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Mechanical properties and durability of alkali-activated slag repair mortars containing silica fume against freeze-thaw cycles and salt scaling attack

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Abstract. Freeze and thaw phenomena in cold regions are the main cause of severe damage to concrete structures. Alkali-activated slag repair mortars, which are introduced as a suitable material for the replacement of Portland cement, can be used as the protective coating for these damaged structures. The mechanical properties and durability of this coating layer should be studied. In this study, the mechanical properties and durability of alkali-activated slag repair mortars with silica fume (SF) participation as inorganic additives against freeze-thaw and salt scaling attacks have been investigated. In order to evaluate the effects of alkaline activators type, the ratio of these solutions to Pozzolan (Pozz), and the use of SF as a substitute base material, these three factors were considered as the main variables to produce 12 alkali-activated slag mortar mixtures. To investigate their mechanical properties, compressive strength, tensile adhesion strength, and drying shrinkage tests were conducted. Also, mortar specimen length change, compressive strength loss, weight loss, and dynamic elastic modulus were measured to evaluate the durability features against freeze-thaw and salt scaling attacks. According to the results, in addition to higher compressive strength and adhesion resistance of alkali-activated slag repair mortars, these mortars showed at least 30% better durability against freeze-thaw and salt scaling attacks than cement-based repair mortar. Also, alkali-activated slag mixtures containing potassium hydroxide, alkaline solution (AS) to Pozz ratio of 0.7, and SF had the best mechanical properties and frost resistance among all mixtures.

Keywords: alkali-activated slag repair mortar; silica fume; durability; freeze-thaw resistance; salt scaling attack

1. Introduction

Today, the early demolition of concrete structures due to their destructive environmental conditions and improper concreting and curing has caused designers to pay more attention to durability than mechanical properties. Freeze and thaw phenomena in cold regions are the main cause of severe damage to concrete structures. Freezing water in concrete can lead to cracking and other concrete damages (Valenz 2007). Various factors such as the distance, which the water flows to release pressure, and the degree of saturation can control the frost resistance of concrete (Sabir 1997). Therefore, the use of air-entraining admixtures and reduced concrete permeability can improve its resistance to freezing (Ebrahimi *et al*. 2018). Due to their permeability, cementitious materials usually exhibit low durability against freeze-thaw cycles (Fan *et al*. 2013). Besides, the cement industry consumes 5% of the total energy consumption in the world industries, and consequently, about 7% of the total carbon dioxide produced in the world is related to this industry (Moodi *et al*. 2021, Ramezanianpour *et al*. 2018). For this reason, numerous studies have been conducted to find a suitable and durable alternative to cementitious materials. Geopolymer is known as one of the most appropriate materials that utilize industrial by-products and reduces greenhouse gas emissions due to cement removal (Amran *et al*. 2020, Rathinam *et al*. 2020).

Geopolymer is an amorphous silicate polymer that is formed in an alkaline environment. In other words, geopolymers are aluminosilicate polymers derived from the alkali activation of minerals containing silicon and aluminum (Nawaz *et al*. 2020, Shaikh 2014). Much research has been performed on the properties of geopolymer materials in the fresh and hardened state. The prior studies show that many factors such as type and concentration of alkali activator, silicate modulus can affect geopolymer materials' properties in the fresh and hardened state. Zhang *et al*. (2018) reported that increasing the concentration of alkali activator reduces the workability of the alkali-activated slag up to 40% due to decreasing water content and the use of KOH solution as an activator has more workability than NaOH solution about 10%. Jafari and Ramezanianpour (2016) demonstrated that the silicate modulus has a vital role in the mortar workability, so that increasing it reduces flow-ability up to 40%. Few studies have been conducted on the effect of water content on the workability of alkali-activated slag mortars. In one of these studies, Kotwal *et al*. (2015) concluded that excessive water use in the activator solution increases workability by about 100%. For this reason, mortar flow-ability will be higher when the AS to Pozz ratio increases.

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It is accepted that geopolymer-based material has mechanical properties enhancement. Some factors, such as curing conditions and temperature, mixture proportions, mass ratios, and alkaline activators molarity, can affect the mechanical properties of alkali-activated slag mixtures (Singhal 2017, Patil *et al*. 2014). Increasing the water to alkaline activator ratio from 10 to 12.5 reduces the compressive strength of mortars by 15 MPa (Jindal 2019). In contrast, increasing the curing time from 10 to 90 hours and temperature from 30 to 90ºC increase the compressive strength of geopolymer mortars up to 100% and 70%, respectively (Rajesh *et al*. 2013, Ridtirud *et al*. 2011). Another major factor influencing alkali-activated slag samples' compressive strength is the type and concentration of alkaline activator consumption such as potassium hydroxide (KOH) and sodium hydroxide (NaOH). In general, potassium hydroxide has a higher compressive strength than sodium hydroxide by about 15% due to its higher molecular mass (Ramezanianpour and Moeini 2018, Rashad and Khalil 2013).

According to previous research, geopolymer materials' porosity up to 50% is lower than the cement-based materials, which is one of the main reasons for the more durability of these materials in destructive environments (Okoye *et al*. 2017, Ramezanianpour *et al*. 2016). Fu *et al*. (2011) reported an only maximum drop of 12% of the relative dynamic elastic modulus (RDEM) in the alkali-activated slag samples after 300 freezing and thawing cycles, which indicates the high durability of these concrete in cold regions. Also, in Değirmenci (2018) research, Results showed that the weight loss and compressive strength loss after 25 freeze-thaw cycles of alkali-activated slag were 3% and 15% less than geopolymer mortar based on fly ash and 12% and 40% less than geopolymer mortar based on natural Pozz, respectively.

Like cementitious-based materials, SF can improve the mechanical properties and durability of alkali-activated slag materials (Ramezanianpour and Moeini 2018, Hasnaoui *et al*. 2019, Kurtoglu *et al*. 2018). In the Rostami and Behfarnia (2017) research, replacing 5% SF instead of furnace slag has been introduced as an optimum replacement level.

In this study, the possibility of using the furnace slag and SF as industrial by-products in the form of alkali-activated slag mortars as a sustainable and durable material to repair damaged structures in cold regions was investigated. Evaluation of the sample's RDEM, length change, weight loss, and compressive strength loss after applying freeze and thaw cycles proposed in the standards and previous research have been used to determine the durability. Besides the compressive strength test, surface adhesion resistance and drying shrinkage tests have been performed to investigate the mechanical properties of the specimens.

