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Oil Shale Ash Addition Effect in Concrete to Freezing-Thawing

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Additional information is available at the end of the chapter

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Abstract

Modifying the properties of cement by including mineral-based additives into cement ensures benefits such as preventing environmental hazards of waste additives and providing increase in cement amount. Based on these main considerations, as a part of the study in which contribution of ash, obtained as a result of the burning of oil shale rocks in fluidized bed thermal plants, to Portland cement (PC) in 15% ratio is chemically proven, this study is performed in order to understand the direction and severity of the effects that oil shale ash (OSA) might have on the properties of cements. For this purpose, freezing-thawing experiments were performed on cement samples produced with PC 42.5 cement, which has 0, 10, 20, and 30% of OSA additions. It is desired to make a conclusion by finding weight loss factor (AF_w) and relative elasticity module (E) loss related with press and ultrasonic test methods based on given experiments. As a result, it was observed that durability of concretes obtained by addition of oil shale rocks in 15% ratio to PC 42.5 cements against freezing/thawing effects is greater than that of PC 42.5 control sample.

Keywords: Portland cement, oil shale ash, concretes, freezing-thawing, resistance

1. Introduction

Concrete should not be damaged by decomposing due to the compressive strength of the ice formed in it. If compression stress in ice that is the resultant of freezing, reaches greater values than original compressive strength of ice, the ice turns to water by decomposing and the negative effect of freezing are removed [1]. Depending on the freezing temperature, there can be increases in the volume of water frozen inside the concrete by up to 12%, and this causes wearing in concrete by creating internal stresses.

Frost occurrence initially starts at big pores in the concrete and then builds up at small pores. The freezing degree of water decreases as the size of gaps inside the material is reduced. Finally the water inside capillary gel gaps gets frozen. This happens as salt crystals cannot be formed in gel gaps that are in microns size, and the freezing of water in these zones cannot be prevented. Temperature value should be under -78°C for water to be frozen inside the mentioned small capillary pores. Here, the major effects are the methods and techniques and materials used during the concrete manufacture and also the fact of adding or not adding additive substances inside cement qualification and the type and amount of the additive substance, if added.

Oil shale is formed by the simultaneous sedimentation of granule mineral fragments and the rotting organisms of low-ranked animals and plants and also is a kind of marlite containing combustible organisms. Several researchers studied about the addition of oil shale ash into cement. Xiang-penga et al. studied the effects of burning temperature on the reactivity of oil shale ash [2]. Al-Hasan [3] searched to replace cement with ash, even with small amounts, and he found it to be an effective way to improve thermal conductivity of concrete mixtures. Further, the higher was the level of cement replacement by oil shale ash, the lower was the compressive strength [3]. Oymael [4] obtained at 700°C by burning oil shale and then added to cement at the ratio of 15 and 30% by its weight. The optimum pozzolanic characteristics and performances were provided by the mixture containing 15% ashes [4]. Smadi and Haddad [5] observed that oil shale ash replacement of cement, sand, or both by about 10% (by weight) yielded the optimum compressive strength. Moreover, its replacement of cement by up to 30% was not reducing its compressive strength [5]. Raado et al. [6] focused on the use of oil shale ash for low-strength concrete. Two main types of oil shale ash and their mixes were tested, and they discovered that expansion and water resistance tests showed the content of CFB ash in OSA binders to increase and water resistance was developed and expansion disappeared [6].

The purpose of this study is to reveal the attribute of concretes made up with pozzolanic oil shale ash added Portland cements (PC) against freezing-thawing effects. Based on these considerations, this study investigates the relationship between compressive strength values and material loss that would happen due to frost under freezing-thawing forcing in air-entrained and non-air-entrained (10 cm^3) concrete samples that are made with PC 42.5 cements as per TSE CEN/TS12390-9 [7].

2. Materials and method

2.1. Cement

PC 42.5 cement manufactured in Gaziantep Cement Factory and brought as bulk to Elazığ El-Beton Prefabricated Concrete Building Elements Factory was used. A chemical and physical property of the mentioned cement is given in **Table 1**.

