

The use of oil shale ash in Portland cement concrete

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Received 3 November 2000; accepted 11 July 2001

Abstract

An experimental investigation was undertaken to study the potential use of Jordanian oil shale ash (OSA) as a raw material or an additive to Portland cement mortar and concrete. Different series of mortar and concrete mixtures were prepared at different water to binder ratios, and different OSA replacements of cement and/or sand. The compressive strength of mortar and concrete specimens, cured in water at 23 °C, was determined over different curing periods which ranged from 3 to 90 days. The results of these tests were subjected to a statistical analysis. Equations were developed by regression analysis techniques to relate the effect of batch constituents on the strength developments of OSA mortars and concretes. The models were checked for accuracy by comparing their predictions with actual test results.

The obtained results indicated that OSA replacement of cement, sand or both by about 10% (by wt) would yield the optimum compressive strength, and that its replacement of cement by up to 30% would not reduce its compressive strength, significantly. It was found that OSA on its own possesses a limited cementitious value and that its contribution to mortar or concrete comes through its involvement in the pozzolanic reactions. The statistical model developed showed an excellent predictability of the compressive strength for mortar and concrete mixes.

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Keywords: Cement concrete; Oil shale ash; OSA

1. Introduction

The use of oil shale as a source of energy has been on the rise since more than two decades. It has been used in countries such as USA, resolved USSR states, Germany, and China, while its feasibility is being studied in other countries [1]. The use of oil shale is accompanied with a by-product known universally as oil shale ash (OSA). Since the latter causes environmental problems and because the cost of its disposal is very expensive, attempts were made to benefit from such material, especially in concrete and asphalt pavement construction [1–7].

The usage of the ash in concrete industry will not only reduce its production cost but also may enhance its fresh and hardened properties. Previous research concerned with this type of ash stated that its composition and properties can vary widely, ranging from high SiO₂ content, which is only pozzolanic in nature, to high CaO

content which has cementitious properties on its own [1]. In view of this, there was a need to evaluate OSA pastes mechanical behavior, chemical composition, and microstructure.

Several studies were concerned with the mechanical behavior and microstructure of an OSA cement paste [2–5]. The findings indicated that oil ash burnt at temperatures which ranged from 600 to 800 °C would have a cementing strength. It was reported that the strength development rate of OSA pastes was similar to that of ordinary Portland cement, although its strength was lower owing to the extra water content added to achieve proper workability due to the higher specific surface of the OSA. The ash was also used as an additive to asphalt concrete and proved to impart better mechanical and durability performance to the mixture [6,7].

In view of the fact that Jordan has a large reserve of oil shale rocks, interest has increased in using such rocks as a new source of energy. After carrying out comprehensive feasibility studies on the extraction of oil from oil shale rocks, the government has signed agreements with international companies to carry out the mining

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Table 1
Chemical compositions of Type I cement, OSA, and Al-wadi sand and limestone

Oxide	Percentage by wt			
	Type I cement	OSA	Al-wadi sand	Limestone
SiO ₂	21.21	35.4	80.3	0.98
CaO	63.69	39.7	5.31	55.46
Fe ₂ O ₃	3.11	2.0	1.33	0.31
Al ₂ O ₃	5.54	3.8	0.55	0.31
MgO	1.5	4.0	0.0	0.25
SO ₃	2.63	4.0	0.0	0
Na ₂ O	0.18	0.5	0.14	0
K ₂ O	0.71	0.6	0.07	0
IR ^a	0.12	—	—	—
LOI ^b	0.96	7.3	12.2	42.5

^a Insoluble residues.

^b Loss on ignition.

processes in the southern part of the kingdom. This project is expected to save the country millions of foreign exchanges, as part of imported crude oil would be partially substituted. Researches toward utilizing the expected huge quantities of the ash by-product have been also initiated and were directed mainly towards investigating its potential use in the concrete industry [8–10].

Smadi et al. [8] investigated the mechanical, physical, and chemical properties of OSA and tested various specimens of pastes of ash, cement and their mixtures. X-ray diffraction and thermogravimetric analysis indicated that the ash consisted mainly of calcite and silica and some calcium silicates, which possess cementitious characteristics. The study concluded that OSA showed a good potential for use as an admixture in cement and concrete.