2. Experimental program

2.1 materials

To prepare the cementitious-based mortar mixtures, type 1 cement supplied by Delijan Company was used. Furnace slag and SF as supplementary cementitious materials were used to prepare the alkali-activated slag-based mortar. Chemical analyses and physical characteristics of these materials were compared in Table 1. Sodium hydroxide with a purity of more than 97% and molecular mass 40 and potassium hydroxide with a purity of more than 85% and molecular mass of 56.11 were dissolved in the tap water to produce six molar alkaline activator. Sodium silicate supplied by Iran silicate industrial Company with silicate modules (SiO_2/Na_2O) of 2.33, specific gravity of 1.559 gr/cm^{3,} and water content of 48% were provided. Silica sand with a specific density of 2.65 based on the ASTM C128 (ASTM 2001) and a maximum diameter of 1 mm has been used to execute layers with 20 to 30 mm thickness alkali-activated slag repair mortar.

Table 1 Physical and chemical properties of binders

Chemical Composition*	Portland Cement (%)	Furnace Slag (%)	Silica Fume (%)
SiO_2	21.55	37.40	94.90
Al_2O_3	5.89	7.10	0.40
Fe_2O_3	3.72	0.55	1.14
CaO	63.78	42.80	0.70
MgO	1.35	6.40	0.56
SO_3	1.89	1.95	0.15
Na_2O	0.53	0.28	0.20
K_2O	0.30	1.14	0.70
Loss On Ignition (LOI)	0.99	2.28	1.25
Physical Properties	Portland Cement	Furnace slag	Silica Fume
Specific gravity	3.09	2.86	2.16
Fineness** (cm^2/gr)	3299	5080	342200

* Chemical composition is specified based on the X-Ray fluorescence method (XRF).

** Fineness is determined by gas adsorption according to ISO 9277 (ISO 2010).

2.2 Mixture proportion

Based on the Ramezanianpour and Moeini (2018) and Jafari and Ramezanianpour (2016) researches, twelve mix designs (Table 2) were considered to evaluate the mechanical properties and durability of alkali-activated slag mortars. In these mixtures, base materials weight of 463.8 Kg/m^3, alkaline activator concentration of 6 molars, aggregate to base material (pozzolan) weight ratio of 2.75, and sodium silicate to alkaline activator ratio of 0.4 were used constantly. While, in order to investigate the effects of alkaline activators type, the ratio of these solutions to pozzolans, and the use of SF as an alternative base material, these three factors were considered the main variables of mix designs.

To compare alkali-activated slag mortars with cement-based mortars, a cement control sample containing 592.6 kg/m^3 of cement and a water-to-cement ratio of 0.487 was made according to Table 2.

Table 2 Alkali-activated slag and cement-based mortars mix proportion

Alkali-activated slag repair mortar						
Mixture ID	AS	Molarity	Agg to Pozz	AS to Pozz	SF (%)	SS to AS
K7M0	KOH	6	2.75	0.7	0	0.4
N7M0	NaOH	6	2.75	0.7	0	0.4
K7M5	KOH	6	2.75	0.7	5	0.4
N7M5	NaOH	6	2.75	0.7	5	0.4
K8M0	KOH	6	2.75	0.8	0	0.4
N8M0	NaOH	6	2.75	0.8	0	0.4
K8M5	KOH	6	2.75	0.8	5	0.4
N8M5	NaOH	6	2.75	0.8	5	0.4
K9M0	KOH	6	2.75	0.9	0	0.4
N9M0	NaOH	6	2.75	0.9	0	0.4
K9M5	KOH	6	2.75	0.9	5	0.4
N9M5	NaOH	6	2.75	0.9	5	0.4

Cement-based repair mortar				
Mixture ID	Cement (kg/m^3)	Agg to cement	W/C	SP (kg/m^3)
OPC	592.6	2.75	0.487	1.778

AS: alkali solution, Agg: aggregate, Pozz: pozzolan, SF: silica fume, SS: sodium silicate.

2.3 Sample preparation

After completion of mixing the mixture components and measuring the workability, the samples required for the intended tests are molded in two layers. The vibrating table was used to properly compaction and remove the air bubbles from the samples in each layer. Then samples were retained in the wet room for one day to prevent excess water evaporation. Finally, the samples were de-molded and were stored in water at 25°C until the test.

3. Test method

3.1 Workability test

American standard test method (ASTM) C1437 (ASTM 2015) and flow table have been used for the workability test of alkali-activated slag and cement fresh mortars. For this purpose, the mortar diameter was measured at two points. The percentage increase in the average diameter of the mortar compared to the initial diameter was reported as the workability test result.

3.2 Mechanical properties tests

According to the ASTM C109 (ASTM 2020), the compressive strength test was performed at five different ages on the cubic specimens of alkali-activated slag and cement-based mortars with dimensions of 50×50×50 mm. In order to investigate the drying shrinkage, the ASTM C596 (ASTM 2018) was used for the prismatic sample with dimensions of 285×25×25 mm. The samples were stored for 91 days in a container with a relative humidity of 50% and a temperature of 25°C, and their dimensions were measured every two weeks. According to the ASTM D7234 (ASTM 2019) and using a bond test device, the adhesion resistance of the 25 mm layer of mortar on the concrete substrate was measured at 7 and 28 days. Measurement of capillary water absorption of mortar samples at the age of 28 days was performed according to BS EN 480 (BS-EN 2005) and disc samples with 100×50 mm dimensions.

3.3 Salt scaling test

Due to the fact that the method presented in the ASTM C672 (ASTM 2012) is related to the performance and durability of concrete samples, and since no acceptable reference has been provided for the durability of mortars, especially repair mortars in these physical conditions, according to the recommendations of previous research, in this study, the procedure proposed in this standard has been used to investigate the salt scaling of samples (Çopuroğlu and Schlangen 2008, Dimitre 2012). To perform this experiment, prismatic samples with dimensions of 285×75×75 mm were cured in water for 14 days and dried in air for 14 days, then placed in 3% sodium chloride solution. Each freezing and thawing cycle includes 16 hours of freezing at -18±3°C and then 8 hours of thawing at 21±3°C. After every five cycles, the samples' weight loss is measured, and the available solution is replaced. This test continued up to 50 cycles according to the standard.

3.4 Rapid freezing and thawing test

In this study, according to previous research recommendations (Cai *et al*. 2013, Garbalińska and Wygocka 2014), the procedure proposed in method A of ASTM C666 (ASTM 2015) has been used to evaluate the performance and durability of mortar samples. Prismatic samples with dimensions of 285×75×75 mm were made to measure weight changes, length changes, and RDEM, and cubic samples with dimensions of 50×50×50 mm were prepared for measuring compressive strength and weight changes. Based on the ASTM C666 (ASTM 2015), specimens for rapid freezing and thawing test were cured for 14 days then placed in the temperature range of -18 to 4°C for each cycle. Temperature changes in each freezing and thawing cycle were performed and controlled using an automatic chamber of the Azmoon Company.

The RDEM of mortar samples was measured according to the BS 188: Part 203 and using the dynamic elastic modulus device of the Proceq Company. In order to measure the wave velocity and transmission time, 54 kHz probes were used for the P wave, and 40 kHz probes were used for the S wave. By measuring a P-wave transmission time and an S-wave transmission time with Pundit Lab, the P-wave modulus (M) and the shear modulus (G) be able to determine.

