



A Novel Type of Alkaline Activator for Geopolymer Concrete Based on Class C Fly Ash

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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, energy consumption and etc. In this experimental research, effects of different alkaline activator solutions on the compressive, Tensile and flexural strengths, water absorption and resistance to acidic condition of fly-based Geopolymer Concrete (GPC) were investigated. Furthermore, a novel type of alkaline activator for GPC was introduced. In this regard, GPC specimens based on Class C fly ash were manufactured and cured in 90 °C. The obtained results showed that 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, Fly ash, 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, 2]. OPC production has major environmental disadvantages, including high energy consumption, natural resources exhaustion and carbon dioxide (CO₂) emissions [2-4] as for production of 1 ton OPC, approximately 0.73-0.99 ton CO₂ would be released [5,6]. On the other hand, climate change due to global warming is one of the most serious environmental issues that has been receiving great attention in recent years. Emission of greenhouse gases, 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 [7, 8]. In recent years, geopolymers have been introduced as environmentally friendly cementitious materials capable of reducing the negative environmental impacts associated with OPC [9]. In 1978, Davidovits introduced geopolymers as a new class of binders belonging to inorganic polymers [10]. Geopolymers are inorganic aluminosilicate materials produced from raw materials, rich in silica (SiO₂) and alumina (Al₂O₃), in combination with an alkaline activator solution [11]. Geo-Polymer Concrete (GPC) possesses better chemical and mechanical properties compared to Portland Concrete (PC), such as higher mechanical strengths [12-14], enhanced durability [15], higher resistance to elevated temperatures and fire [16-18], lower permeability, improved resistance to solvents and acids [19,20] and lower creep effects [21,22]. Depending on the required properties, cost, and availability of materials, raw materials for 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) [23]. Usage of industrial by-products in GPC not only helps to reduce environmental pollution, but also alleviates storage costs of these materials [24, 25]. 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 (Na₂SiO₃) or potassium silicate (K₂SiO₃) [26]. However, the most widely used alkaline solution is a combination of NaOH and Na₂SiO₃ [27]. Xu et al [28] 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 to the crucial role of the alkaline activator solution in the polymerization reaction and revealed that addition of a silicate solution to the NaOH and/or KOH solution would promote the polymerization rate. Palomo et al [29] reported that using a combination of NaOH and Na₂SiO₃ solutions would result in higher compressive strength than using KOH and K₂SiO₃. Regarding the influential factors on the compressive strength of fly ash-based geopolymer, Sharma et al [30] and Parveen et al [31] reported that by increasing the NaOH concentration up to 16 M, the compressive strength increased, but with further increase to 18 M, no significant change was observed. Contrary, Patel et al [32] 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 [33] studied the impact of the Na₂SiO₃/NaOH weight ratio on the compressive strength of silicate and bentonite-based GPC. They concluded that by increasing the weight ratio of Na₂SiO₃/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 [34] showed that increasing the weight ratio of Na₂SiO₃/NaOH up to 2.5



would result in 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 has been studied. Rajiwala et al [35] 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 [36] showed that for Metakaolin based GPC, increase of NaOH concentration from 4 to 12 M would increase flexural strength. Mishra et al [37] concluded that NaOH concentration increase will improve tensile strength for fly ash-based GPC. Regarding fly ash-based GPC, Morsy et al [38] concluded that increase of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ weight ratio from 0.5 to 1 and 1 to 2.5, would increase and decrease flexural strength, respectively. Contrarily, Sanni et al [34] showed tensile and flexural strength increase for $\text{Na}_2\text{SiO}_3/\text{NaOH}$ weight ratio up to 2.5. Since the reported results regarding the impact of alkaline solution on the mechanical strengths of GPC are not uniform, this study aims at investigating the influences of a novel alkaline activator solutions on the compressive, tensile and flexural strengths and also, water absorption and resistance to acid of fly ash-based GPC to provide insight into its developing practical applications. In previous research [26] 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 fly ash used in this study is illustrated in Table 1. NaOH with 98% purity, KOH with 90% purity, and liquid Na_2SiO_3 with $\text{SiO}_2/\text{Na}_2\text{O}$ molar ratio of 2 were used to prepare the alkaline activator solution. Table 2 illustrates the chemical analysis of the Na_2SiO_3 , NaOH, and KOH substances. Aggregates with granular sizes of 7-10 mm was 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 [39]. SSD specific gravity and water absorption tests were conducted on the coarse and fine aggregates using the ASTM C127 [40] and ASTM C128 [41] procedures, respectively, illustrated in Table 3. The fineness modulus (using ASTM C136 [42]) and sand equivalent (using ASTM D2419 [43]) values of the fine aggregates were measured equal to 3.01 and 73, respectively. To reduce water content and improve workability of concrete, polycarboxylate-based Super Plasticizer (SP) was incorporated.

