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Original Research

A Novel Type of Alkaline Activator for Geopolymer Concrete Based on Zeolite

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Abstract: Geopolymers, as a new class of green cement binders, have been considered as environmental-friendly alternatives to Ordinary Portland Cement (OPC) in recent years. They can potentially reduce negative environmental impacts of OPC; including carbon footprint and energy consumption. In the current work, effects of different alkaline activator solutions on the compressive, indirect tensile, and flexural strengths, water absorption, and resistance to an acidic condition of zeolite-based Geopolymer Concrete (GPC) were investigated. Additionally, a novel type of alkaline activator for GPC was introduced. In this regard, GPC specimens based on zeolite were manufactured and cured at 65 ˚C. The results showed that the addition of NaOH to the mixture after 3 min of mixing KOH and Na_2SiO_3 with dry components (1/3 of the total mixing duration) resulted in the highest compressive, tensile, and flexural strengths as well as the lowest water absorption capacity and weight loss under acidic condition, amongst other cases.

Keywords: Geopolymer concrete; Zeolite; Alkaline activator; Compressive strength.

Introduction

Ordinary Portland cement (OPC) is the most widely used cement in the construction sector [1]. OPC production has significant environmental consequences, including high energy and natural resource consumption, as well as carbon dioxide (CO_2) emissions [2]. Production of 1 ton of OPC produces approximately 0.73-0.99 ton of CO_2 [3]. On the other hand, climate change is one of the most important environmental challenges that has attracted much attention in recent years [4]. Geopolymers have been recently introduced as environmentally safe cementitious materials that can help reduce the environmental impact of OPC. Davidovits introduced geopolymers as a new family of mineral polymer binders in 1978. Geopolymers are mineral aluminosilicate compounds composed of silica (SiO_2) and alumina-rich raw materials (Al_2O_3) plus an alkaline activating solution [5]. Compared to Portland concrete (PC), geopolymer concrete (GPC) has better chemical and mechanical properties than conventional concrete, such as higher mechanical strength [6], higher durability [7], higher resistance to high temperatures and fire [8], Lower permeability, much higher resistance to solvents and acids [9] and less creep effects [10]. Raw materials for the synthesis of geopolymers can be natural materials such as zeolite, synthetic materials such as metakaolin, or

industrial waste materials such as fly ash and granular blast furnace slag (GGBFS) [11]. The use of industrial by-products in GPC not only helps to minimize pollution, but also reduces the storage cost of these materials [12]. Alkaline activating solution, which is usually a combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate (Na_2SiO_3) or potassium silicate (K_2SiO_3), plays an important role in the geopolymerization process. [13, 14]. However, the most commonly used alkaline solution is a mixture of NaOH and Na_2SiO_3 [15]. Xu and co-workers [16] used NaOH and KOH instead of silicate solution and discovered that KOH has a higher compressive strength. Palomo and co-workers [17] found that the combination of NaOH and Na_2SiO_3 solutions provides better compressive strength than the use of KOH and K_2SiO_3. Sharma and co-workers [18] and Parveen and co-workers [19] reported that increasing the NaOH concentration up to 16 M increased the compressive strength, but no significant change was observed when the concentration was increased to 18 M. Patel and co-workers [20] discovered that raising the NaOH concentration up to 12 M increased compressive strength, but that increasing it beyond that decreased compressive strength. Petrus et al [21] investigated the effect of the Na_2SiO_3/NaOH weight ratio on the compressive strength of silicate and bentonite-based GPC in another investigation. They came to the conclusion that raising the weight ratio of Na_2SiO_3/NaOH from 1 to 1.5 will increase the compressive strength of GPC. Sunny et al [22] showed that increasing the weight ratio of Na_2SiO_3/NaOH to 2.5 leads to an increase in compressive strength for fly ash-based GPCs. Rajivala and co-workers [23] showed that in fly ash-based GPC, an active alkaline solution of KOH has higher tensile and flexural strength than NaOH at different ages. So far, only a small number of studies have been performed on the effect of alkaline solution parameters affecting the tensile and flexural strength of GPC. Wang and co-workers [24] found that increasing the NaOH concentration from 4 to 12 M increased the flexural strength of metakaolin-based GPCs. Mishra and co-workers [25] found that increasing the NaOH concentration improves the tensile strength of fly ash-based GPC. Morsy and co-workers [26] found that increasing the Na_2SiO_3/NaOH weight ratio from 0.5 to 1 and 1 to 2.5, respectively, increased and decreased flexural strength in fly ash-based GPC. Sanni and co-workers [22], on the other hand, found that increasing the Na_2SiO_3/NaOH weight ratio to 2.5 increased tensile and flexural strength.