P-wave modulus (M)

$$M = \rho V_P^2 \tag{1}$$

Where ρ is the density of the material in kg/m^3, and V_P is the pulse velocity of the P-wave in km/s.

Shear – modulus (G)

$$G = \rho V_S^2 \tag{2}$$

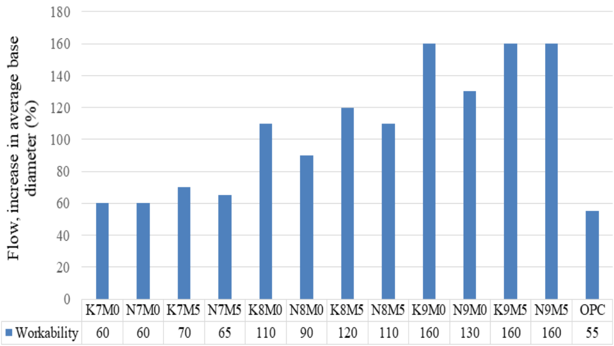


Fig. 1 Flow of fresh mortars

Where ρ is the density of the material in kg/m^3, and V_S is the pulse velocity of the S-wave in km/s.

Poison's ratio (v) can be determined by using the equations above

$$v = \frac{M-2G}{2M-2G} = \frac{\rho V_P^2 - 2\rho V_S^2}{2\rho V_P^2 - 2\rho V_S^2} = \frac{V_P^2 - 2V_S^2}{2V_P^2 - 2V_S^2} = \frac{V_P^2 - 2V_S^2}{2(V_P^2 - V_S^2)} \tag{3}$$

Eventually, dynamic elastic modulus (E_d) in MN/m^2 is given by the following equation

$$E_d = \rho v^2 \frac{(1+v)(1-2v)}{(1-v)} \tag{4}$$

Where v is the dynamic Poison's ratio, ρ is the density in kg/m^3, v is the pulse velocity in km/s.

4. Result and discussion

4.1 Workability (ASTM C1437-15)

Fig. 1 shows the results of the workability test of the alkali-activated slag and control fresh mortars. According to observations, mixtures with the AS to the Pozz ratio of 0.9 had severe bleeding due to the high amount of water used in the mix. These four mixtures were removed from the repair mortar mixtures due to non-compliance with the required workability parameters. Tests of mechanical properties and durability were performed on the remaining eight combinations. Mortar samples prepared with AS to the Pozz ratio of 0.8, due to their higher water content, had better workability (approximately 40%) than mixtures with a ratio of 0.7. Since the potassium hydroxide molecular mass is higher, the mortars made with KOH activator had higher workability by an average of 10% than mixtures containing NaOH activator. Unlike the cement-based compounds, SF has increased the workability of alkali-activated slag mortars by an average of 12%, similar to results observed in research (Jafari and Ramezanianpour 2016, Ramezanianpour and Moeini 2018).

Table 3 Pull-off adhesion strength test results

Mixture ID	7 days		28 days	
	Bond strength (MPa)	Failure Mode	Bond strength (MPa)	Failure Mode
K7M0	1.11	Repair mortar	1.34	Interface
N7M0	1.34	Concrete substrate	1.61	Concrete substrate
K7M5	1.25	Interface	1.40	Interface
N7M5	1.56	Concrete substrate	1.70	Concrete substrate
K8M0	0.97	Repair mortar	1.29	Repair mortar
N8M0	1.31	Interface	1.47	Concrete substrate
K8M5	1.29	Interface	1.43	Concrete substrate
N8M5	1.38	Concrete substrate	1.54	Concrete substrate

4.2 Compressive strength (ASTM C109-20), tensile adhesion strength (ASTM 7234-19), and capillary water absorption (BS EN 480-05)

The results achieved for compressive strength, pull-off adhesion strength, and capillary water absorption of mixtures are demonstrated in Fig. 2, Table 3, and Fig. 3, respectively.

Based on the results of the compressive strength and

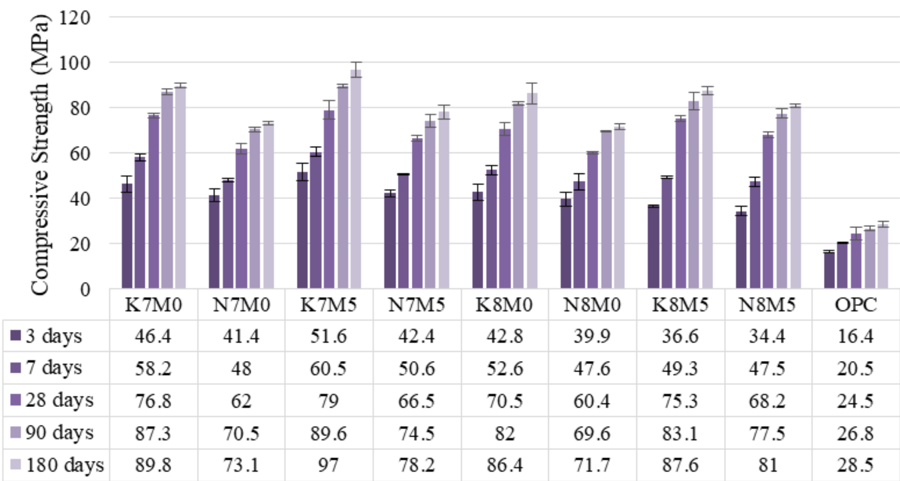


Fig. 2 Compressive strength test results

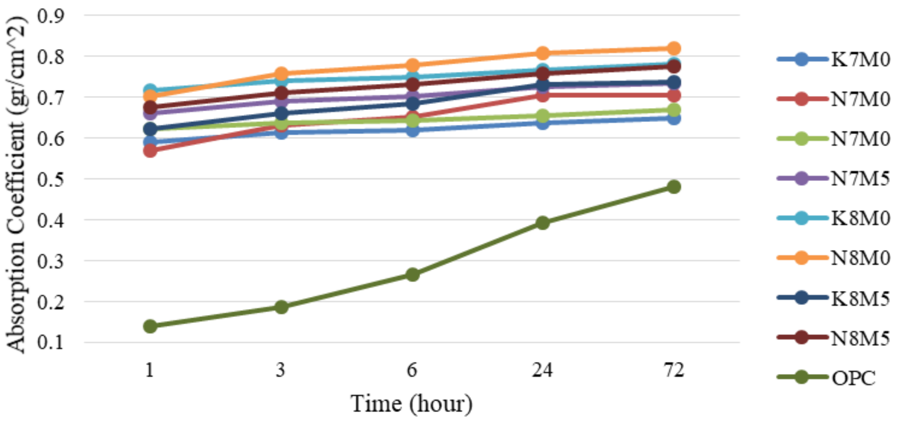


Fig. 3 Capillary water absorption test results

Table 4 the effect of SF according to statistical analysis

Variable: SF (%)	Mixture ID	Average of Compressive Strength (MPa)	"*t*" Calculated value	Average of Tensile Adhesion Strength (MPa)	"*t*" Calculated value	Average of Water Absorption (gr/cm^2)	"t" Calculated value	"t" Critical Two-tail Value
0	K7M0	76.8	1.003	1.34	10.392	0.650	1.732	4.302
5	K7M5	79.0		1.40		0.670		
0	N7M0	62.0	5.196	1.61	2.598	0.707	9.699	
5	N7M5	66.5		1.70		0.735		
0	K8M0	70.5	4.619	1.29	4.850	0.783	15.588	
5	K8M5	75.3		1.43		0.738		
0	N8M0	60.4	11.258	1.47	6.062	0.821	5.196	
5	N8M5	68.2		1.54		0.776		