In a study by Khedaywi et al. [9], the pozzolanic activity of Jordanian OSA was experimentally inves-

tigated. The results indicated that OSA has good pozzolanic activity, yet it was not as high as that of other commercial pozzolans. The authors referred this to the relatively lower specific surface of the OSA used as compared to that of other pozzolans.

Yeginobali et al. [10] studied the effectiveness of Jordanian OSA in reducing the alkali-silica reaction expansion. According to the results obtained with Pyrex glass aggregate, the ash only partially complied with the specified expansion requirements but still performed better than two locally available pozzolana admixtures. In addition, the ash was found to be helpful in reducing the expansion of mortars containing local chert aggregates.

It can be seen that the work reported on Jordanian OSA was limited mostly to paste behavior and properties. The present study, however, is directed towards investigating the short-term properties of OSA mortar and concrete mixes. In addition, a parametric study was carried out in order to develop empirical models for the prediction of strength development of both mixes. The experimental program included casting and testing a large number of mortar and concrete specimens under short-term compressive loading. Different water to binder (*w/b*) ratios with partial to full replacement of either cement and/or natural sand by OSA were used. The specimens were moist cured for different periods ranging from 3 to 90 days.

2. Experimental program

Series of mortar and concrete specimens incorporating Jordanian OSA were prepared and tested under short-term compressive strength loading. Physical and chemical properties of the materials used were also

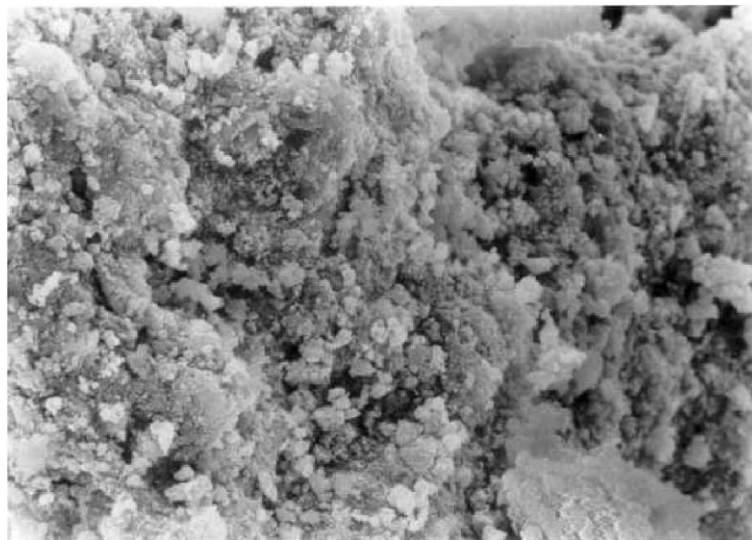


Fig. 1. SEM picture of Jordanian OSA.

Table 2
Mineral sand physical properties

	BSG (SSD)	ASG	BSG(D)	A (%)	FM
Al-wadi	2.43	2.58	2.34	4	3.5
Fine limestone	2.61	2.66	2.57	2.66	2.73
Coarse limestone	2.56	2.65	2.54	2.17	NA

BSG (SSD): Bulk specific gravity, saturated surface dry; ASG: Apparent specific gravity; BSG(D): Bulk specific gravity, fully dry; A: Absorption; FM: Fineness modulus; NA: Not applicable.

found. All series were tested in groups of three specimens at various age levels. In performing the tests, pertinent Jordanian, ASTM or British standards were followed.

2.1. Materials

Mortar and concrete mixes were prepared using Type I cement (blended with OSA) and natural sand and/or crushed limestone aggregate. Type I ordinary Portland cement conforming with Jordanian Standards JSS 219 was used. The chemical composition was obtained and is listed in Table 1. The Blaine fineness was found to be about 350 m²/kg.

The OSA provided by the Natural Resources Authority (NRA) of Jordan was obtained from El-Lajjun shale quarries, located in the south of Jordan, where there is a large reserve of oil shale rocks. The OSA was obtained after a retorting process in a rotary kiln and subsequent burning of the residual at a temperature of around 650 °C. Its Blaine fineness ranged from 500 to 600 m²/kg. The particles were angular with rough and porous surfaces, as shown in the scanning electronic microscope picture (Fig. 1). The specific gravity of the ash was found to be about 2.75. Its chemical analysis, as obtained using the X-ray fluorescence (XRD), is listed in Table 1.