Since the effects of alkaline activating solutions on the mechanical and chemical properties of GPC based on zeolite contradictor have been reported, this study was conducted to investigate the effects of alkaline activating solutions on compressive, flexural, tensile strength, water absorption as well as acid. The effects of concomitant use of KOH and NaOH solutions in the previous study [13] were investigated and the results showed that concomitant use of KOH and NaOH solutions and interference in the reactivity of Na^+ and K^+ reduces the mechanical strength of GPC. In this study, a new method for problem-solving (interference of chemical reaction tendencies of sodium and potassium) was investigated.

Materials and Methods

The aluminosilicate source in this study was natural zeolite (Clinoptilolite), which was prepared from the Cemnan mine in Iran. The chemical analysis of zeolite used in this research using X-ray fluorescence (XRF) and X-ray diffraction (XRD) spectroscopy is presented in Tables 1 and 2 and Figure 1. NaOH with 98% purity, KOH with 90% purity, and liquid Na_2SiO_3 with a SiO_2/Na_2O molar ratio of 2 were used to prepare the alkaline activator solution. Table 3 represents the chemical analysis of the Na_2SiO_3, NaOH, and KOH substances. Aggregates with granular sizes of 7-10 mm were used as

coarse aggregate (sand) and < 4 mm sized aggregates were used as fine aggregate. Fine and coarse aggregates were sieved according to ASTM C33 [27]. SSD specific gravity and water absorption tests were conducted on the coarse and fine aggregates using the ASTM C127 [28] and ASTM C128 [29] procedures, respectively, gathered in Table 4. The fineness modulus (using ASTM C136 [30]) and sand equivalent (using ASTM D2419 [31]) values of the fine aggregates were measured equal to 3.01 and 73, respectively. To reduce water content and improve the workability of concrete, polycarboxylate-based Super Plasticizer (SP) was incorporated.

Table 1: The XRF analysis of zeolite used.

Chemical substance	SiO_2	Al_2O_3	CaO	Fe_2O_3	Na_2O	K_2O	MgO	P_2O_5	LOI
Weight %	67.7	10.4	1.6	1.5	2.2	3.2	0.99	0.13	18.86

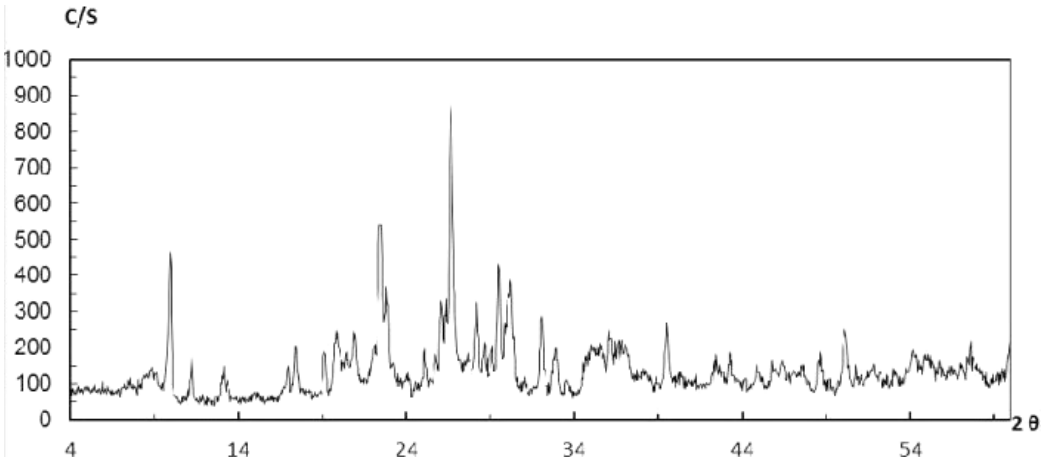

Figure 1: XRD simulated spectrum of Semnan zeolite.