pull-off adhesion strength tests, increasing the curing period (age) has increased the compressive strength of alkali-activated slag and control mortars. Also, in all ages, the compressive strength of all alkali-activated slag mix designs up to 300% is higher than the cement-based mixture. Among all combinations of all ages, the mixtures of K7M5 by compressive strength of 97 MPa after 180 days and N7M5 by tensile adhesion strength of 1.7 had the highest compressive strength and tensile adhesion strength, respectively. According to capillary water absorption test results, it is clear that all alkali-activated slag mortars had higher water absorption rates than the control sample, which high cracking potential of alkali-activated slag mixtures with slag based in environments with a temperature of more than 50°C can be interpreted as the main reason for it. The percentage of alumina in the furnace slag chemical composition has a significant effect on the performance of the sample. If it is more than 3%, the CAH10 and C4AH13 hexagonal hydrates are converted to C3AH6 cubic hydrates due to heat. Crystallization in these steps is the main cause of loss of performance and cracking of slag-based paste containing high alumina amounts. Among the alkali-activated slag mortars, mixtures of K7M0 and N8M0 had the lowest (0.65 gr/cm^2) and highest (0.82 gr/cm^2) water absorption, respectively.

In the next step, statistical analysis was used to evaluate the effect of SF, type of alkaline solution, and the ratio of AS to Pozz on the compressive strength, tensile adhesion strength, and 72-hour water absorption of 28 days cured alkali-activated slag mortars. For statistical analysis, the results achieved in the laboratory tests compare by paired-samples t-test (α=0.05).

4.2.1 Silica fume

The statistical analysis results for evaluating the effect of using SF as substitutes for furnace slag are summarized Table 4.

The results indicate that the incorporation of SF increased the compressive strength and tensile adhesion strength of mortar specimens due to its filling effect and the formation of more resistant gels. The statistical analysis showed the significant difference in mixtures with and without SF, which confirms the positive impact of SF on improving alkali-activated slag-based mortars' mechanical properties. Also, it is evident that silica fume addition is effective in the case of strength development. Since several SF mixtures with strength lower than mixtures without SF at earlier ages showed higher compressive strengths at 180 days, this is mainly attributed to the high pozzolanic reactivity of silica fume, which produces more C-S-H gel in the paste (Ramezanianpour and Moeini 2018). The experimental and statistical analysis results demonstrate that the incorporation of SF had no constant effect on the 72 hours of water absorption of alkali-activated slag-based mortars. Based on these results, SF in mixtures with AS to Pozz ratio of 0.7 increased the water absorption while reducing the water absorption in mixtures with AS to Pozz ratio of 0.8.

4.2.2 Alkaline solution

Table 5 The effect of the alkaline solution according to statistical analysis

Variable: Alkaline solution	Mixture ID	Average of Compressive Strength (MPa)	"t" Calculated value	Average of Tensile Adhesion Strength (MPa)	"t" Calculated value	Average of Water absorption (gr/cm^2)	"t" Calculated value	"t" Critical Two-tail Value
K	K7M0	76.8	14.241	1.34	15.588	0.650	5.807	4.302
N	N7M0	62.0		1.61		0.707		
K	K7M5	79.0	6.186	1.40	25.981	0.670	56.292	
N	N7M5	66.5		1.70		0.735		
K	K8M0	70.5	6.032	1.29	31.177	0.783	5.485	
N	N8M0	60.4		1.47		0.821		
K	K8M5	75.3	122.976	1.43	4.763	0.738	32.909	
N	N8M5	68.2		1.54		0.776		

Table 6 The effect of AS to Pozz ratio according to statistical analysis

Variable: AS to Pozz	Mixture ID	Average of Compressive Strength (MPa)	"t" Calculated value	Average of Tensile Adhesion Strength (MPa)	"t" Calculated value	Average of Water absorption (gr/cm^2)	"t" Calculated value	"t" Critical Two-tail Value
0.7	K7M0	76.8	4.744	1.34	8.660	0.650	11.518	4.302
0.8	K8M0	70.5		1.29		0.783		
0.7	N7M0	62.0	1.155	1.61	24.249	0.707	21.939	
0.8	N8M0	60.4		1.47		0.821		
0.7	K7M5	79.0	1.942	1.40	1.732	0.670	23.556	
0.8	K8M5	75.3		1.43		0.738		
0.7	N7M5	66.5	9.815	1.70	9.238	0.735	71.014	
0.8	N8M5	68.2		1.54		0.776		

The type of AS is one of the effective parameters for alkali-activated slag-based mortars and concrete properties. In this study, mixtures with KOH and NaOH as alkaline solutions were compared, which experimental tests and statistical analysis results are displayed in Table 5.

The statistical analysis results revealed a significant difference in compressive strength, tensile adhesion strength, and water absorption of samples with KOH and NaOH as an alkaline activator to produce alkali-activated slag mortars. According to the experimental test results, using the KOH activator solution, due to higher molecular mass and less water content, increased the compressive strength of alkali-activated slag mortars by 15%. At the same time, the specimens made with NaOH had the higher tensile adhesion strength. More porosity of the NaOH mixture can be a probable reason for its better adhesion to the concrete substrate. As is evident from the statistical analysis results of water absorption, there were significant differences between the mixtures made with KOH and NaOH. Due to its lower water content, KOH alkaline activator reduces the porosity and, subsequently, water absorption of alkali-activated slag mortar mixtures by 7%. This reduction of water absorption by using KOH alkaline activator was reported in the research of Rashad and Khalil (2013), Law *et al*. (2015), which indicated the use of KOH as an alkaline activator, improves the mechanical properties and capillary water absorption of alkali-activated slag mixtures by 10%.

4.2.3 Alkaline solution to Pozzolan ratio

As mentioned in section 2.2, the ratio of AS to Pozz was considered the main variable of mix designs studied in this research. Table 6 shows the experimental and statistical analysis results for comparing mixtures with alkaline solution to the Pozz ratio of 0.7 and 0.8.

