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

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ABSTRACT

In recent years, geopolymers, as a new class of green cement binders, have been considered as an environmental-friendly alternative to Ordinary Portland Cement (OPC), which can potentially reduce negative environmental impacts of OPC, including carbon footprint and energy consumption. In this experimental research, effects of different alkaline activator solutions on the compressive, indirect tensile, and flexural strengths, water absorption, and resistance to an acidic condition of metakaolin-based Geopolymer Concrete (GPC) were investigated. Furthermore, a novel type of alkaline activator for GPC was introduced. In this regard, GPC specimens based on metakaolin were manufactured and cured at 90 ˚C. The results showed that the addition of NaOH to the mix after 3 min of mixing KOH and Na2SiO3 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, Metakaolin, Alkaline activator, Compressive strength, Resistance to acidic condition

1. INTRODUCTION

Ordinary Portland Cement (OPC) as the main constituent of conventional concrete is the most widely used cementitious material in the construction industry [1]. OPC production has major environmental disadvantages, such as high energy and natural resources consumption, as well as carbon dioxide (CO2) emissions [2]. As for the production of 1 ton OPC, approximately 0.73-0.99 ton CO2 would be released [3]. On the other hand, climate change due to global warming is one of the most severe environmental issues that has been receiving significant attention in recent years. Greenhouse gas emissions, CO2 in particular, is the main contributing factor to global warming [2]. The production process of OPC accounted for 7 to 10% of global CO2 emissions [4]. In recent years, geopolymers have been introduced as environmentally friendly cementitious materials capable of reducing the negative environmental impacts associated with OPC. In 1978, Davidovits introduced geopolymers as a new class of binders belonging to inorganic polymers. Geopolymers are inorganic alumino-silicate materials produced from raw materials, rich in silica (SiO2) and alumina (Al2O3), in combination with an alkaline activator solution [5]. Geo-Polymer Concrete (GPC) possesses better chemical and mechanical properties compared to Portland Concrete (PC), such as higher mechanical strengths [6], enhanced durability [7], higher resistance to elevated temperatures and fire [8], lower permeability, improved resistance to solvents and acids [9] and lower creep effects [10]. Depending on the required properties, cost, and availability of materials, raw materials for the production of geopolymers could be natural materials such as zeolite, synthetic materials such as metakaolin, or waste industrial materials such as fly ash and Granulated Ground Blast Furnace Slag (GGBFS) [11]. Usage of industrial by-products in GPC not only helps to reduce environmental pollution but also alleviates the storage costs of these materials [12].The alkaline activator solution is another

pillar of the geopolymerization process playing an important role in the formation of crystalline structures of Si and Al, which is typically a combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate (Na_2SiO_3) or potassium silicate (K_2SiO_3) [13]. However, the most widely used alkaline solution is a combination of NaOH and Na_2SiO_3 [14]. Xu et al. [15] also used NaOH and KOH without using silicate solution and found that KOH would result in better compressive strength. Most of these researchers pointed out the crucial role of the alkaline activator solution in the polymerization reaction and revealed that the addition of a silicate solution to the NaOH and/or KOH solution would promote the polymerization rate. Palomo et al [16] reported that using a combination of NaOH and Na_2SiO_3 solutions would result in higher compressive strength than using KOH and K_2SiO_3.Regarding the influential factors on the compressive strength of fly ash-based geopolymer, Sharma et al. [17] and Parveen et al. [18] reported that by increasing the NaOH concentration up to 16 M, the compressive strength increased, but with a further increase to 18 M, no significant change was observed.Contrary, Patel et al. [19] found that by increasing the NaOH concentration up to 12 M, the compressive strength would increase, and beyond that, the compressive strength would decrease. In another study, Petrus et al. [20] studied the impact of the Na_2SiO_3/NaOH weight ratio on the compressive strength of silicate and bentonite-based GPC. They concluded that by increasing the weight ratio of Na_2SiO_3/NaOH from 1 to 1.5, the compressive strength of GPC would increase. The highest compressive strength was recorded for the weight ratio of 1.5 and by raising the ratio from 1.5 to 2.5, the compressive strength decreased significantly. Sanni et al. [21] showed that increasing the weight ratio of Na_2SiO_3/NaOH up to 2.5 would result in a compressive strength increase for fly-ash-based GPC.At a lower extent than compressive strength, tensile and flexural strengths of various GPC configurations and corresponding parameters have been studied. Rajiwala et al. [22] showed that a KOH active alkali solution would yield higher tensile and flexural strengths than NaOH in fly ash-based GPC at different ages. Limited research has been conducted so far regarding the effects of influential alkali solution parameters on tensile and flexural strengths of GPC. Wang et al. [23] showed that for Metakaolin-based GPC, an increase of NaOH concentration from 4 to 12 M would increase flexural strength. Mishra et al. [24] concluded that NaOH concentration increase will improve tensile strength for fly ash-based GPC. Regarding fly ash-based GPC, Morsy et al. [25] concluded that the increase of Na_2SiO_3/NaOH weight ratio from 0.5 to 1 and 1 to 2.5, would increase and decrease flexural strength, respectively. Contrarily, Sanni et al. [21] showed tensile and flexural strength increase of Na_2SiO_3/NaOH weight ratio up to 2.5.Since the reported results regarding the impact of the alkaline solution on the mechanical strengths of GPC are not uniform, this study aims at investigating the influences of alkaline activator solutions on the compressive, tensile, and flexural strengths and also, water absorption and acid resistance of metakaolin-based GPC to provide insight into its developing practical applications. In previous research [13], effects of simultaneous usage of KOH and NaOH solutions were investigated, and results showed that simultaneous usage of KOH and NaOH solutions decreased 3-, 7- and 28-day compressive strengths of GPC due to the interference in Na^+ and K^+ reactivity. In this paper, a novel method to solve the mentioned problem was studied.