Table 2: Results obtained in determining the crystal system of Semnan zeolite.

H	K	L	DOBS	DCAL	DOBS-DCAL	2TH.OBS	2TH.CAL	DIF.2TH.
2	0	-1	11.89650	11.89865	-0.00215	7.425	7.424	0.001
1	0	-2	8.96336	8.96128	0.00208	9.860	9.862	-0.002
3	0	-1	7.89028	7.90227	-0.01199	11.205	11.188	0.017
2	0	1	6.76570	6.75849	0.00722	13.075	13.089	-0.014
3	0	0	6.63189	6.62169	0.01020	13.340	13.361	-0.021
3	0	-3	5.93098	5.93605	-0.00508	14.925	14.912	0.013
0	1	0	5.59213	5.58688	0.00525	15.835	15.850	-0.015
4	0	-3	5.34409	5.34745	-0.00336	16.575	16.565	0.010
0	1	1	5.24513	5.24422	0.00091	16.890	16.893	-0.003
3	0	1	5.11001	5.11344	-0.00344	17.340	17.328	0.012
2	1	-1	5.05362	5.05923	-0.00561	17.535	17.515	0.020
5	0	-3	4.63931	4.63147	0.00785	19.115	19.148	-0.033
5	0	-1	4.51078	4.51409	-0.00331	19.665	19.650	0.015

Table 3: Chemical analysis of NaOH, KOH, and Na_2SiO_3 solutions.

NaOH			**KOH**			**Na_2SiO_3**		
Chemical substance	**Result**	**Unit**	**Chemical substance**	**Result**	**Unit**	**Chemical substance**	**Result**	**Unit**
NaOH	98	%	KOH	90.7	%	SiO_2	30	%
Na_2CO_3	1	%	K_2CO_3	0.3	%	Na_2O	14.5	%
NaCl	200	ppm	KCl	0.006	%	Water	55.5	%
Fe	6	ppm	Fe	2	ppm	n/a	n/a	n/a
SiO_2	15.7	ppm	NaOH	1.2	%	n/a	n/a	n/a

Table 4: Specific gravity and water absorption of aggregates.

Material	SSD Specific gravity (gr/cm^3)	Water absorption (%)
Coarse aggregates	2.62	1.3
Fine aggregates	2.59	3.2

Mix Designs

To conduct the first part of the study, focusing on investigating the influence of different alkaline solutions on the compressive, tensile, and flexural strengths of GPC, 5 alkaline solutions were prepared, as given in Table 5. The concentration of all the NaOH and KOH solutions was 12 M and the weight ratios of Na_2SiO_3/NaOH and Na_2SiO_3/KOH and Na_2SiO_3/KOH+NaOH were set to 1.5. Furthermore, the weight ratio of the alkaline solution/zeolite and fine/coarse aggregate ratio in preparing the first series of specimens were 0.9 and 1, respectively. Table 6 represents the mixed design of specimens for the first part of the study.

Table 5: Composition of the alkaline solutions.

Alkaline solution ID	NaOH 12M (%)	KOH 12M (%)	Addition Time delay (min)
N	100	0	-
K	0	100	-
T-K50N50	50	50	0
3-K50N50	50	50	3
6-K50N50	50	50	6

Table 6: Mix design of specimens (kg/m^3).

Mix Design ID	Zeolite	NaOH 12M	KOH 12M	Na_2SiO_3	Coarse Aggregate	Fine Aggregate	Extra Water	SP
N	400	144	0	216	850	850	10	**8**
K	400	0	144	216	850	850	10	**8**
T-K50N50	400	72	72	216	850	850	10	**8**
3-K50N50	400	72	72	216	850	850	10	**8**
6-K50N50	400	72	72	216	850	850	10	**8**

Figure 2: Samples made of geopolymer concrete based on zeolite.