2.2. Oil shale ash

Oil shale rocks of Ankara-Çayırhan region are broken to aggregate size and grinded to fineness of cement. By firing this powdered material in laboratory-type ovens, at the rate of 125

Chemical properties (%)		Physical properties		
CaO	63.92	Specific gravity (g/cm ³)		3.11
SiO ₂	19.57	Blaine fineness (cm ² /g)		3510
Al ₂ O ₃	5.72	Compressive strength (N/mm ²)	7 days	41.9 (lim: 31.5) N/mm ²
Fe ₂ O ₃	3.69			
MgO	1.17			
SO ₃	3.19		28 days	54 N/mm ²
Unidentified	0.24			
(Na ₂ O + K ₂ O)	0.15 + 0.62			
Chloride	≤0.1			
Loss on ignition	1.73			
C ₃ S	58.68			
C ₂ S	11.83			
C ₃ A	8.91			
C ₄ AF	11.23			

Table 1. Properties of Portland cement.

capsules for 1.5 h at 500, 600, 700, 800, 900, and 1030°C, oil shale ash is obtained. Obtaining oil shale ash as waste substance in industry can be made by firing/combusting oil shale rocks as combustion of low calorie lignite in fluidized bed thermal plants. If the most suitable values of ash were obtained by firing at 700°C [8] in the study, this temperature at fluidized bed thermal plants would be needed to determine and control the rate of material to be transferred into the oven and air circulation (**Table 2**).

The values obtained in activity experiments performed according to TS25/T1 [9] and TS EN 197-1 [10] are given in **Table 3**. In the activity experiment performed by lime, the amount of ash is found by the formula of (specific weight of ash/specific weight of lime) × 300 and that

Firing temperature (°C)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	CaO (%)	MgO (%)	SO ₃ (%)	Loss on ignition (%)	Insoluble residue (%)	Unidentified (%)
500	29.26	10.15	4.65	36.13	6.82	2.30	3.24	4.21	3.24
600	28.6	8.97	4.47	32.80	8.04	3.79	6.47	3.88	2.98
700	39.32	7.80	4.20	26.40	9.26	5.21	3.82	1.28	1.11
800	28.4	9.40	4.40	31.18	7.70	2.27	8.82	4.30	3.53
900	20.25	13.00	4.35	29.00	6.63	4.25	15.4	5.37	1.87
1030	14.54	17.45	4.90	26.82	5.35	6.23	19.84	3.94	0.93

Table 2. Chemical change of oil shale ash depending on firing temperature.

Firing temperature (°C)	Lime activity (TS25/T1) [9]		Cement activity (TS EN 197-1) [10]					
	Compressive strength (N/mm ²)	Flexural strength (N/mm ²)	Compressive strength (N/mm ²)			Flexural strength (N/mm ²)		
	7 days		2 days	7 days	28 days	2 days	7 days	28 days
500	—	—	—	—	—	—	—	—
600	9.1	3.0	—	—	—	—	—	—
700	13.0	3.6	15.3	23.9	29.5	3	4.5	8.1
800	10.1	3.0	11.1	22.2	23.3	2.7	4.3	5.8
900	8.7	2.6	—	—	—	—	—	—
1030	4.2	1.3	—	—	—	—	—	—

Table 3. Activity experiments values and results of OSAs.

amount of ash is mixed with 150 g of lime, 239 g of water, and 1350 g of standard sand and standard mortar is obtained. However, in cement experiment, mortar is obtained by mixing the ash, for which the amount is firstly found by the formula of $(147 \times \text{specific weight of ash}) / \text{specific weight of cement}$, with 293 g of Portland cement, 1350 g of standard sand and water in an amount of 0.5 (Portland cement + ash). For experiment of activity with lime, after the mortar mixtures were given shape in $40 \times 40 \times 160$ mm of standard molds and kept under room temperature for 24 h, the open surfaces of the molds were covered with glass and their surroundings were made airtight with paraffin, and they were kept in an oven for 6 days at $55 \pm 2^\circ\text{C}$. Those mortar samples were cured in water for 2, 7, and 28 days for experiment of activity with cement. Activity experiment values and results are given in **Table 3**.

The mean numeric relationship between addition ratios of OSA at the temperatures is obtained, and its fineness is given in **Table 4**. The ash at 700°C , which was sieved through mentioned sieves, was seen to give the optimum fineness values. Blaine fineness for ashes obtained as a result of firing was found to be $3180\text{--}3500$ cm²/g without performing any additional grinding process.