According to the laboratory test and statistical analysis results, the compressive strength test results are contradictory. There were no statistically significant differences between the compressive strength of mixtures N7M0 and N8M0 and K7M5 and K8M5. Also, experimental test results for other combinations show the dual function of increasing the AS to Pozz ratio. Less water content in mixtures with AS to Pozz ratio of 0.7 and more workability and better compaction for mix designs with AS to Pozz ratio of 0.8 can be mentioned as a probable reason for this dual function. Statistical analysis displays significant differences for tensile adhesion strength of all mixtures except between K7M5 and K8M5. The rest of the results indicate the better adhesion of mixtures with AS to Pozz ratio of 0.7 to the concrete substrate. The existence of significant differences in statistical analysis for water absorption illustrates that the AS to Pozz ratio of 0.7 decreases the water absorption of alkali-activated slag mortars to a considerable extent. This reduction is due to less water content and less porosity of mixtures with AS to the Pozz ratio of 0.7.

4.3 Drying shrinkage test (ASTM C596-18)

The results of the drying shrinkage of alkali-activated slag and control mortars are shown in Fig. 4. As it is clear from the results, the drying shrinkage of alkali-activated slag samples was more than the control sample due to the differences in geopolymerization and hydration processes.

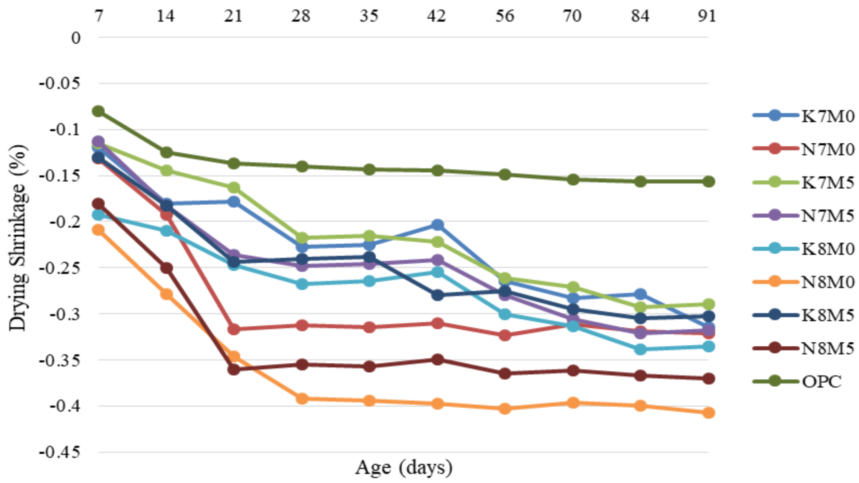


Fig. 4 Drying shrinkage test results

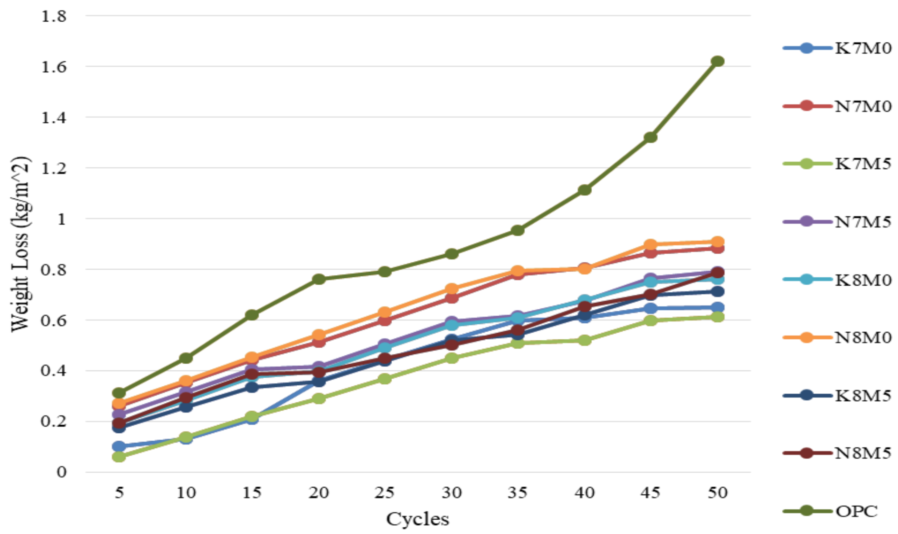


Fig. 5 Weight loss results of salt scaling test

In the geopolymerization process, water is released, and this water is removed from the alkali-activated slag matrix during curing and subsequent drying periods. In contrast, water is used in the cement hydration process. Ramezanianpour *et al*. (2018), in their research, noted that alkali-activated slag concrete has more drying shrinkage than cement-based concrete. Also, KOH alkaline activator and 5% SF as a substitute for furnace slag reduced the shrinkage of alkali-activated slag mortars by 7.5% and 14%, respectively. As expected, due to the higher water content, the mixture with the AS to the Pozz ratio of 0.8 had more drying shrinkage by 15%. It is evident from the results that more than 90% of the drying shrinkage of NaOH mixtures is until 28 days. While drying shrinkage in mixtures containing KOH alkaline activator continues up to 91 days. One possible reason could be less water content in samples made with potassium hydroxide. Less water content causes smaller capillary pores, which increases the time it takes for water to evaporate from the sample. However, more research is needed to state the main reasons.

4.4 Salt scaling test (ASTM C672-12)

The weight loss resulting from the salt scaling test of alkali-activated slag and control mortar is shown in Fig. 5. According to the results, alkali-activated slag mixtures had high durability against freezing and thawing cycles in the 3% sodium chloride solution. In contrast, the control mixture had a high weight loss before the end of the 50 cycles. Among alkali-activated slag mixtures, K7M5 had the best performance in this environmental condition, following the results of compressive strength tests and capillary water absorption. In contrast, the worst performance among alkali-activated slag repair mortars is related to the N8M0, which includes sodium hydroxide as the alkaline activator, the AS to the Pozz ratio 0.8, and without SF. Due to the high amount of water, higher AS to Pozz ratio, the lack of sufficient formation of silicate gels in this mixture, and the compressive strength test results and capillary water absorption have been somewhat predictable. As shown in Fig. 5, the weight loss rate for all mixtures is higher in the earlier cycles. During measuring the samples weight loss in the initial cycles, it was observed that the severity of the sample failure was higher in the corners and edges due to the stress concentration. Over time, the corners