The chemical composition of natural Al-wadi sand and crushed fine or coarse limestone is presented in Table 1. For fine particles, the gradation and fineness modulus

were determined according to ASTM C136, and the specific gravity and absorption were determined according to the ASTM C128 test method. The corresponding tests for crushed coarse limestone (with a maximum aggregate size of 12.5 mm) were determined according to ASTM C136 and ASTM C127, respectively. The physical properties of Al-wadi sand and crushed fine and coarse limestone aggregate are listed in Table 2.

2.2. Mix proportioning

Four series of mortar mixes were proportioned at different *w/b* ratios. Mortar mixtures of series I were prepared at a water to binder ratio of 0.7, and at replacements of Type I cement by OSA from 0% to 100% (by wt), as given in Table 3. Series II mortar specimens were proportioned at different weight replacements of sand by OSA, ranging from 0% to 30%, as given in Table 4. Specimens of series III were proportioned at different mixed replacement levels of both cement and sand by OSA, at contents ranging from 0% to 15% and from 0% to 10%, respectively, as presented in Table 5. Series IV specimens were proportioned at a *w/b* ratio of 0.60 with 0% and 10% cement weight replacements by OSA, and two types of fine particles; Al-wadi sand and crushed limestone. In series IV, and I the ratio of fine particles to binder was kept constant at 2.5 (by wt).

Concrete mixtures were proportioned according to the ACI mix design method at three *w/b* ratios of 0.45, 0.6, and 0.7 by weight. For each *w/b* ratio, various

Table 3
Proportions of series I mixtures prepared at a *w/b* ratio of 0.70 using different replacements of OSA

Mix No.	Percent replacement	Materials weights (g)			
		Water	Sand	Cement	OSA
1	0	70	250	100	0
2	10	70	250	90	10
3	20	70	250	80	20
4	30	70	250	70	30
5	40	70	250	60	40
6	50	70	250	50	50
7	60	70	250	40	60
8	70	70	250	30	70
9	80	70	250	20	80
10	90	70	250	10	90
11	100	70	250	0	100

Table 4

Compressive strength of ash/cement mortars with sand replacement (Series II)

Mix No.	Sand/binder	Water/binder	$S^a(\%)$	Compressive strength (MPa)			
				3 days	7 days	28 days	90 days
1	2.5	0.70	0	15.5	26.1	37.5	45.2
2	1.80	0.56	10	25.9	34.1	45.1	55.5
3	1.33	0.47	20	20.3	24.9	33.4	42.1
4	1.00	0.40	30	13.2	18.2	25.4	32.2

^a Sand replacement by OSA (%).

Table 5

Compressive strength of ash/cement mortars with sand and cement replacement (Series III)

Mix No.	Sand/binder	Water/binder	$C^a(\%)$	$S^b(\%)$	Compressive strength (MPa)			
					3 days	7 days	28 days	90 days
1	2.5	0.70	0	0	15.5	26.1	37.5	44.2
2	2.11	0.62	5	5	25.1	30.2	43.4	50.3
3	1.80	0.56	5	10	20.3	29.7	40.1	45.2
4	1.80	0.56	10	10	17.8	26.6	34.5	41.3
5	1.55	0.51	10	15	14.7	20.2	25.4	29.7

^a Cement replacement by OSA (%).^b Sand replacement by OSA (%).

mixtures were made at different replacements of cement by OSA of 0%, 10%, 20%, and 30% by weight. Al-wadi sand and coarse limestone were used in all concrete mixes.

2.3. Mixing, molding, and testing

Mortar mixtures were prepared by blending dry Al-wadi sand or crushed fine limestone with the cement, the OSA, and tap water in a mechanical mixer according to the ASTM C305 test method. Constant water content was maintained in all mortar mixes. It was noticed that as the OSA content increased, the consistency of mortar decreased. This is referred to the higher specific surface of OSA as compared to that of Type I cement. Cube mortar specimens ($2.5 \times 2.5 \times 2.5 \text{ cm}^3$) were cast by pouring mortar into two layers, and tamping each eight times.