2. MATERIALS AND METHODS

2.1. MATERIALS

The X-Ray Fluorescence (XRF) chemical analysis of the metakaolin used in this study is given in Table 1. NaOH with 98% purity, KOH with 90% purity, and liquid Na2SiO3 with SiO2/Na2O molar ratio of 2 were used to prepare the alkaline activator solution. Table 2 represents the chemical analysis of the Na2SiO3, 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 [26]. SSD specific gravity and water absorption tests were conducted on the coarse and fine aggregates using the ASTM C127 [27] and ASTM C128 [28] procedures, respectively, gathered in Table 3. The fineness modulus (using ASTM C136 [29]) and sand equivalent (using ASTM D2419 [30]) 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. Physical and chemical properties of Metakaolin used

Content	Result	Unit
SiO_2	54	**%**
Al_2O_3	31.7	**%**
TiO_2	1.41	**%**
Fe_2O_3	4.89	**%**
ZrO_2	0.1	**%**
K_2O	4.05	**%**
Na_2O	2.32	**%**
MnO	0.11	**%**
L.O.I	1.41	**%**
SiO_2/ Al_2O_3	1.71	-
Specific Gravity	2.67	**g/cm^3**
Fineness	21400	**cm^2/g**

Table 2. Chemical analysis of NaOH, KOH, and Na_2SiO_3 solutions

NaOH			KOH			Na_2SiO_3		
Chemical sub stance	Result	Unit	Chemical sub stance	Result	Unit	Chemical sub stance	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			
SiO_2	15.7	ppm	NaOH	1.2	%			

Table 3. 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**

2.2. EXPERIMENTAL PROGRAM

2.2.1. 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 4. 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 was set to 1.5. Furthermore, the weight ratio of the alkaline solution/metakaolin and fine/coarse aggregate ratio in preparing the first series of specimens were 0.9 and 1, respectively. Table 5 represents the mixed design of specimens for the first part of the study

Table 4. 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 5. Mix design of specimens (kg/m3)

Mix design ID	metakaolin	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**

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 Na2SiO3 solution 24 hours before 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 metakaolin, 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), Na2SiO3, and SP were added to the dry mix and mixed for a further 10 min. For the 3-K50N50 and 6-K50N50 mixtures, The KOH and Na2SiO3 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 delay in the NaOH addition to the mixing process, respectively. Like other mixtures, the total duration of mixing time for the 3-K50N50 and 6-K50N50 mixtures was 10 min.

2.2.2. 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 (100x100 x100mm) for compressive testing. Based on previous research studies [13], the prepared GPCs were dry-cured at 90℃ for 24 hours and then allowed to set at ambient temperature. After that, compressive tests on 7- and 28-day specimens were

conducted according to BS1881: Part116 [31]. For tensile strength tests, 3 cylindrical specimens (300x150mm) were produced for each mix design and tested at 7- and 28 days according to the indirect tensile strength testing method of ASTM C496 [32]. As for flexural tests, 3 beam specimens (500x100x100mm) were considered for each mix design and tested according to ASTM C293 [33] 3-point bending test protocol. The water absorption capacity of the GPC specimens was studied following the ASTM C642 [34] 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 (m0). 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):

$$W=(m-mo)/mo\times100 \quad \text{Eq. (1)}$$

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.