Initially, NaOH and KOH solutions with a concentration of 12 M were prepared. Afterward, to prepare the mix designs N, K, and T-K50N50, these solutions were added to the Na_2SiO_3 solution 24 h prior to conducting the experiments. The “T” prefix indicates the simultaneous addition of KOH and NaOH solutions into the mixing process. To prepare the GPC specimens, dry components

including zeolite, and coarse and fine aggregates were mixed for 3 min. Then the alkaline activator solution including NaOH (mix design N) or KOH (mix design K) or NaOH+KOH (mix design T-K50N50), Na_2SiO_3, and SP was added to the dry mix and mixed for a further 10 min. For the 3-K50N50 and 6-K50N50 mixtures, The KOH and Na_2SiO_3 solutions with SP and dry components were mixed. Then, NaOH solution was added to the mixtures after 3 and 6 min of mixing; this was done to evaluate the effect of time delay in adding the NaOH solution on the compressive strength of GPC. The "3" and "6" prefixes in the mix design IDs indicate 3- and 6-minute delays in the NaOH addition to the mixing process, respectively. Similar to other mixtures, the total duration of mixing time for the 3-K50N50 and 6-K50N50 mixtures was 10 min.

Testing

In the preparation process of the specimens, after completion of material mixing, the GPC specimens were molded. Each GPC mix was batched to produce 3 cube specimens (100 x 100 x 100 mm^3) for compressive testing. Based on previous research studies [13], the prepared GPCs were dry-cured at 90˚C for 24 h and then allowed to sit at ambient temperature. Thereafter, compressive tests on 7- and 28-day specimens were conducted according to BS1881:Part116 [31]. For tensile strength tests, 3 cylindrical specimens (300 x 150 mm^2) were produced for each mix design and tested at 7- and 28-days according to the indirect tensile strength testing method of ASTM C496 [33]. As for flexural tests, 3 beam specimens (500 x 100 x 100 mm^3) were considered for each mix design and tested according to ASTM C293 [34] 3-point bending test protocol. The water absorption capacity of the GPC specimens was studied following the ASTM C642 [35] procedures. For this purpose, 3 compressive cube specimens were considered for N, T-K50N50, 3-K50N50, and K mix designs. For water absorption capacity, the 28-day specimens were initially placed in a 105 ˚C oven to reach a stable dry weight and then weighted (m_0). The specimens were then placed in a water tank for 3 days. They were then taken out and after drying the surface water, were weighted again (m). The 3-day water absorption capacity (W) is calculated by Eq. (1):

$$W = \frac{m-mo}{mo} \times 100 \quad (1)$$

Also, to test the chemical resistance of GPC specimens, the 28-day specimens of N, T-K50N50, 3-K50N50, and K mix designs were placed in a solution of water and sulfuric acid at pH equal to 1 for 28 days. Then, a weight-loss test was taken from specimens.

Results and Discussion

The 7- and 28-day compressive, tensile and flexural strengths of specimens are represented in Table 7 (along with corresponding coefficients of variation) and gathered ted in Figures 3-5, respectively. As it can be seen, the highest initial (7-day) and lateral (28-day) compressive strengths, equal to 20.8 and 23.1 MPa, respectively, were recorded for the mix 3-K50N50, in which the NaOH solution was added to the mixture after 3 min of adding KOH and Na2SiO3 to the dry components. The lowest 7- and 28-day compressive strengths belonged to the mix T-K50N50, showing 11.8 and 13.2 MPa, respectively. The obtained results of experiments indicate that the strength gaining of the mix N after 7 days of curing was better than that of all other mixes (91%). For the mix K, 74% of the 28-day compressive strength was obtained after 7 days of curing. On the other hand, the rate of strength gaining for the mix K from 7 days to 28 days was the most significant one amongst all, i.e. around 31% growth.

Table 7: Compressive, tensile, and flexural strength and corresponding coefficients of variation values.