Concrete mixes were prepared by blending coarse limestone aggregate, and fine Al-wadi sand with the cement, OSA and tap water in a tilting-type mixture of 0.04 m^3 volume according to the ASTM test method C 192. The workability of concrete showed a limited decrease as the OSA content increased. Therefore, concrete maintained an acceptable level of consistency that allowed easy molding of specimens. Cube specimens ($7 \times 7 \times 7 \text{ cm}^3$) were cast by placing concrete in two layers, and compacting each 25 times.

The surfaces of mortar and concrete specimens were finished by a trowel. The specimens were then left in a moist room for 24 h before being demolded and placed in water at 23°C . Hardened mortar and concrete spec-

imens were crushed to determine their compressive strength at ages ranging from 3 to 90 days.

3. Test results and discussion

The present study aimed at investigating the best ways of incorporating Jordanian OSA in concrete. The methodology followed involved the “replacement” of cement and/or sand in control mortar and concrete mixes with OSA at various levels by weight. A parametric study was also carried out, in order to find a mathematical relationship between the relative effects of the constituent materials on the strength development with time. The results of experimental and statistical studies on both mortar and concrete series are presented below.

3.1. Results of tests on mortar

The effect of cement replacement by OSA on the compressive strength development of mortar specimens of series I with time is shown in Fig. 2. The cement replacement varied from 0% to 100% by weight, while the water to binder ratio of 0.70 was maintained for all mixes. Compressive strength testing was performed at curing periods of 3, 7, 28, and 90 days.

The results indicate that the compressive strength increased initially as the OSA content is increased up to 10% replacement, and then decreased. The reduction in strength was moderate at 20% replacement and started to be significant at 30% replacement. At 30% replacement, the percentage reductions in strength for mortar

specimens cured in water for 7 and 90 days were 53 and 30, respectively. The compressive strengths of mortar specimens with 0%, 10%, 20%, and 30% cement replacement by OAS and cured for 28 days were 37, 40, 29, and 24 MPa, respectively. The 30% replacement of cement by OSA appears to be the limit for possible benefit of using OSA cement in structural and non-structural applications. On the other hand, using OSA at 10% replacement results in the optimum strength development for almost all mixes. The results showed that the effect of curing period on compressive strength development is minimal at high cement replacement contents by OSA.

The effect of OSA replacement of sand on the strength of OSA mortar cured for different periods is illustrated through results of series II specimens listed in Table 4. The data indicated that OSA replacement of sand by 10% yielded a higher strength than that of the control mix (without replacement). Moreover, it is clear that sand replacement by OSA up to 30% would yield strength in excess of 25 MPa which is the strength specified for most structural applications. Table 5 lists the strength values for OSA mortar specimens of series III. The result showed that replacement of cement and sand by OSA at contents of 5% each yielded the optimum compressive strength.

The compressive strength data presented indicated that OSA possesses a limited cementitious properties on its own and that its contribution to mortar compressive strength comes through its pozzolanic reactions with hydration products of cement [8,9]. It is important to indicate that the low strength growth gain and the relatively low reduction in OSA concrete or mortar strength at OSA replacement percentage up to 30%

would make its use recommended in structural applications where low heat concrete is needed.

3.2. Results of tests on concrete

The compressive strength development with curing age for concrete mixtures prepared at 0.45, 0.6, and 0.7 w/b ratios, with cement replacement by OSA ranging from 0% to 30%, is demonstrated in Figs. 3–5. The results show that both plain concrete and OSA concrete have similar strength growth trends: the strength generally increased up to 28 days of curing and then showed

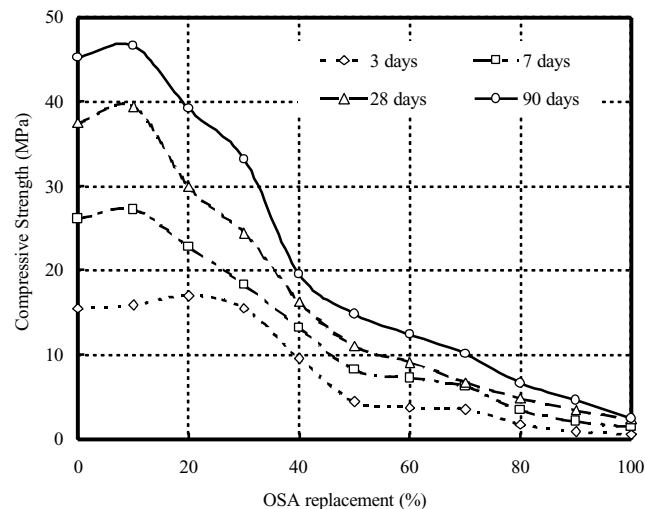


Fig. 2. Effect of OSA replacement % on the compressive strength for OSA mortar specimens cured for different periods.