Table 1. XRF chemical analysis of fly ash.

Chemical substance	SiO_2	Al_2O_3	CaO	Fe_2O_3	Na_2O	K_2O	MgO	MnO	P_2O_5	Cl	LOI
Weight %	70.7	21.1	1.13	3.9	0.26	1.09	0.77	0.05	0.05	0.03	0.8



Table 2. Chemical analysis of NaOH, KOH, and Na₂SiO₃ solutions.

NaOH			KOH			Na ₂ SiO ₃		
Chemical substance	Result	Unit	Chemical substance	Result	Unit	Chemical substance	Result	Unit
NaOH	98	%	KOH	90.7	%	SiO ₂	30	%
Na ₂ CO ₃	1	%	K ₂ CO ₃	0.3	%	Na ₂ O	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 illustrated in Table 4. Concentration of all the NaOH and KOH solutions was 12 M and the weight ratios of Na₂SiO₃/NaOH and Na₂SiO₃/KOH and Na₂SiO₃/KOH+NaOH was set to 1.5. Furthermore, the weight ratio of the alkaline solution/fly ash and fine/coarse aggregate ratio in preparing the first series of specimens were 0.5 and 1, respectively. Table 5 illustrates the mix 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	Fly ash	NaOH 12M	KOH 12M	Na ₂ SiO ₃	Coarse aggregate	Fine aggregate	Extra water	SP
N	400	80	0	120	850	850	10	8
K	400	0	80	120	850	850	10	8
T-K50N50	400	40	40	120	850	850	10	8
3-K50N50	400	40	40	120	850	850	10	8
6-K50N50	400	40	40	120	850	850	10	8



Initially, NaOH and KOH solutions with a concentration of 12 M were prepared. Afterwards, to prepare the mix designs N, K and T-K50N50, these solutions were added to the Na₂SiO₃ solution 24 hours 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 fly ash, 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. Similar to 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 (100x100x100mm) for compressive testing. Based on previous research studies [26], the prepared GPCs were dry cured at 90 °C for 24 hours and then allowed to set at ambient temperature. Thereafter, compressive tests on 7- and 28-day specimens were conducted in accordance with BS1881: Part116 [44]. For tensile strength tests, 3 cylindrical specimens (300x150mm) were produced for each mix design and tested at 7- and 28 days according the indirect tensile strength testing method of ASTM C496 [45]. As for flexural tests, 3 beam specimens (500x100x100mm) were considered for each mix design and tested according to ASTM C293 [46] 3-point bending test protocol. Water absorption capacity of the GPC specimens was studied following the ASTM C642 [47] 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:

$$W = \frac{m - m_0}{m_0} \times 100 \quad (1)$$

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, weight loss test was taken from specimens.