Mix ID	Compressive strength (MPa)		Tensile strength (MPa)		Flexural strength (MPa)	
	7-day	28-day	7-day	28-day	7-day	28-day
N	16.9±0.5	18.4±0.8	1.05±0.1	1.2±0.2	1.96±0.3	2.19±0.4
T-K50N50	11.8±0.2	13.2±0.4	0.75±0.1	0.84±0.1	1.34±0.2	1.54±0.2
3-K50N50	20.8±0.6	23.1±1	1.33±0.3	1.54±0.2	2.38±0.4	2.67±0.3
6-K50N50	18.4±0.7	22.1±0.4	1.19±0.2	1.47±0.1	2.17±0.3	2.52±0.2
K	15.6±0.4	20.3±0.8	0.98±0.3	1.3±0.2	1.68±0.2	2.31±0.2

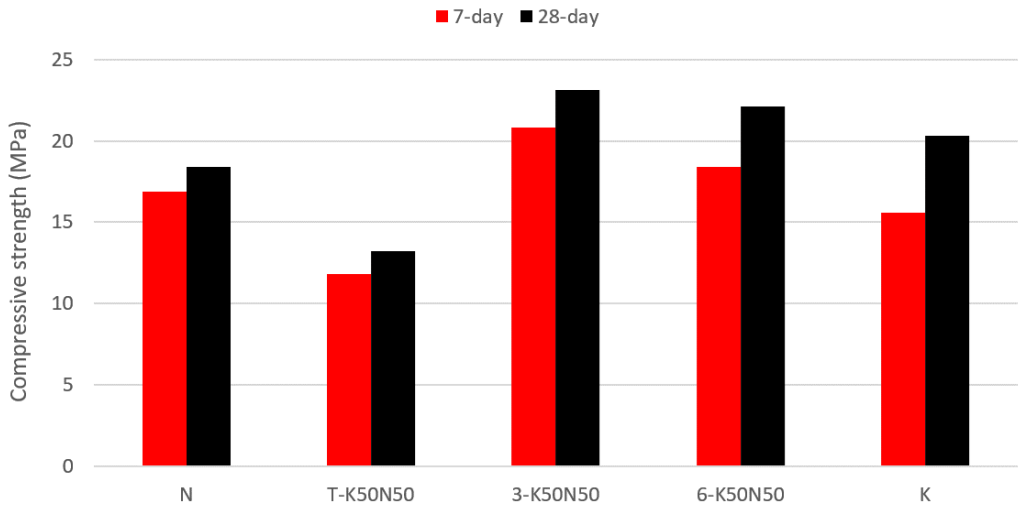

Figure 3: 7-day and 28-day compressive strengths of GPC specimens.

The tensile and flexural test results show a similar pattern to compressive strengths. The simultaneous and equal incorporation of KOH and NaOH solutions (mix T-K50N50) displayed the lowest values of tensile strength, i.e. approximately 29 and 37% lower than the N and K single solution mix designs, respectively. On the other hand, mix 3-K50N50 showed the highest tensile strength (approximately 28, 16, and 84% higher than the N, K, and T-K50N50 mix designs), indicating the significant beneficial effect of a 3-minute time delay in KOH addition to the mix. The same general aforementioned trends are followed in the flexural strengths too