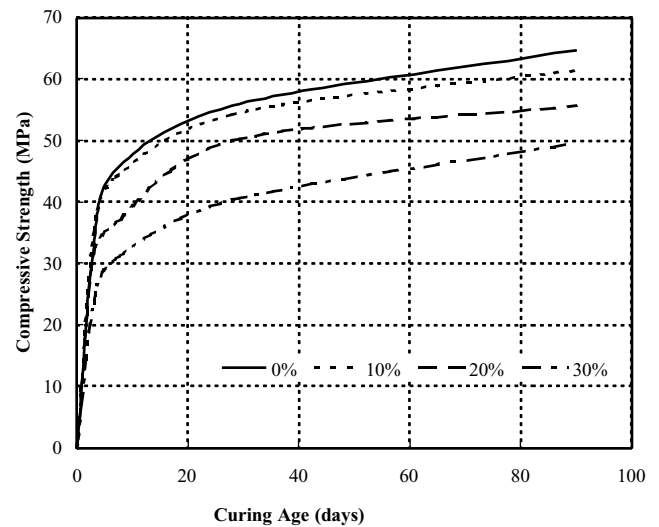


Fig. 3. Compressive strength versus curing age for concrete prepared at a w/b ratio of 0.45 using different replacements of OSA.

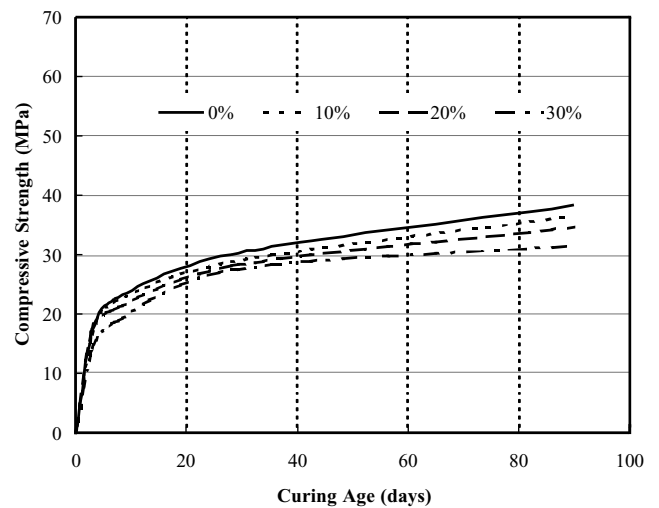


Fig. 4. Compressive strength versus curing age for concrete prepared at a w/b ratio of 0.6 using different replacements of OSA.

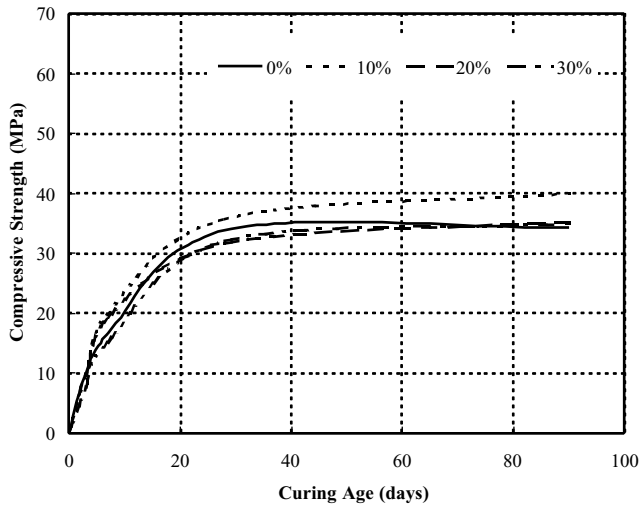


Fig. 5. Compressive strength versus curing age for concrete prepared at a w/b ratio of 0.7 using different replacements of OSA.