Firing temperature (°C)	200 µm Sieve (%)	90 µm Sieve (%)	Blaine fineness (cm ² /g) (average)
PC-Control	0.5	6.7	3180–3500
500	0.7	5.9	
600	0.9	4.0	
700	0.6	4.0	
800	1.0	9.0	
900	1.8	9.8	
1030	1.1	4.7	

Table 4. Relationship between firing temperature and fineness.

Particle group (mm)	Volume spec, weight (DYK)	Water absorption (%)	Present humidity (%)	Density (kg/m ³)		Abrasion (%) (Tuvennan)	
				Loose	Tight	100 cycles	500 cycles
0–8	2.69 (2.59)	1.15	0.7	1650	1755	6.98	18.63
8–16	2.69	1.2	0.62	1580	1720		
16–19	2.69	1.2	0.44	1455	1650		

DYK, saturated dried surface.

Table 5. Granulometry of Tuvennan aggregate.

The specific weight of oil shale ash had shown a relative increase from 2.26 to 2.77 g/cm³ based on the firing temperatures at 500, 600, 700, 800, 900, and 1030°C. As this is lower than the specific weight of Portland cement, this causes a partial increase in the volume of the cement.

2.3. Standard sand

All of the mortar mixtures in the study were used in the standard sand produced by the Pınarhisar Cement Plant which is in compliance with TS EN 196-21 [11].

2.4. Aggregate

In cement experiments, washed Palu aggregate used in Fırat University Elazığ Vocational School Laboratories was used. The maximum aggregate size was selected by using one-fifth of the experiment sample molds (19 mm) as a basis. Three sizes (0–8, 8–16, 16–19 mm) of aggregates were used. Properties of granulometric structured aggregate in accordance with TS 706 EN 12620 + A1 [12] are given in **Table 5**.

3. Experiment method and results

In the study, PC 42.5 Portland cement and aggregate were used. In the design of concrete mixture, water amounts were specified based on DYK (saturated dried surface) aggregate (**Table 7**). It was taken as a basis to have 105–110% dispersion in mixtures and to add liquid air-entraining admixture chemical substance specified in TS EN 934-4 [13] to water amount in air-entrained samples. In the experiment, OSA in 0, 10, 20, and 30% ratios were added to PC 42.5 cement, and in order to meet the predicted dispersion values, corrections in water amounts were made. It was observed that in concrete mixtures as OSA, percentage ratio is increased, the water requirement is also increased, and dispersion value is decreased. This fact reflects on the water amount in concrete mixture design and accordingly on the fresh concrete density. Non-air-entrained concrete mixture design is given in **Table 6**, and air-entrained concrete mixture design is given in **Table 7**.

For the study, cubic samples of 10 cm size were prepared in two groups. The first group includes air-entrained concrete samples, whereas the second group includes non-air-entrained (normal) concrete samples. Each group of samples was exposed to three experiments. First of

Mixture	0% PC-Control	10%	20%	30%
Cement (kg)	360	324	288	252
OSA (kg)	—	36	72	108
Fine aggregate (0–8 mm)	663	663	663	663
Coarse aggregate (8–16 mm)	807	807	807	807
Coarse aggregate (16–19 mm)	346	346	346	346
*Water + HK** (kg)	202 + 1.2	209 + 1.2	216 + 1.3	245 + 1.5
Water/binder (%)	0.52	0.54	0.6	0.68
Dispersion (mm)	115	116	114	116
Fresh concrete density (kg/m ³)	2378	2385	2392	2421

*Net water (aggregate DYK).

**Air is predicted, included to the fresh concrete weight.

Table 6. Non-air-entrained concrete mixture design.

them was the experiment on determination of weight loss factor (AF_w), the second one was the experiment on press compressive strength loss factor, and the third one was the experiment on determination of dynamic elasticity module.

In the experiment on determination of weight loss factor (AF_w) in freezing/thawing experiment, following 28 days of cure period in water at average (+22°C) temperature, the samples for compressive strength were broken by press. Other samples were exposed to freezing-thawing experiments at Elazığ El-Beton Concrete Prefabricated Building Elements Factory Laboratory.