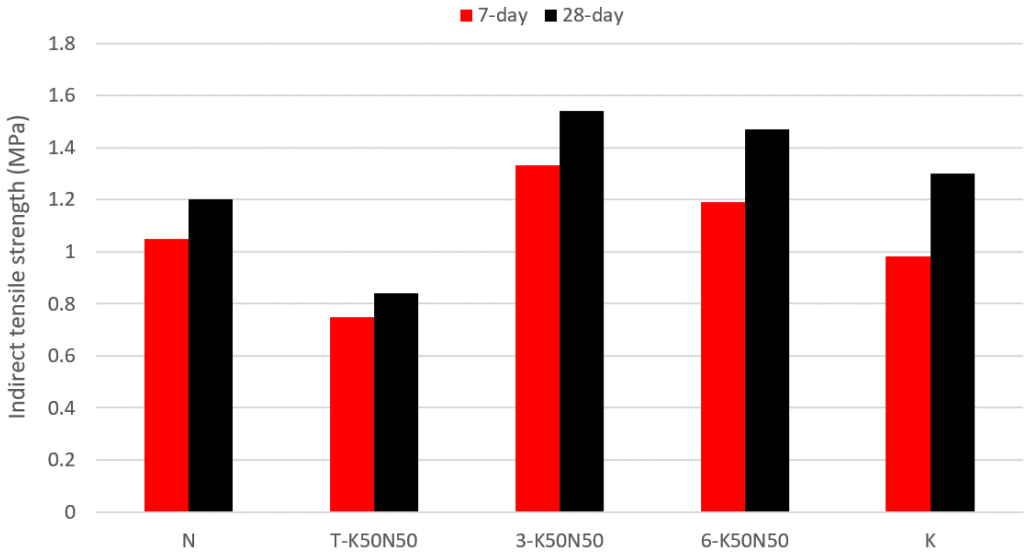

Figure 4: 7-day and 28-day tensile strengths GPC specimens.

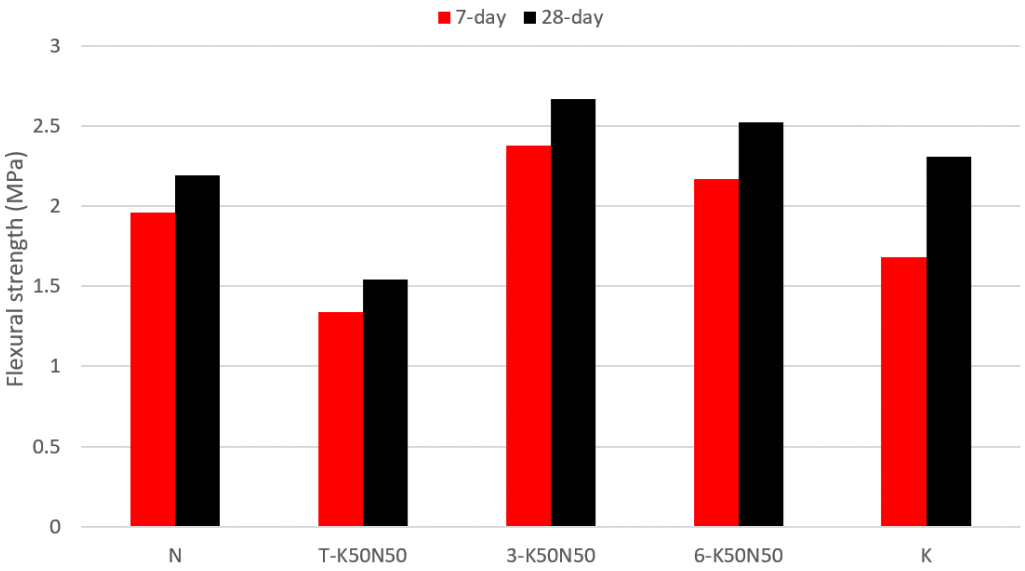

Figure 5: 7-day and 28-day flexural strengths GPC specimens.

Figures 6 and 7 represent the influence of time delay in the addition of NaOH to the fresh mixes, i.e., T-K50N50, 3-K50N50, and 6-K50N50, on the compressive, tensile, and flexural strengths of corresponding GPCs. As it is evident, the addition of NaOH by intervals of 3 and 6 min resulted in higher 7- and 28-day compressive, tensile, and flexural strengths than the simultaneous addition of NaOH, KOH, and Na_2SiO_3 to the dry components of the mix designs. The highest initial and lateral compressive, tensile and flexural strengths were achieved by the addition of NaOH to the mix, 3 minutes after adding KOH and Na_2SiO_3, for which 77% and 6% for compressive, 84% and 5% for tensile, and 73% and 6% for flexural strength increases were observed, respectively. By increasing the delay time to 6 min, both 7- and 28-day compressive, tensile and flexural strengths showed a decreasing trend; however, they still displayed higher values than the results for the simultaneous addition of NaOH and KOH. Thus, it can be concluded that in the case of using a combination of NaOH and KOH alkaline solutions, the delay time of 3 min, which is equal to 1/3 the total mixing time, is the optimum interval for adding NaOH to the mix, leading to the highest initial and lateral compressive strengths for the zeolite-based GPC.