a slow gain rate. The optimum strength for the mixture prepared at 0.45 and 0.6 w/b ratio was achieved at 10% OSA replacement while that for the mix prepared at a w/b ratio of 0.7 was achieved at 0% OSA replacement (plain concrete). Mixtures prepared at 20% and 30% OSA replacement showed generally a lower early yet higher later strength compared to those prepared at 0% OSA replacement. The data indicate that the percentage reduction in compressive strength upon replacement of cement by OSA at contents of 20% and 30% decreased at higher water to binder ratios of 0.6 and 0.7. This suggest that the use of OSA in ordinary concrete (strength range 20–25 MPa) at contents up to 30 % would cause limited loss in strength.

4. Parametric study

4.1. OSA mortar

To develop a statistical model for predicting the compressive strength of OSA mortar, the data collected from compressive strength measurements of OSA mortar of series I–III were used. This model shall be applicable for a wide range of sand to binder ratios from 1 to 2.5, water to binder ratio ranges from 0.40 to 0.70, and for OSA weight replacement contents of 0–100%. A non-linear model was obtained using the statistical package SPSS. The formulation of obtained empirical model was found to be as follows:

$$F_{cm} = K_{cm} \left(t, \frac{a}{b} \right) * \left(\frac{w}{b} \right)^{-0.87}, \quad (1)$$

where F_{cm} : compressive strength for OSA mortar (MPa); $K_{cm}(t, a/b) = 12.07t^{0.24} \text{Exp}(-4.25(a/b))^{2.17}$: mortar model

coefficient; t : curing period (days); a/b : OSA to binder (OSA + cement) ratio by weight; w/b : water to binder ratio by weight.

The multiple coefficient of determination (R^2) was found to be about 0.90. The standard errors for the model powers or coefficients, 12.07, 0.24, -4.21 , 2.17, and -0.87 , are 1.07, 0.02, 0.79, 0.29, and 0.17, respectively. These statistical parameters indicate that the developed model fits well the data used.

To further examine how well the obtained model fits the data used, the residual plot for the model was drawn (Fig. 6). As can be seen, there are few large residuals (and hence limited apparent outliers) and that there is no trend to indicate that the non-linear model is inappropriate. Fig. 7, which presents predicted versus measured compressive strengths for OSA mortars, indicates a similar conclusion: the inclination of the fitted straight line is around 45° , reflecting an excellent fit between the actual and predicted strengths.

To evaluate the predictability of the developed model, measured compressive strength data of series IV (not used in developing the model) were compared with those predicted by the model, Figs. 8 and 9. It can be clearly concluded that the model provides an excellent prediction, especially at later ages (high strength values) and that its predictability would not be significantly affected by the type of fine particles used, whether it is Al-wadi sand or crushed limestone.

4.2. OSA concrete

The ability of the model developed for an OSA mortar to predict the compressive strength of OSA

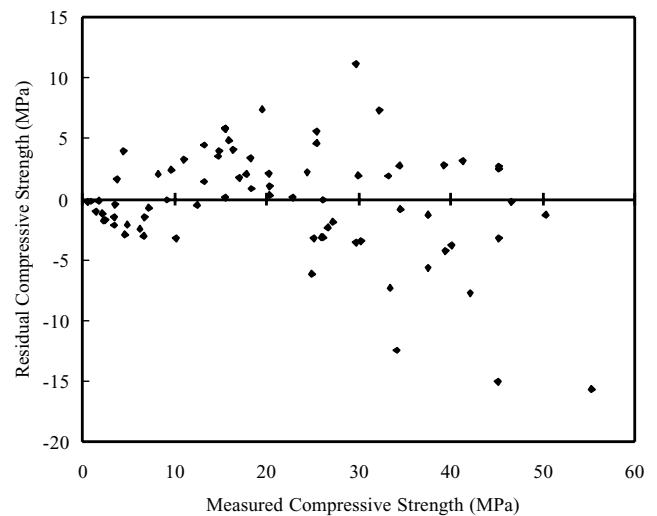


Fig. 6. Residual compressive strength versus measured compressive strength for different OSA mortar mixes.