3. Results and Discussion

The 7- and 28-day compressive, tensile and flexural strengths of specimens are illustrated 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 Na₂SiO₃ 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 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 40% 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	69.2±0.9	76±1.5	3.9±0.1	4.4±0.3	6.8±0.4	7.9±0.6
T-K50N50	40.4±1	49.7±1.7	2.7±0.2	3.1±0.1	3.7±0.6	5.2±0.5
3-K50N50	84.3±1.3	93.7±1.8	4.9±0.2	5.5±0.4	8.2±0.4	9.7±0.7
6-K50N50	71.5±1.2	80.5±2	3.8±0.1	4.5±0.2	7.1±0.5	8.2±0.4
K	63.1±0.8	84.8±1.4	3.4±0.3	4.7±0.0.5	5.8±0.3	8.3±0.8

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 lowest values of tensile strength, i.e. approximately 28 and 33% lower than the N and K single solution mix designs, respectively. On the other hand, mix 3-K50N50 showed highest tensile strength (approximately 25, 20 and 75% 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.

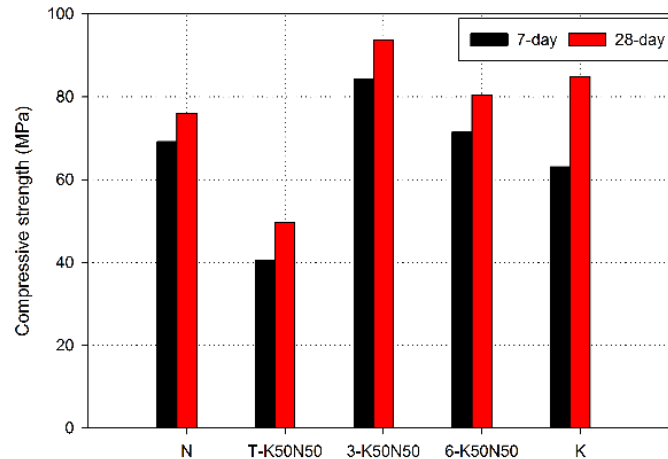


Figure 1. 7-day and 28-day compressive strengths GPC specimens.

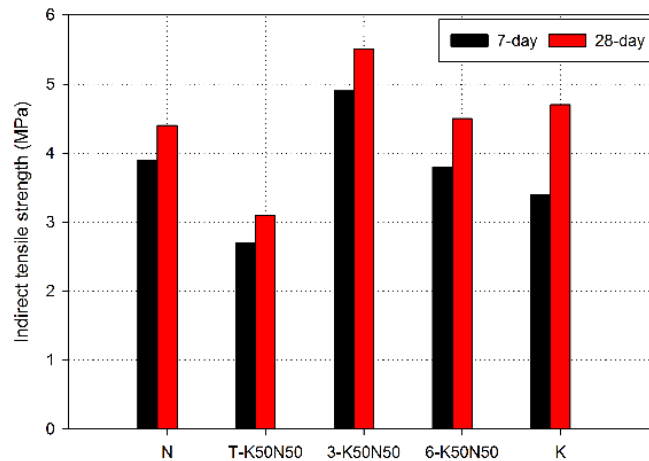


Figure 2. 7-day and 28-day tensile strengths GPC specimens.

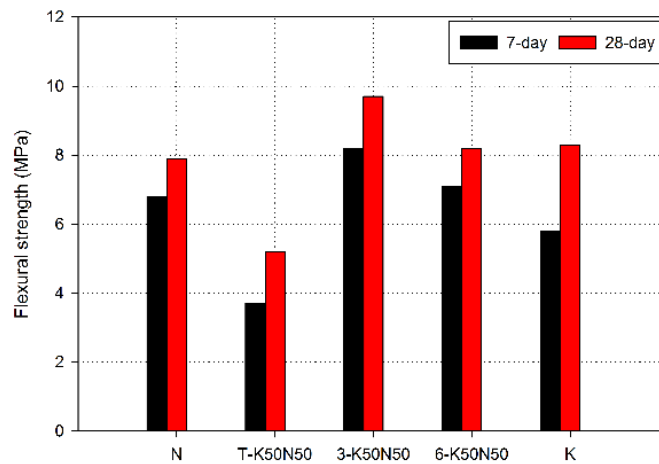


Figure 3. 7-day and 28-day flexural strengths GPC specimens.

