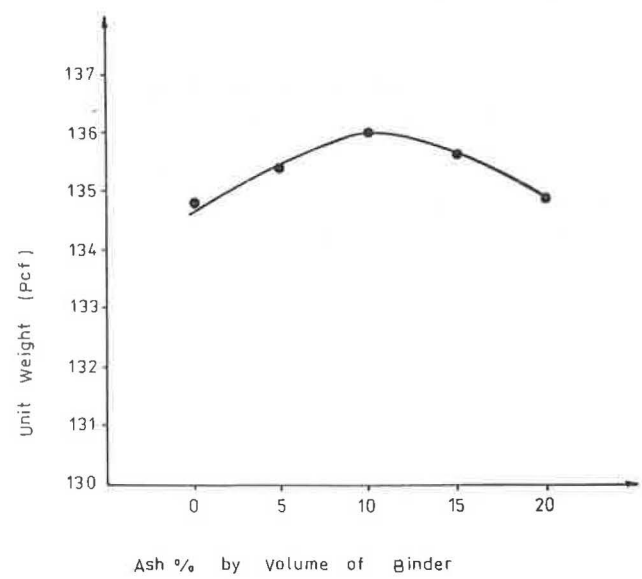

FIGURE 10 Effect of replacing asphalt with oil shale ash on unit weight.

In addition, the results of this phase, as shown in Figures 8 to 10, indicated that the air voids decreased with the addition of ash. The reduction in air voids was small at 5 and 10 percent ash, but increased substantially at 15 and 20 percent ash, as shown in Figure 8. The addition of oil shale ash reduced the flow as shown in Figure 9. The addition of ash, however, produced a substantial increase in the unit weight up to 10 percent ash and then decreased. Such a trend agrees with preliminary data obtained previously (*10*).

In phase 3, the indirect tensile test results are presented in Table 9 and shown in Figures 11 to 13. The results in Figure 11 indicate that the addition of ash resulted in a substantial increase in tensile strength up to 10 percent ash and then slightly decreased. The same trend is observed for the stiffness modulus. The minimum difference between results from dry and wet conditions is at 10 percent ash and the maximum, at 0 percent ash. Figure 13 indicates that the index of retained tensile strength is maximum at 10 percent ash (91 percent) and minimum at 0 percent ash (81 percent).

From the test results presented, it can be seen that the oil shale ash has significant effects on some mechanical properties of the bituminous concrete mixes, and the beneficial effects were associated with ash content of 10 percent. It seems that fine oil shale ash acts as an asphalt substitution; however, it also interacts with asphalt cement and stiffens the asphalt mix. While the large oil shale ash particles constitute part of the

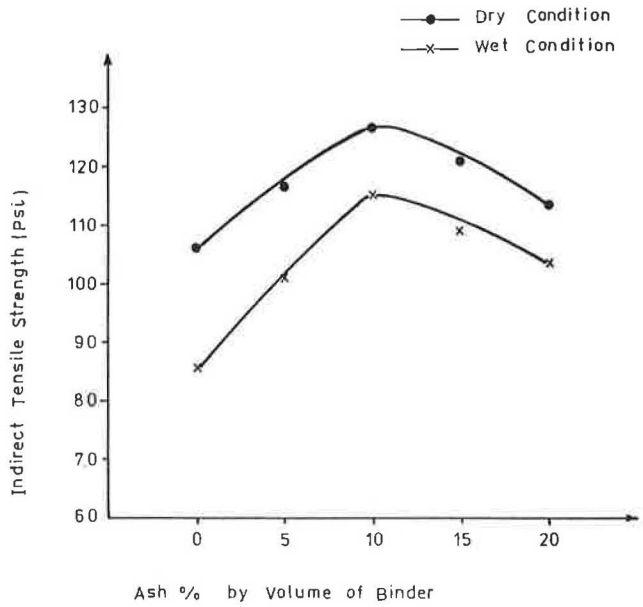

FIGURE 11 Effect of replacing asphalt with oil shale ash on indirect tensile strength in dry and freeze-thaw conditions.

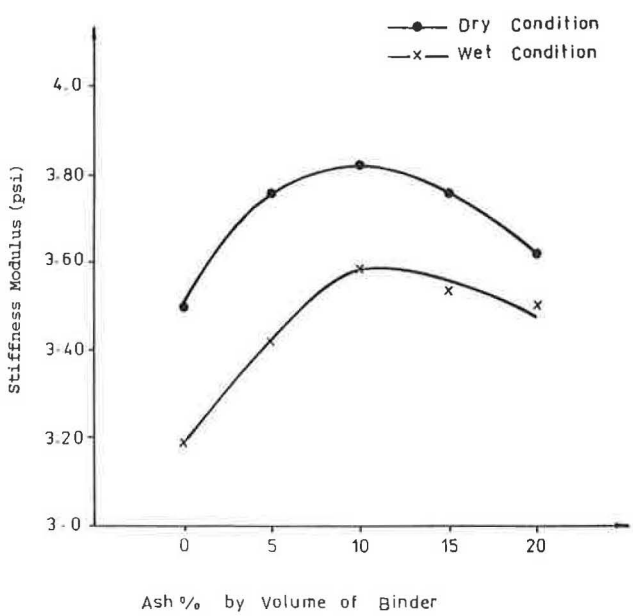

FIGURE 12 Effect of replacing asphalt with oil shale ash on stiffness modulus in dry and freeze-thaw conditions.