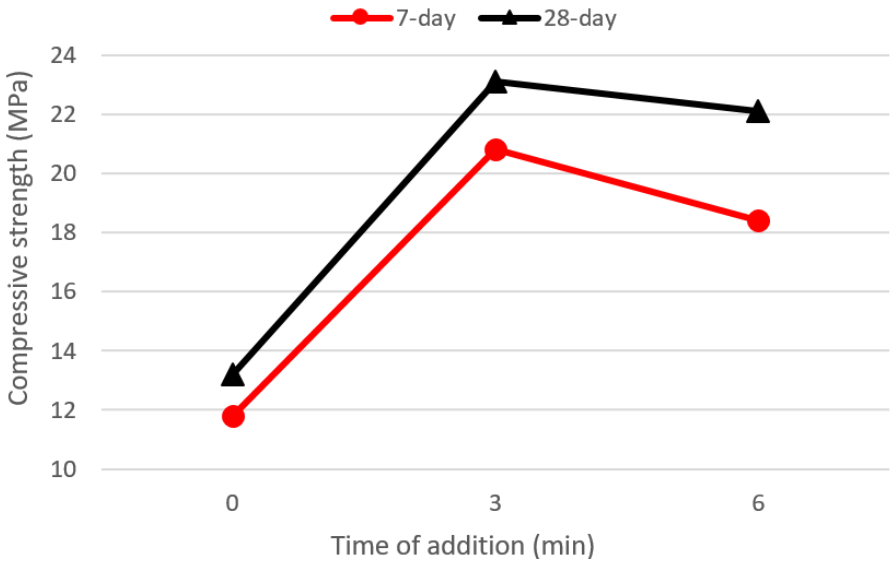

Figure 6: Impact of time of addition of NaOH solution on the compressive strengths of T-K50N50, 3-K50N50, and 6-K50N50 mixes.

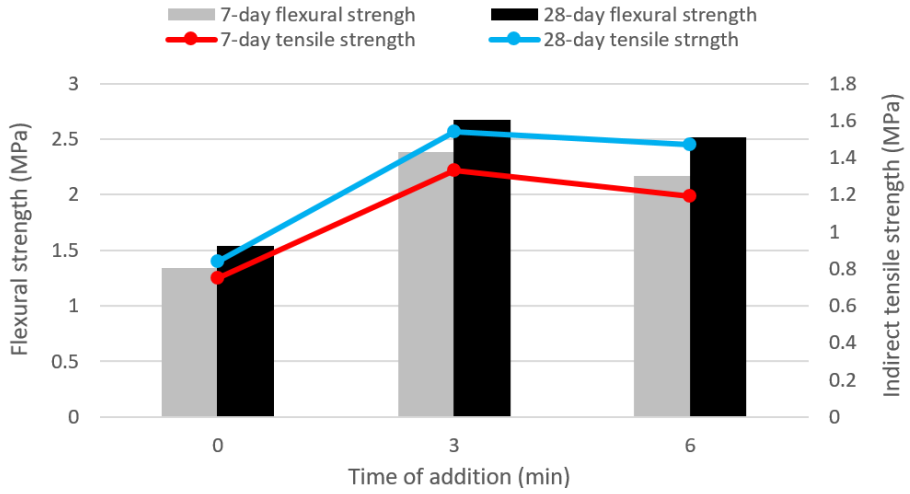

Figure 7: Impact of time of addition of NaOH solution on the flexural and tensile strengths of T-K50N50, 3-K50N50, and 6-K50N50 mixes.