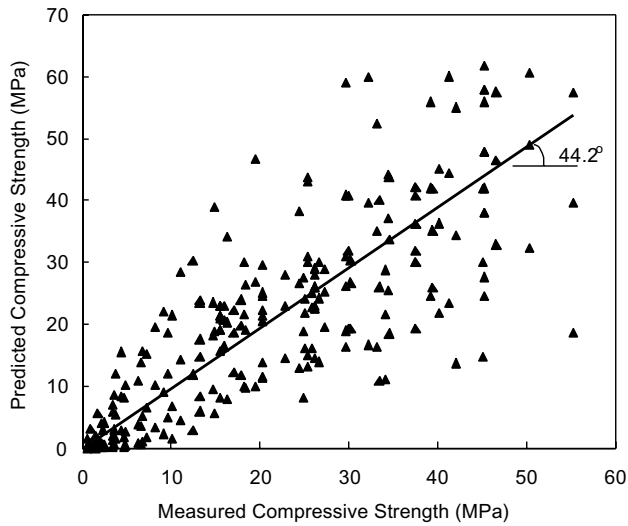


Fig. 7. Predicted versus experimental compressive strength for different OSA mortar mixes.

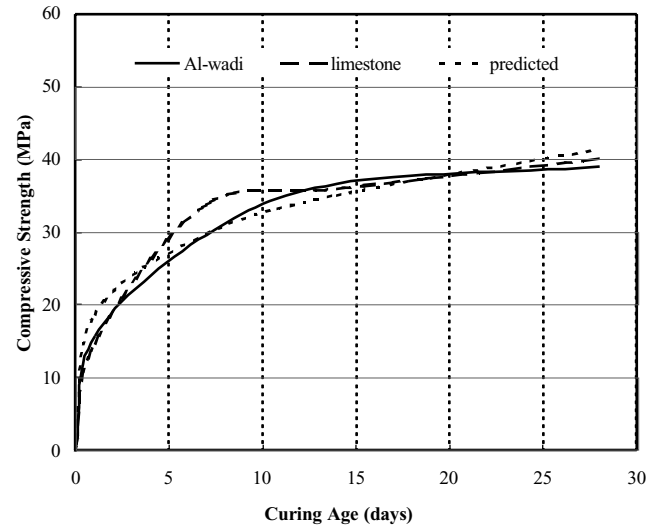


Fig. 9. Compressive strength versus curing age for mortar prepared at 10% OSA replacements.

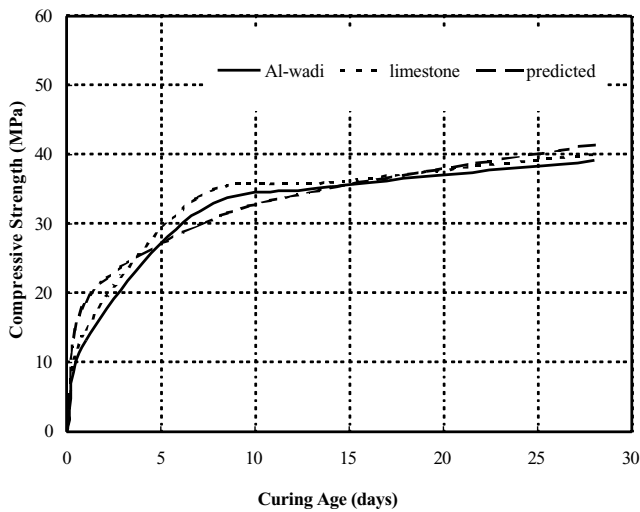


Fig. 8. Compressive strength versus curing age for plain mortar mixes.

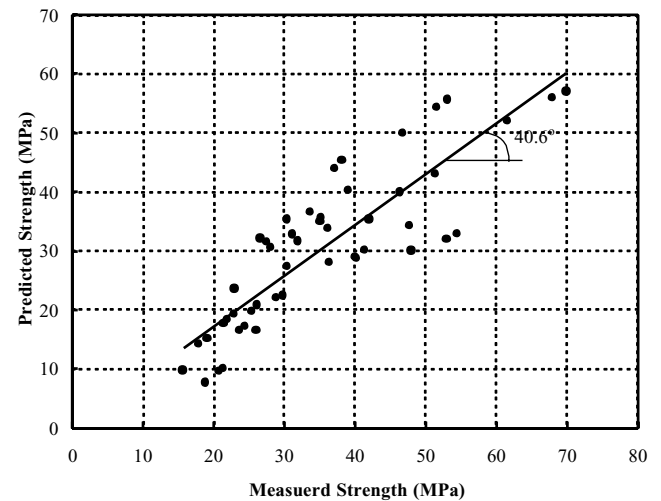


Fig. 10. Predicted versus experimental compressive strength for different OSA concrete mixes (using mortar model).