3. RESULTS AND DISCUSSION

The 7- and 28-day compressive, tensile and flexural strengths of specimens are represented in Table 6 (along with corresponding coefficients of variation) and gathered ted in Figs. 1- 3, respectively. As it can be seen, the highest initial (7-day) and lateral (28-day) compressive strengths, equal to 84.3 and 93.7 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 Na_2SiO_3 to the dry components. The lowest 7- and 28-day compressive strengths belonged to the mix T-K50N50, showing 40.4 and 49.7 MPa, respectively. The obtained results indicate that the strength gaining of the mix N after 7 days of curing was better than that of all other mixes (92%). 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 6. Compressive, tensile and flexural strength and corresponding coefficients of variation values

Mix ID	Compressive strength (MPa)		Tensile strength (MPa)		Flexural strength (MPa)	
	7-days	28-days	7-days	28-days	7-days	28-days
N	24.2±0.5	26.4±0.8	1.5±0.1	1.7±0.2	2.8±0.3	3.1±0.4
T-K50N50	16.9±0.2	18.9±0.4	1.06±0.1	1.2±0.1	1.9±0.2	2.2±0.2
3-K50N50	29.8±0.6	33.5±1	1.9±0.3	2.2±0.2	3.4±0.4	3.8±0.3
6-K50N50	26.4±0.7	31.7±0.4	1.7±0.2	2.1±0.1	3.1±0.3	3.6±0.2
K	22.3±0.4	29.1±0.8	1.4±0.3	1.9±0.2	2.4±0.2	3.3±0.2

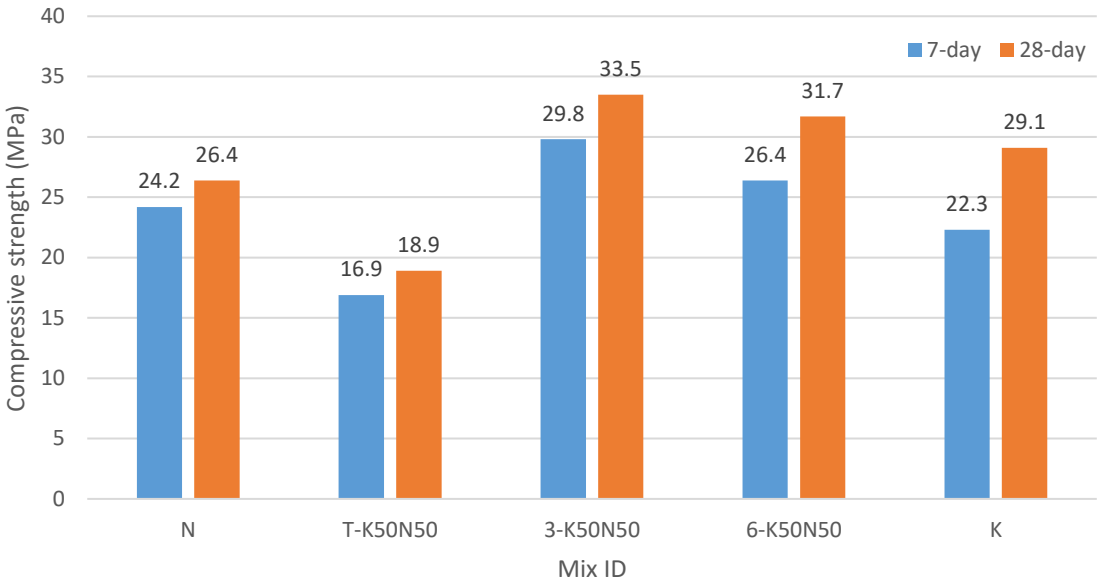

Figure 1. 7-day and 28-day compressive strengths GPC specimens

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 3-minute time delay in KOH addition to the mix. The same general aforementioned trends are followed in the flexural strengths too.