Experiment samples were dried at $110 \pm 5^\circ\text{C}$ in an oven until they reached constant weight, then they were taken from the oven and cooled in a desiccator until they reached room temperature. Then, they were weighed with 0.1 g precision, and W_0 is found. Those experiment

Mixture	0% PC-Control	10%	20%	30%
Cement (kg)	360	324	288	252
OSA (kg)	—	36	72	108
Fine aggregate (0–8 mm)	663	663	663	663
Coarse aggregate (8–16 mm)	807	807	807	807
Coarse aggregate (16–19 mm)	346	346	346	346
*Water + HK** (kg)	207 + 1.8	214 + 1.8	236 + 1.8	243 + 1.8
Water/binder (%)	0.58	0.6	0.66	0.68
Dispersion (mm)	105	111	115	116
Fresh concrete density (kg/m ³)	2384	2391	2413	2441

*Net water (aggregate DYK).

**HK: air entraining ratio 0.5%.

Table 7. Air-entrained concrete mixture design.

samples were saturated with water under normal atmospheric conditions and were frozen in the freezer. The cooling rate of the freezer was adjusted as it would decrease to -20°C within 4 h. After it was observed that the temperature of the freezer decreased to -20°C , the experiment samples that were kept at this temperature for about 2 h were taken out at the end of the period. By immersing them in water as they would be completely under water and keeping them as such for 2 h, it was ensured that the ice completely melted. At the end of the freezing-thawing processes repeated for 25 times like this, after the experiment samples that reached constant weight in the oven at $110 \pm 5^{\circ}\text{C}$ were cooled in a desiccator, they were weighed with 0.1 g precision (W_n). As can be seen in **Tables 8** and **9**, the decrease in mass, weight loss factor (AF_w), caused by the portions that break and leave from the samples due to freezing-thawing effect is calculated by the following formula (Eq. (1)).

$$AF_w = 1 - W_n/W_0 \times 100 \quad (1)$$

Here AF_w : weight loss factor (%); W_0 : mean compressive strength of samples before frost experiment (N/mm^2); W_n : arithmetic mean of compressive strength of concrete after freezing (N/mm^2).

According to weight loss and compressive strength											
Mixture (air addition (HK))	Number of samples	According to weight loss			According to compressive strength						
		Mean initial weight (g) (W_0)	Mean final weight (g) (W_n)	$AF_w = 1 - W_n/W_0 \times 100(\%)$	Initial compressive strength (N/mm ²) (f_0)	Final compressive strength (N/mm ²) (f_n)	$DF_f = 1 - f_n/f_0 \times 100 (\%)$				
0% HK, PC-Control	6	2.32	2.303	0.7	20.1	16	20.3				
10% + HK	6	2.28	2.254	1	13.5	10.1	25.1				
20% + HK	6	2.27	2.238	1.4	12.7	9.5	23.6				
30% + HK	6	2.18	2.139	2	11.3	8.3	26.5				
According to relative dynamic elasticity module											
Mixture (air addition (HK))	Number of samples	Unit weight (kg/dm ³)	Number of freezing–thawing cycles and ultrasonic measurements						$E = 10^6 \times v^2 \times \Delta/g$	$E = 10^6 \times v^2 \times \Delta/g$	$E = 10^6 \times v^2 \times \Delta/g$
			V_0 (m/s)			V_n (m/s)			V_n (m/s)		
			0	5	10	15	20	25	E_0 (N/mm ²)	E_n (N/mm ²)	EMF (%)
0% HK, PC-Control	6	2.32	4.44	4.16	4.14	4.11	4.08	3.9	466,213	35,976	23
10% + HK	6	2.28	4.30	4.06	4.06	4.0	3.86	3.61	429,737	302,886	29
20% + HK	6	2.27	4.20	3.93	3.92	3.79	3.63	3.45	408,183	275,419	33
30% + HK	6	2.18	4.00	3.87	3.86	3.86	3.80	3.15	355,555	225,000	38

Table 8. Calculation table of freezing-thawing experiment for air-entrained samples.