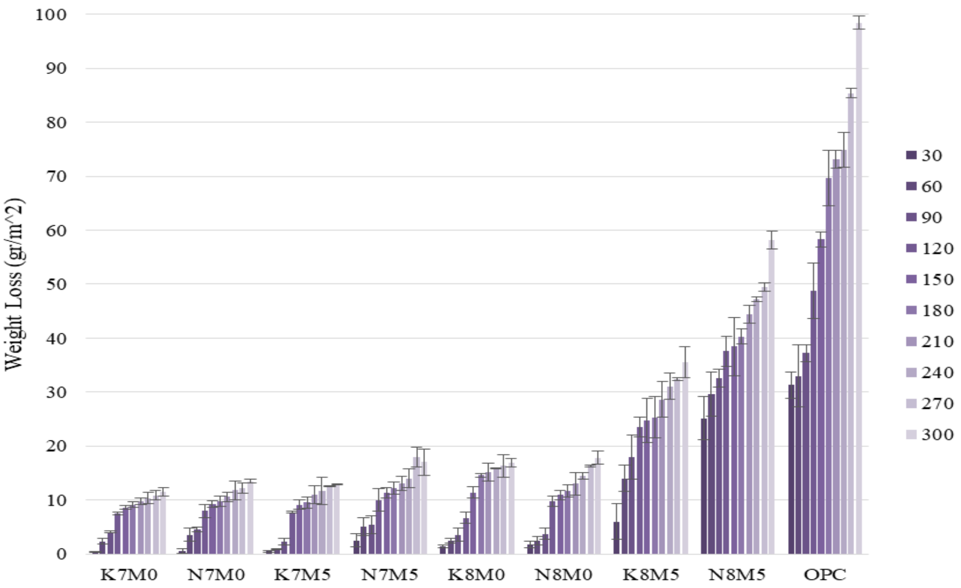


Fig. 6 Weight loss results of freezing and thawing test for prismatic samples

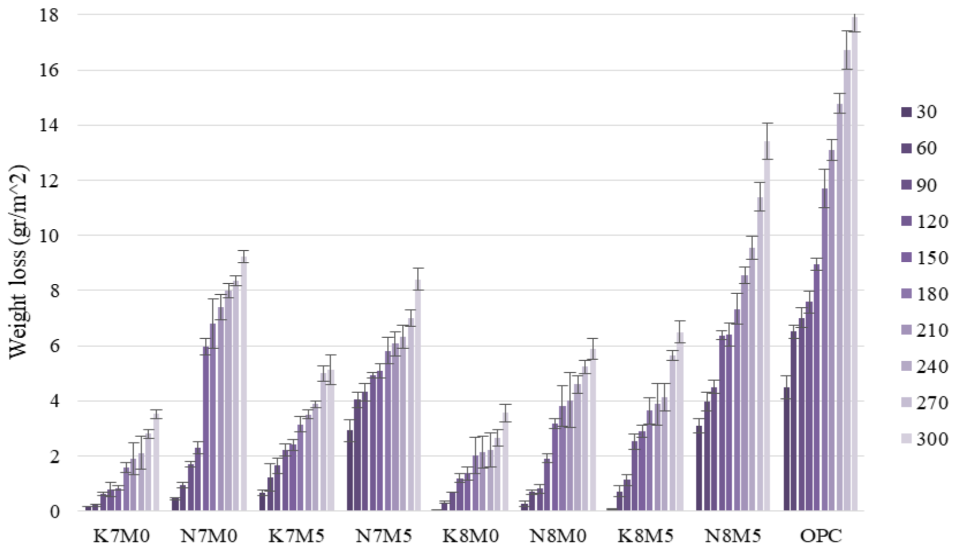


Fig. 7 Weight loss results of freezing and thawing test for cubic samples.

and edges of the specimens were completely destroyed and the weight loss rate decreased.

4.5 Weight loss, length change and dynamic elastic modulus at rapid freezing and thawing test (ASTM C666-15)

One of the main factors for investigating the durability against freezing and thawing cycles is weight loss measurement. The results of this experiment for alkali-activated slag and control mortars in two forms of prismatic samples with dimensions of 285×75×75 mm and cubic samples with dimensions of 50×50×50 mm are indicated in Figs. 6-7, respectively. According to the results shown in these figures, all alkali-activated slag mixtures had a lower weight loss than the control mixture. The comparison of cubic and prismatic specimens displays that the weight loss is related to the samples' surface area. For this reason, prismatic specimens with more surface area had more weight loss up to 5 times than cubic samples. For example, OPC cubic samples had 12 gr/m^2 weight loss, while OPC prismatic specimens had 98 gr/m^2 weight loss after 300 freezing and thawing cycles. Among all mix designs, OPC and N8M5 had the worst freeze-thaw resistance in both the cubic and prismatic samples test.

Another factor proposed in the standard is the measurement of the length change of the samples during the freezing and thawing cycles, in which its results are shown in Fig. 8. The results comparison shows that all alkali-activated slag mortars had lower length change than the control samples. According to the results, OPC and N8M5 with length increase of 0.013 and 0.010 had the highest length increase, respectively. While K7M5 and K7M0 had the best performance with length increase of 0.002. The lower length change of KOH mixtures can be related to its higher molecular mass than sodium hydroxide and higher