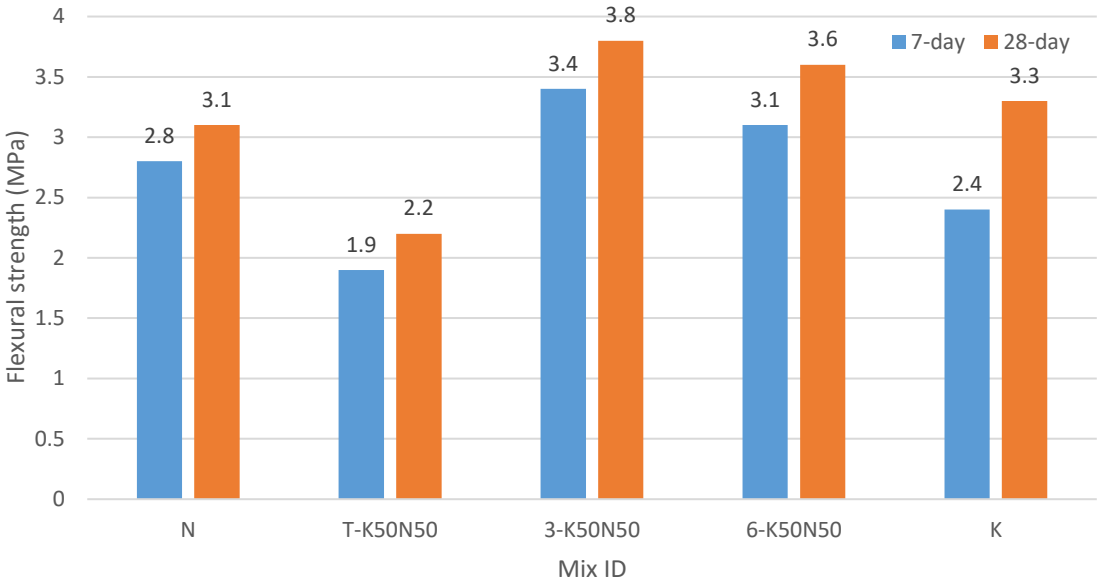

Figure 2. 7-day and 28-day flexural strengths GPC specimens

Figs 3 and 4 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 Na2SiO3 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 Na2SiO3, 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 the 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 metakaolin-based GPC.

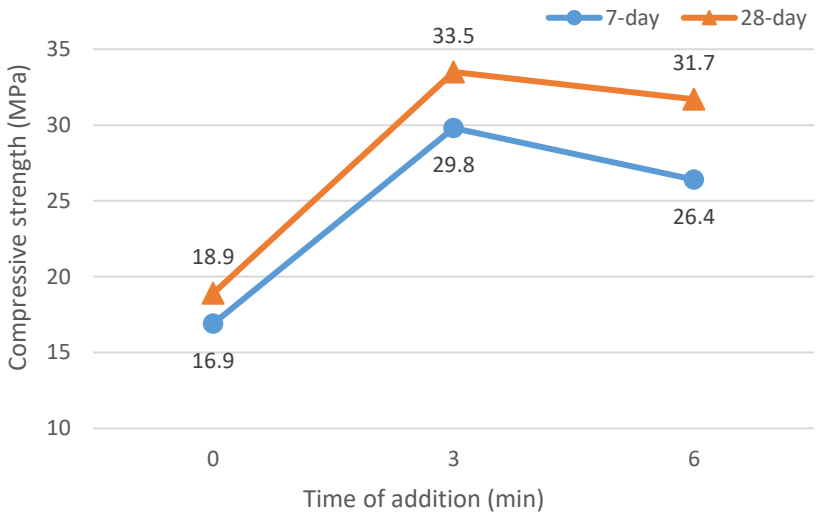

Figure 3. Impact of time of addition of NaOH solution on the compressive strengths of T-K50N50, 3-K50N50, and 6-K50N50 mixes

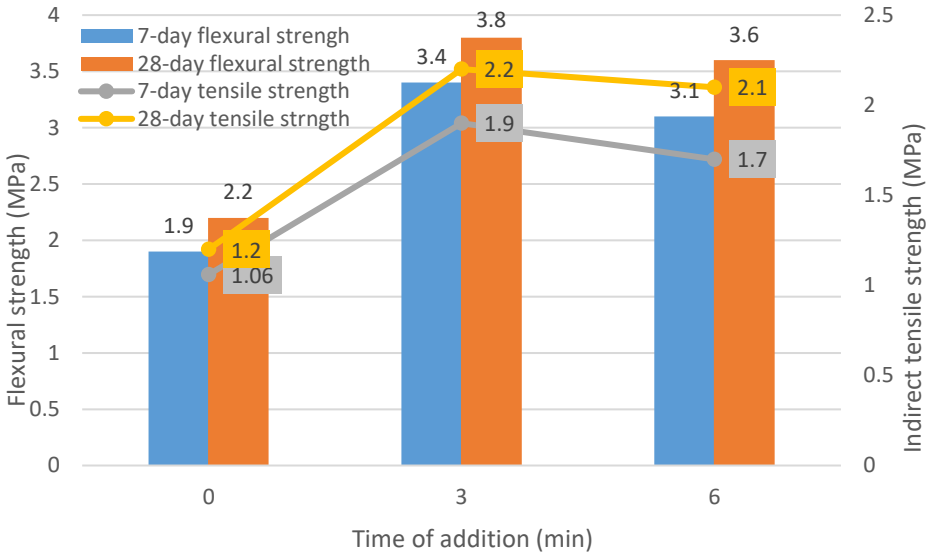

Figure 4. Impact of time of addition of NaOH solution on the flexural and tensile strengths of T-K50N50, 3-K50N50, and 6-K50N50 mixes