According to weight loss and compressive strength											
Mixture	Number of samples	According to weight loss			According to compressive strength						
		Mean initial weight (g) (W_0)	Mean final weight (g) (W_n)	$AF_w = 1 - W_n/W_0 \times 100(\%)$	Initial compressive strength (N/mm ²) (f_0)	Final compressive strength (N/mm ²) (f_n)	$DF_f = 1 - f_n/f_0 \times 100 (\%)$				
0% PC-Control	6	2.408	2.383	1	22.5	16.3	27.5				
10%	6	2.443	2.411	1.3	20.6	14.3	30.5				
20%	6	2.369	2.319	2.1	15.6	9.1	41.6				
30%	6	2.293	2.233	2.6	11	6.1	39				
According to relative dynamic elasticity module											
Mixture	Number of samples	Unit weight (kg/dm ³)	Number of freezing–thawing cycles and ultrasonic measurements						$E = 10^6 \times v^2 \times \Delta/g$	$E = 10^6 \times v^2 \times \Delta/g$	$E = 10^6 \times v^2 \times \Delta/g$
			V_0 (m/s)		V_n (m/s)				E_0 (N/mm ²)	E_n (N/mm ²)	EMF (%)
			0	5	10	15	20	25			
0% PC-Control	6	2.408	4.58	4.32	4.32	4.25	4.18	4.13	514,894	418,685	19
10%	6	2.443	4.46	4.27	4.25	4.06	3.86	3.83	495,363	365,301	26
20%	6	2.369	4.40	4.23	4.11	4.04	3.47	3.38	467,521	275,885	41
30%	6	2.293	4.31	4.03	4.00	3.93	3.27	3.11	434,199	226,076	48

Table 9. Calculation table of freezing-thawing experiment for non-air-entrained samples.

In the second part of the experiment, a study taking losses at compressive strength as a basis was performed. Decrease factor of compressive strength (DF_f) is calculated by the following formula by finding mean compressive strength of samples after experiment (f_n) for which mean compressive strength values at press (f_0) for equivalents of original samples of experiment were found before freezing-thawing experiment (Eq. (2)).

$$DF_f = 1 - f_n/f_0 \times 100$$

(2)

Here DF_f = decrease factor of compressive strength (%); f_0 = mean compressive strength of sample before experiment (N/mm²); f_n = mean compressive strength of sample after freezing (N/mm²).

In the third part of the experiment, ultrasonic pulse velocity ($v = \text{km/s}$) of samples exposed to freezing-thawing were found before experiment (DYK) and during experiment at the end of 5, 10, 15, 20, and 25 cycles. Based on these values, the dynamic elasticity modules (E) of samples are calculated by the following formula (Eq. (3)).

$$E = 106 \times v^2 \times \Delta / g. \quad (3)$$

Here E = relative dynamic elasticity module (N/mm^2); v = ultrasonic pulse velocity (km/s) (V_0 = values before experiment; V_n = values after experiment); Δ = unit weight of concrete (kg/dm^3); g = gravitational acceleration (g/cm^2).

In experiments conducted as per TSE CEN/TS 12390-9 [7] principles, comparative values of after frost losses of weight and compressive strength and relative dynamic elasticity module of air-entrained and non-air-entrained samples which were exposed to freezing-thawing in water are shown in **Figures 1–3** and **Tables 8** and **9**.

When AF_w values after 25 cycles of freezing–thawing in air-entrained concrete samples are examined, it is observed that for those with 10% addition, partial frost loss is 0.30 times less than the control sample and for those with 20 and 30% addition, partial frost loss is 2.42 and 2.85 times less than the control sample, respectively. In general, those values are 27% less than those with no air entrainment.

For air-entrained and non-air-entrained samples, decrease factor of compressive strength (DF_f) as given in **Tables 8** and **9** (if pressure loss value at 20% OSA ratio is excepted), it is seen that there is a linear relation between pressure loss value and addition ratio, and as the addition ratio increases, the pressure loss also increases. Both in added or not added samples, considering that the pressure loss due to freezing-thawing experiments (DF_f) cannot be more than 20% [14], it is understood that 15% of the addition ratio would be more suitable.

In evaluations performed according to relative elasticity module (E) in air-entrained samples, the value for 0% added (control) sample was 23%, whereas it was 29, 33, and 38% for those with 0, 20, and 30% addition, respectively. After 25 cycles, if maximum 0.30 [14] limit is considered for decrease in relative dynamic elasticity module of concrete samples, it can be suggested that 15% added one would be suitable. This result is also valid for non-air-entrained samples. Relative dynamic elasticity module values are given in **Figure 3**.