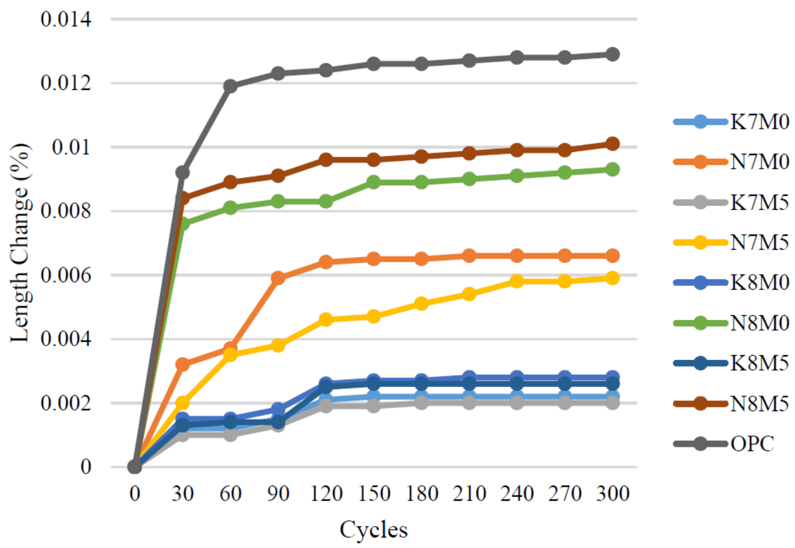


Fig. 8 Length change result of freezing and thawing test

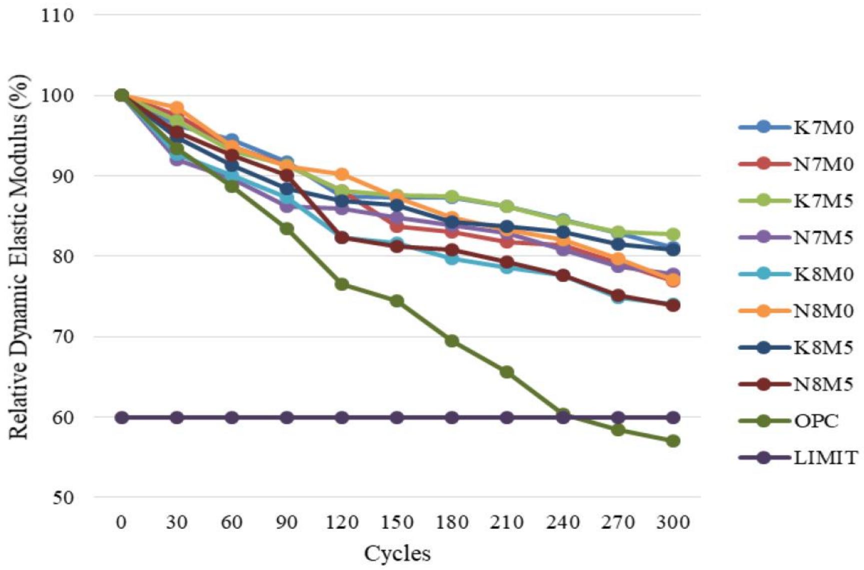


Fig. 9 Dynamic elastic modulus results of the freezing-thawing test

compressive strength than OPC and NaOH mixtures.

Fig. 9 illustrates the results of measuring the RDEM of repair mortars after applying freezing and thawing cycles. As demonstrated in the graph, none of the alkali-activated slag mortars reached 60% RDEM until the completion of the 300 cycles applied, which indicates the high durability of them in these physical conditions. Among the alkali-activated slag mixtures, K7M5 with RDEM OF 84% had the best performance, and N8M5 with RDEM of 74% had the worst performance, while the OPC mixture had the RDEM OF 57% after applying 300 freeze-thaw cycles, which indicate better ductility of alkali-activated slag based mortars. Similar to these results were achieved in Fu *et al.* (2011) research. They reported that the geopolymer concrete had a 12% reduction of RDEM after 300 freeze-thaw cycles.

Similar to section 4.2, statistical analysis was conducted to investigate the effect of the main variables of this research (SF, type of alkaline solution, and the ratio of AS to Pozz) on the weight loss, length increase, and RDEM of alkali-activated slag repair mortars after applying 300 freezing and thawing cycles. Paired-samples t-test (α=0.05) was used to compare experimental test results.

4.5.1 Silica fume

Table 7 lists the experimental and statistical analysis results to investigate the effect of SF as a Pozzolan on the durability of alkali-activated slag mortars.

The statistical analysis results indicate no significant differences in weight loss in mixtures with and without SF made with AS to Pozz ratio of 0.7. While in the combinations made with AS to Pozz ratio of 0.8, SF's incorporation increases the weight loss dramatically. Statistical analysis displayed no significant differences for mix designs with and without SF in measuring length change of specimens. Probably, less amount of SF cannot affect the length change of samples. Although, in the measuring and statistical analysis of mixtures with AS to Pozz ratio of 0.7, the incorporation of SF increases the RDEM

Table 7 The effect of SF on freeze-thaw durability according to statistical analysis

Variable: SF (%)	Mixture ID	Average of Weight loss (gr/m^2)	"t" Calculated value	Average of Length increase (%)	"t" Calculated value	Average of RDEM (%)	"t" Calculated value	"t" Critical Two-tail Value
0	K7M0	11.49	2.134	0.0022	2.371	81.11	29.098	4.302
5	K7M5	12.87		0.0020		82.79		
0	N7M0	13.45	2.385	0.0066	1.102	77.00	5.196	
5	N7M5	17.03		0.0059		77.75		
0	K8M0	16.87	12.907	0.0028	3.464	74.01	117.260	
5	K8M5	35.50		0.0026		80.78		
0	N8M0	17.84	139.880	0.0093	1.125	77.05	109.812	
5	N8M5	58.22		0.0101		73.88		

Table 8 The effect of the alkaline solution on freeze-thaw durability according to statistical analysis

Variable: Alkaline solution	Mixture ID	Average of Weight loss (gr/m^2)	"t" Calculated value	Average of Length increase (%)	"t" Calculated value	Average of RDEM (%)	"t" Calculated value	"t" Critical Two-tail Value
K	K7M0	11.49	2.442	0.0022	38.105	81.11	71.187	4.302
N	N7M0	13.45		0.0066		77.00		
K	K7M5	12.87	2.511	0.0020	7.506	82.79	174.591	
N	N7M5	17.03		0.0059		77.75		
K	K8M0	16.87	3.360	0.0028	28.146	74.01	105.309	
N	N8M0	17.84		0.0093		77.05		
K	K8M5	35.50	26.235	0.0026	43.301	80.78	119.512	
N	N8M5	58.22		0.0101		73.88		

Table 9 The effect of AS to Pozz ratio on freeze-thaw durability according to statistical analysis

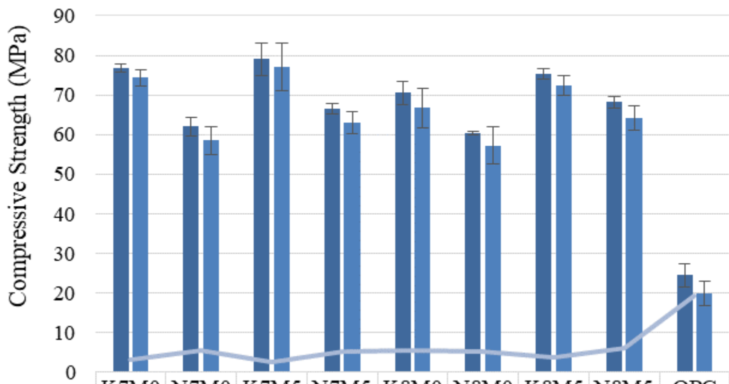
Variable: AS to Pozz	Mixture ID	Average of Weight loss (gr/m^2)	"t" Calculated value	Average of Length increase (%)	"t" Calculated value	Average of RDEM (%)	"t" Calculated value	"t" Critical Two-tail Value
0.7	K7M0	11.49	4.683	0.0022	10.392	81.11	153.720	4.302
0.8	K8M0	16.87		0.0028		74.01		
0.7	N7M0	13.45	6.912	0.0066	23.383	77.00	0.377	
0.8	N8M0	17.84		0.0093		77.05		
0.7	K7M5	12.87	11.631	0.0020	10.392	82.79	43.518	
0.8	K8M5	35.50		0.0026		80.78		
0.7	N7M5	17.03	71.343	0.0059	5.596	77.75	95.758	
0.8	N8M5	58.22		0.0101		73.88		

in the mixtures with AS to Pozz ratio of 0.8 had the dual function. In general, it can be concluded that the use of SF not only improved the durability of alkali-activated slag mortars but also increases the weight loss of mixtures after applying freezing and thawing cycles.