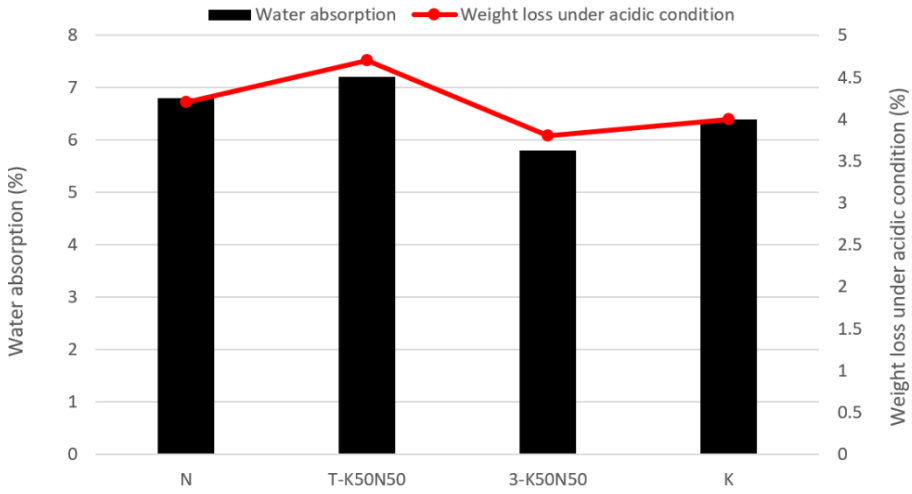

Figure 8: Water absorption and weight loss under acidic conditions of N, T-K50N50, 3-K50N50, and K mixes.

To explain the observed trends, the performance mechanism of the alkaline solutions should be considered. By using KOH, more geopolymers are produced, leading to a stronger and more compact microstructure, which will result in low 7-day compressive, tensile, and flexural strengths, slow hardening, and high 28-day compressive tensile and flexural strengths in comparison with NaOH. On the other hand, at the same concentration, NaOH is capable of dissolving more inorganic components than KOH, which leads to a faster reaction rate for Na^+ than for K^+. Due to the higher reaction rate of Na^+, higher initial compressive strength and more rapid hardening would be observed by using NaOH. However, simultaneous inclusion of NaOH and KOH would reduce the compressive, tensile, and flexural strengths of GPC noticeably. This could be attributed to the different performances of NaOH and KOH during the geopolymerization process. The high reactivity of Na+ could not be balanced with the tendency of K+ towards condensation reaction. However, as it can be seen for the mixes 3-K50N50 and 6-K50N50, by the addition of NaOH with 3- and 6-min intervals, both K^+ and Na^+ would have enough time to form bonds inappropriate directions, resulting in the formation of larger amounts of geopolymer gel and a denser geopolymer cement matrix.

Figure 7 illustrates the results of water absorption and weight loss under acidic condition tests of GPC specimens. The water absorption capacity of N, T-K50N50, 3-K50N50, and K mix designs were measured at approximately 6.8, 7.2, 5.8, and 6.4%, respectively. As well as, weight loss under acidic

conditions N, T-K50N50, 3-K50N50, and K mix designs were measured at approximately 4.2, 4.7, 3.8, and 4%, respectively. By observing the results of Figure 8, water absorption capacity and weight loss in acidic conditions of the 3-K50N50 mix design were less compared to other specimens, mainly due to the higher density of the geopolymeric matrix structure in this mix design.

Conclusion

In this comprehensive experimental study, a novel type of alkaline activator for GPC Based on zeolite, was studied. The following conclusions can be drawn based on the results of the current experimental studies. The combination of NAOH alkaline solution with Na_2SiO_3 has a 7-day higher compressive, tensile, and flexural strength than the use of K OH with Na_2SiO_3 solution, which results in less activation energy of sodium than potassium to dissolve aluminum and silica ions. Incorporation of the KOH alkaline solution with Na_2SiO_3 would result in higher 28-day compressive, tensile, and flexural strengths than using NaOH with Na_2SiO_3 solution, which is due to the higher activation energy of potassium than sodium and the formation of denser networks. Simultaneous application of NaOH and KOH with Na_2SiO_3, as the alkaline solution, decreased compressive, tensile, and flexural strengths of zeolite-based GPC due to the interference in the Na^+ and K^+ reactivity. Nevertheless, time delay in the addition of NaOH solution to the fresh mix resulted in higher compressive strengths. In this study, the addition of NaOH to the mix after 3 min of mixing KOH and Na_2SiO_3 with dry components (1/3 of the total mixing duration) resulted in the highest compressive, tensile, and flexural strengths among other cases. The addition of NaOH to the mix after 3 min of mixing KOH and Na_2SiO_3 with dry components (1/3 of the total mixing duration) resulted in the lowest water absorption and weight loss under acidic conditions. It is proposed that the new alkaline solution composition approach developed in this study be used to get higher mechanical and chemical properties of geopolymer concretes based on different sources of alumina silicate.

Disclosure Statement

The author(s) did not report any potential conflict of interest.

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