mineral aggregate, they also fill space and improve the interlocking bond between larger aggregate particles; this results in more resistance to freeze-thaw damage and a more stable mix.

The effects of freezing and thawing on the tested samples were determined by comparing the results of Marshall stability at 60°C, tensile strength at 25°C, and the stiffness modulus at 25°C with those obtained at wet conditions after 10 cycles of freezing and thawing. The results indicated a reduction of stability, tensile strength, and stiffness modulus due to freeze-thaw conditions compared with dry conditions at all ash levels. Figures 6 and 7 and Figures 11 to 13 indicate that the peak values for the three properties under both conditions were at

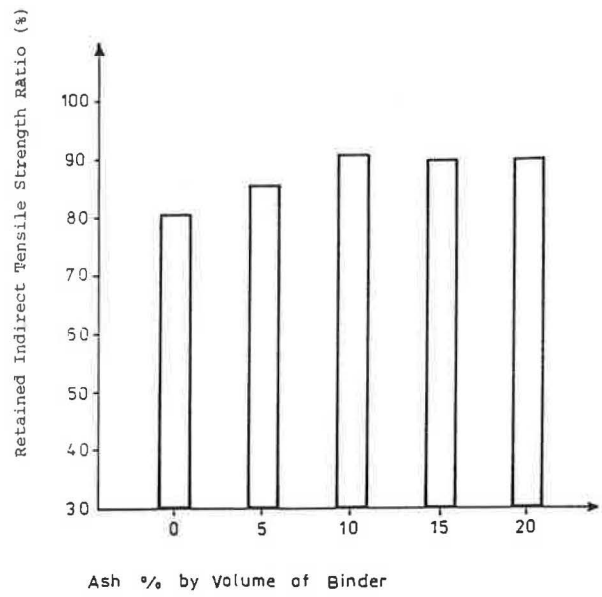

FIGURE 13 Relationship between oil shale ash and retained indirect tensile strength ratio.

ash level of 10 percent. However, the peak value at 10 percent ash in freeze-thaw conditions is still higher than that for the control sample with no ash in dry conditions. In addition to that, the minimum difference between the results of dry and freeze-thaw conditions is noted to be at 10 percent ash level, as shown in Figures 7 and 13.

Under both normal and wet conditions, the Marshall stability, tensile strength, and stiffness modulus were found to increase with an increase in ash content up to 10 percent and then to decrease. From Figure 8, it can be seen that after 10 percent the air voids have increased substantially, in addition to the considerable reduction in asphalt content that affects the cohesion resistance. The increase in strength from 0 percent ash up to 10 percent ash is due to the fact that the addition of ash fills space and provides more interlocking. Furthermore, Figure 11 indicates that the optimum ash content is 10 percent, which appears to provide a maximum tensile strength in both dry and freeze-thaw conditions.

In summary, the test results gave numerical values for Marshall stability, tensile strength, stiffness modulus, air voids, and flow at different oil shale ash contents of 0, 5, 10, 15, and 20 percent and also gave comparative values for dry and freeze-thaw conditions. According to test results, the addition of oil shale ash was found to improve both the resistance to freeze-thaw damage and the mechanical strength properties. The optimum ash content is 10 percent, which fulfills the most preferable design criteria of strength, durability, flexibility, and workability.

CONCLUSIONS

On the basis of the results of this study, the following conclusions are made:

1. The addition of oil shale ash improves the behavior of asphalt concrete mixes under dry and freeze-thaw conditions.

The optimum ash content was found to be 10 percent of the volume of the binder.

2. The stability of bituminous concrete was found to increase as the ash content increases, up to an ash level of 10 percent, and then to decrease, under dry as well as wet conditions after 10 cycles of freezing and thawing.

3. The index of retained Marshall stability was maximum at 10 percent ash and minimum at 0 percent ash.

4. Like stability, the tensile strength of bituminous concrete increases as the ash content increases up to 10 percent ash and then decreases under both dry and wet conditions.

5. The stiffness modulus of bituminous concrete mixtures increases as the percentage of ash in the mix increases up to 10 percent ash and then slightly decreases under both dry and wet conditions.

6. The ash was found to increase slightly the retained indirect tensile strength ratio up to 10 percent ash.

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