4.5.2 Alkaline solution

The type of AS as an activator in alkali-activated slag mixtures is one of the main factors that can affect the mechanical properties and durability of samples. For this reason, experimental tests and statistical analysis were used to investigate the effect of alkaline solutions on the durability of alkali-activated slag mixtures after 300 freeze-thaw cycles. Their results are indicated in Table 8.

Based on the paired-samples t-test results, there are no significant differences between the mixtures with KOH or NaOH as an alkaline activator except between mixtures K8M5 and N8M5. The weight loss results for K7M5 and N7M5 had 41% differences, while the "t" calculated value for them is 2.511, indicating no significant differences between them. Since the value of t is calculated based on the mean and variance of 3 results for each mixture, statistical analysis is more reliable for comparing different mixtures. This was predictable, based on the results of compressive strength and capillary water absorption on the abrasion resistance of the external aspects of the samples. In the case of N8M5, the higher amount of alkaline activator and water, and the lack of sufficient formation of silicate gels due to them, had led to more pores for the formation of ice crystals. Significant differences were obtained from the statistical analysis for length change and RDEM of mixtures with different alkaline activators. Mortars with KOH alkaline activator had a lower length increase and higher RDEM, which can be due to the higher molar mass and subsequently less porosity of the KOH alkaline activator. Generally, it can be expressed that using KOH as an



	K7M0	N7M0	K7M5	N7M5	K8M0	N8M0	K8M5	N8M5	OPC
28 days*	76.8	62.0	79.0	66.5	70.5	60.4	75.3	68.2	24.5
after 300 cycles	74.3	58.5	77.0	63.0	66.7	57.2	72.4	64.1	19.8
compressive strength loss (%)	3.26	5.65	2.53	5.26	5.39	5.30	3.85	6.01	19.18

Fig. 10 Compressive strength results of freezing and thawing test
(*Results were achieved in section 4.2 (Fig. 2))

alkaline activator increases the durability of alkali-activated slag mortars against freeze-thaw cycles.

4.5.3 Alkaline solution to Pozzolan ratio

The durability of mortar mix designs with AS to Pozz ratios of 0.7 and 0.8 in freezing and thawing environment were compared by experimental and statistical analysis. Their results are demonstrated in Table 9.

As it can clearly be observed, there are significant differences in the weight loss, length increase, and RDEM of all mortar mixtures with AS to Pozz ratios of 0.7 and 0.8 except RDEM of N7M0 and N8M0. Due to less water content and less porosity, mixtures with AS to Pozz ratio of 0.7 had lower weight loss and length increase and higher RDEM, which indicates its effects on the improvement of alkali-activated slag durability mortar mixes in cold regions. These results are consistent with the impact of AS to Pozz ratios on compressive strength and capillary water absorption (section 4.2.3). Based on the results, in most mixtures, higher compressive strength and lower capillary water absorption cause higher durability in freeze and thaw environment. It can be concluded that the reduction of AS to Pozz from 0.8 to 0.7 ratio significantly affected durability mixtures.

4.6 Compressive strength loss at rapid freezing and thawing test

Due to the lack of a standard for evaluating alkali-activated slag repair mortars' durability against freezing and thawing cycles, another factor proposed in previous studies was to compare the compressive strength of cubic samples after applying 300 with 28-day compressive strength of the samples without any cycles. The results of this experiment are presented in Fig. 10. As the results display, the compressive strength loss of alkali-activated slag mixtures was 14.5 lower than the control mixture due to their lower porosity and higher mechanical properties. Which again indicates that alkali-activated slag repair mortars can have a good performance in increasing durability against freezing and thawing cycles. Among the alkali-activated slag mix designs, mixtures containing KOH as the alkaline activator had a lower compressive strength loss than mixtures containing NaOH by 2%, which was expected due to the results of compressive strength tests and other tests of freezing and thawing cycles. In Değirmenci (2018) research, the lowest weight loss and compressive strength loss are reported for alkali-activated slag mortar samples.

5. Conclusions

In this study, 12 alkali-activated slag mortar mix designs were used to investigate mechanical properties and durability in cold environmental conditions. In the second phase of the study, due to severe bleeding of some mixtures, eight final alkali-activated slag mixtures containing furnace slag and SF as a base material and a cement-based control mixture were selected to explore mechanical properties and durability against freezing and thawing cycles. The summarized results of these experiments are mentioned as the following:

- Increasing the AS to Pozz ratio in alkali-activated slag samples improves the workability, but it causes samples bleeding in the ratio of 0.9. The use of SF has also increased the flow-ability of alkali-activated slag mixtures by 12%.
- The formation of secondary gels and the filling role of SF have increased the compressive strength and tensile adhesion strength of alkali-activated slag samples. While both the experimental and statistical analysis results demonstrate that the incorporation of SF had no constant effect on the 72 hours of water absorption of alkali-activated slag-based mortars.
- According to the test results, using the KOH activator solution due to higher molecular mass and less water content increased the compressive strength by 10%. It decreased the capillary water absorption of alkali-activated slag mortars, while the specimens made with NaOH up to 15% gave higher tensile adhesion strength.

• Due to the nature of the geopolymerization process and the release of water compared to water consumption in the cement hydration process, all alkali-activated slag samples' drying shrinkage was more than the control sample. Also, samples containing NaOH had higher shrinkage by about 7.5% than mixtures made with KOH due to the lower molecular mass of NaOH and increasing the mixture water content. In this study, SF's use as a substitute material up to 14% improved the performance of the samples and decreased the drying shrinkage.

• Despite the high durability and slight weight loss of alkali-activated slag repair mortars against salt scaling cycles, the use of SF had a dual function in this environment. While using it improves the durability of mixtures containing potassium hydroxide, it can have a negative effect on the scaling resistance of the other mortars due to the filler performance and the closure of the pores controlling the expansion of the frozen solution.

• According to the experiment results, measuring the RDEM can be a suitable and non-destructive method to assess the performance of repair mortars at any age and physical condition.

• Due to the formation of more silicate gels and creating an alkali-activated slag matrix because of its higher molecular mass, the mix designs containing KOH performed better than mixtures containing NaOH and shown slight weight loss and less RDEM reduction.

• Because of less water content and less porosity, mixtures with AS to Pozz ratio of 0.7 had the lower weight loss (up to 19%) and relative length increase (about 50%) and higher RDEM (approximately 4%) than mixtures with AS to Pozz ratio of 0.8 after applying 300 freezing and thawing cycles. These properties indicate the positive effect of using AS to Pozz ratio of 0.7 on the durability of alkali-activated slag mortar mixes in cold regions.

• According to all experiments conducted in this study, the two mixtures K7M5 and K7M0 performed better than other combinations. They can be used as high-durable repair mortars against cold and frost environmental conditions. In general, mixtures containing potassium hydroxide (than sodium hydroxide), mix designs with the AS to Pozz ratio of 0.7 (than 0.8), and mixtures containing SF (than without SF) have better mechanical and durability properties.

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