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 in appropriate directions, resulting in the formation of larger amounts of geopolymer gel and a denser geopolymer cement matrix. Fig 5 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 approximately 6.2, 6.6, 5.3, and 5.8%, respectively. As well as, weight loss under acidic condition N, T-K50N50, 3-K50N50, and K mix designs were measured approximately 3.8, 4.3, 3.5, and 3.6%, respectively. By observing the results of fig 5, water absorption capacity and weight loss in an acidic condition of 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.

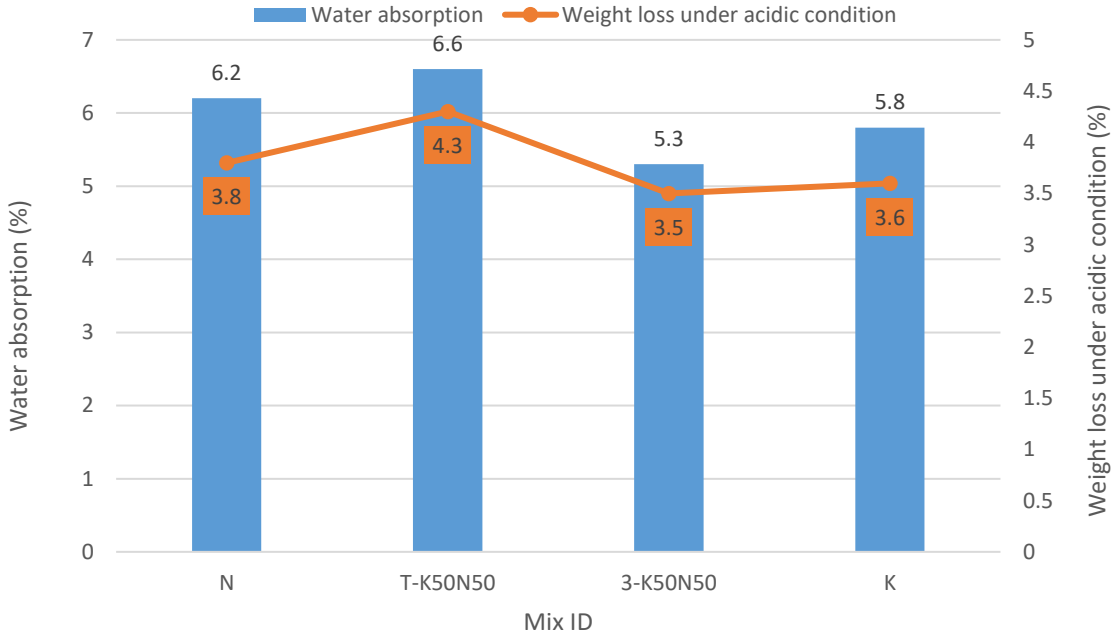

Figure 5. Water absorption and weight loss under the acidic condition of N, T-K50N50, 3-K50N50, and K mixes

4. CONCLUSION

In this comprehensive experimental study, a novel type of alkaline activator for GPC Based on metakaolin, was studied. The following conclusions can be drawn based on the results of the current experimental studies

- Incorporation of the KOH alkaline solution with Na2SiO3 would result in higher 28-day compressive, tensile, and flexural strengths than using NaOH with Na2SiO3 solutions.
- Simultaneous application of NaOH and KOH with Na2SiO3, as the alkaline solution, decreased compressive, tensile, and flexural strengths of metakaolin-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 Na2SiO3 with dry components (1/3 of the total mixing duration) resulted in the highest compressive, tensile, and flexural strengths amongst other cases.
- The addition of NaOH to the mix after 3 min of mixing KOH and Na2SiO3 with dry components (1/3 of the total mixing duration) resulted in the lowest water absorption and weight loss under acidic condition.

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AUTHORS CONTRIBUTION

This work was carried out in collaboration among all authors.

CONFLICT OF INTEREST

The author (s) declared no potential conflicts of interests with respect to the authorship and/or publication of this paper.

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