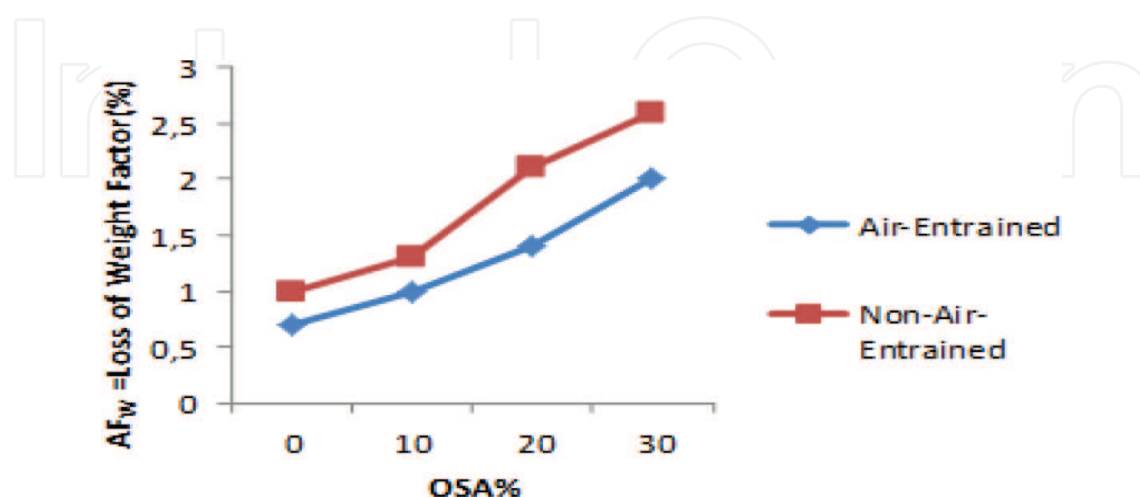


Figure 1. Relationship between weight loss and OSA percentage in samples.

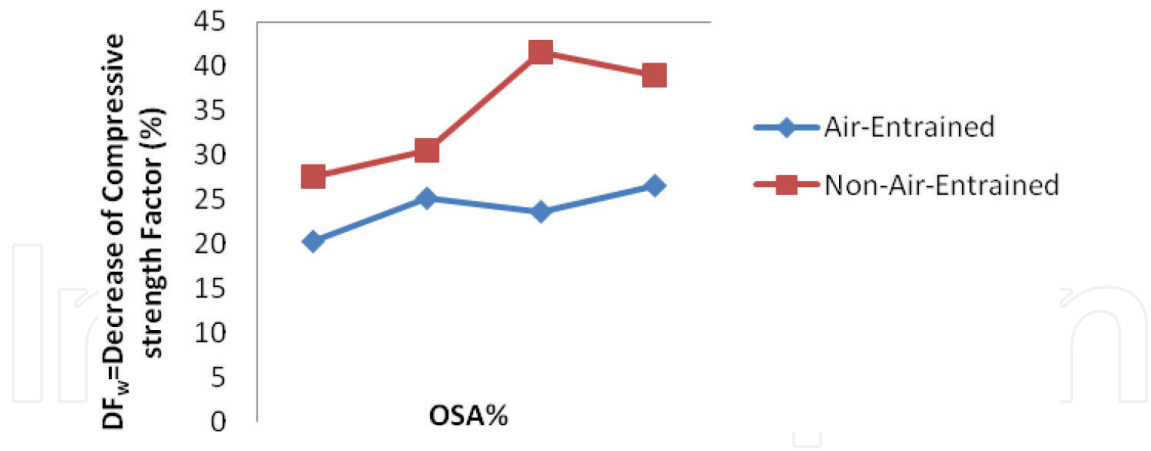


Figure 2. Relationship between compressive strength factor and addition percentage in air-entrained and non-air-entrained samples.