concrete was examined through Fig. 10, which shows predicted versus measured compressive strength. As can be seen, the slope of the fitted straight line deviates significantly from 45° . Therefore, it would be concluded that the developed model although predicts OSA mortars strength well provides a poor prediction of OSA concrete's strength. Hence, another statistical model was developed for the OSA concrete based on the compressive strength data presented in Figs. 3–5. Using the SPSS statistical package, a similar model form of that obtained for mortar was developed and is given as

$$F_{cc} = K_{cc} \left(t, \frac{a}{b} \right) * \left(\frac{w}{b} \right)^{-1.48}, \quad (2)$$

where $K_{cc}(t, a/b) = 8.14t^{0.21}\text{Exp}(-0.74(a/b))$: concrete model coefficient; F_{cc} : compressive strength of OSA concrete (MPa).

Definitions of other parameters are as given in Eq. (1).

The multiple coefficient of determination (R^2) was found to be about 0.89. The standard errors for the model powers or coefficients, 8.14, -0.74 , 0.21, and -1.48 , are 0.82, 0.18, 0.02, and 0.16, respectively. These values indicate that the model fits well the processed compressive strength data.

To further examine how well the developed model fits the used data, Fig. 11 that shows predicted versus measured compressive strengths was prepared. The slope of

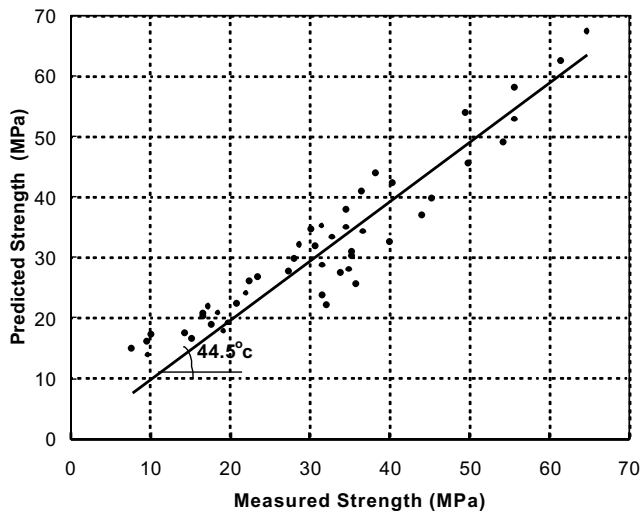


Fig. 11. Predicted versus experimental compressive strength for different OSA concrete mixes (using concrete model).

the fitted straight line was found to be about 44.5° , which is very close to 45° ; perfect fitting. Therefore, the present model is expected to provide a very good level of compressive strength prediction of OSA concrete.

It is important to mention that mix design tables can be developed based on Eqs. (1) and (2) to proportion OSA mortar and concrete mixtures, respectively. The data presented in such tables enable the civil engineer to choose the appropriate water to binder ratio, as well as, the OSA replacement content based on the required early and later design strengths.

5. Conclusions

Based on the results of the present investigation, the following conclusions can be made:

1. OSA can be used as an additive in Portland cement concrete to economically produce structural and non-structural concrete with the same range of design strengths as conventional concrete.
2. The use of OSA as a partial replacement of cement or sand in concrete or mortar would contribute to its compressive strength, especially at an optimum replacement level of 10% by weight.

3. OSA may be used to replace cement in mortar or in concrete by up to 30% by weight, without detrimentally affecting the latter's compressive strength.
4. The beneficial use of OSA in concrete construction can contribute significantly to solving the otherwise hazardous environmental problem associated with utilizing oil shale rocks in energy production.
5. OSA, when used as an additive in Portland cement concrete, can have a retarding effect on early strength gain; therefore when early strength as well as final strength is a factor in design, a proper mix incorporating OSA may be developed to accommodate this effect.
6. The statistical models developed in this work provide an excellent prediction of the compressive strength of OSA mortars and concretes.

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