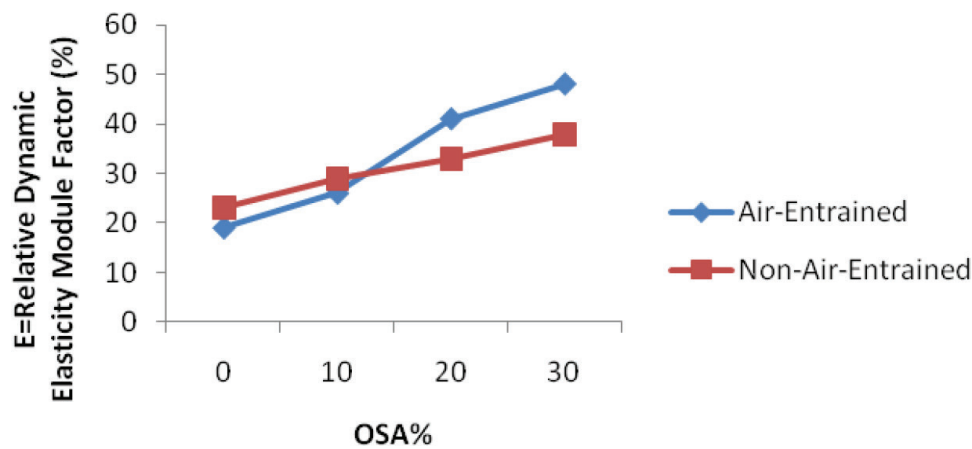


Figure 3. Relationship between relative dynamic elasticity module and addition percentage in air-entrained and non-air-entrained samples.

4. Conclusion and recommendations

By firing oil shale rocks at different temperatures, oil shale ash (OSA) was obtained. With a series of physical and chemical tests performed, in order to obtain the maximum performance, the ratio of the ash to be added to PC (Portland cement) was suggested. As a conclusion, it was determined that all chemical test values of the cement obtained as a result of addition of OSA, which was obtained by firing at 700 °C, to PC (Portland cement) with 15% ratio (when ignition is considered separately, which was also 0.65% greater) were within the limits of TS EN 197-1 [15].

In this study which was performed to determine the resistance of concrete produced with 15% OSA-added cement to freezing-thawing effects, air-entrained and non-air-entrained concretes were produced and weight loss factor, press strength factor, and relative dynamic elasticity module was investigated. As a result of the performed studies, it was observed that optimum results could be taken from concrete samples produced by addition of 15% of OSA to PC 42.5 cement.

In examinations about weight loss of air-entrained and non-air-entrained samples, it was seen that by freezing-thawing cycles there occurred more breaks and weight decreases for air-entrained samples. This fact demonstrates the protection of concrete against frost by air-entraining substance.

In experiments of press compressive strength, the compressive strength (loss) factor (DF_f) found by interpolation for those with 15% addition was 24.3% for non-air-entrained samples and 20.7% for air-entrained samples. Therefore, for resistance to freezing-thawing, the air entraining (HK) chemical substance that is added as 0.5% of the cement weight decreased the strength (loss) factor (DF_f) by about 14%. These values comply with the condition that compressive strength loss in freezing-thawing experiments should be not more than 20%.

Although frost resistances of air-entrained concrete samples are more than that of non-air-entrained concrete samples, their compressive strength is less. The alterations in compressive strength might change based on amount and type of air entrainment.

According to relative dynamic elasticity module (E) examination, for those with 15% addition the value found by interpolation is 33% for non-air-entrained samples and 31% for air-entrained samples. When this result is compared with the limit value for which the relative dynamic elasticity module loss would be maximum 30%, the suitability of air-entrained 15% OSA + HK samples (although the limit is a little bit forced) was observed. Relative dynamic elasticity module (E) is an indicator of durability in concretes.

In air-entrained samples, there is an inverse relation between OSA addition ratios and ultrasonic measurement values that are basis for compressive strength. As pozzolanic addition ratio increases in concrete structure, compactness increases, but pressure loss does not increase because there is a relation between water/binder ratio and compressive strength of air-entraining substance. As air-entraining substance makes closed air gaps in the inner structure of the concrete, the compressive strength is negatively affected. By decreasing Water/Binder ratio in concrete, the number and size of capillary small channels might be decreased. So, less water would enter inside of concrete by capillaries. This fact also shows itself by decreasing permeability.

In places where freezing-thawing is frequently happening, concrete with 15% OSA addition can be used. It is preferred to use mentioned concrete in detail elements, not in structural building elements that are exposed to flexural strength. This includes construction of pavement concrete and so on.

As a conclusion, as 15% OSA-added concretes show performance over desired limit values against freezing-thawing, they can be used in engineering structures of airports, highways, railways and in building elements in buildings as walls, floorings, columns and beams, and in every kind of coatings. So, by adding OSA, which was a waste product, to Portland cement, an economic benefit would be obtained.

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