Figs 4 and 5 illustrate 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, 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 89% and 17% for compressive, 80% and 21% for tensile and 86% and 18% 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 fly ash-based GPC.

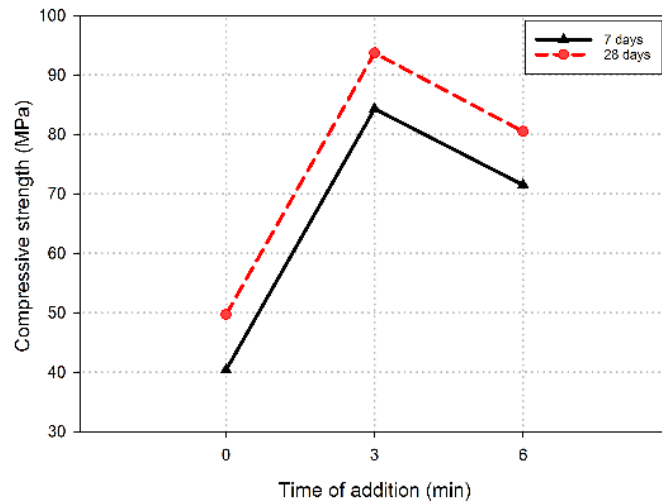


Figure 4. Impact of time of addition of NaOH solution on the compressive strengths of T-K50N50, 3-K50N50, and 6-K50N50 mixes.

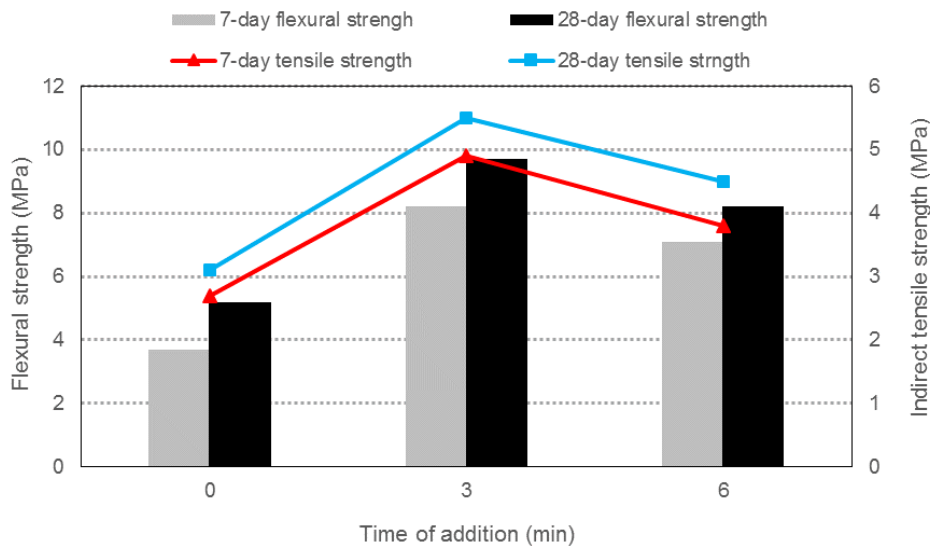


Figure 5. Impact of time of addition of NaOH solution on the flexural and tensile strengths of T-K50N50, 3-K50N50, and 6-K50N50 mixes



To explain the observed trends, the performance mechanism of the alkaline solutions should be considered [13]. By using KOH, more geopolymers are produced, leading to a stronger and more compact microstructure [48], 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 faster reaction rate for Na^+ than for K^+ [28,49]. 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 [26, 49]. 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. Figure 6 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 5.1, 5.5, 4.7 and 5%, respectively. As well as, weight loss under acidic condition N, T-K50N50, 3-K50N50 and K mix designs were measured approximately 5.1, 5.5, 4.7 and 5%, respectively. By observing the results of fig 6, water absorption capacity and weight loss in acidic condition of 3-K50N50 mix design were less compared to other specimens, mainly due to higher density of the geopolymeric matrix structure in this mix design.

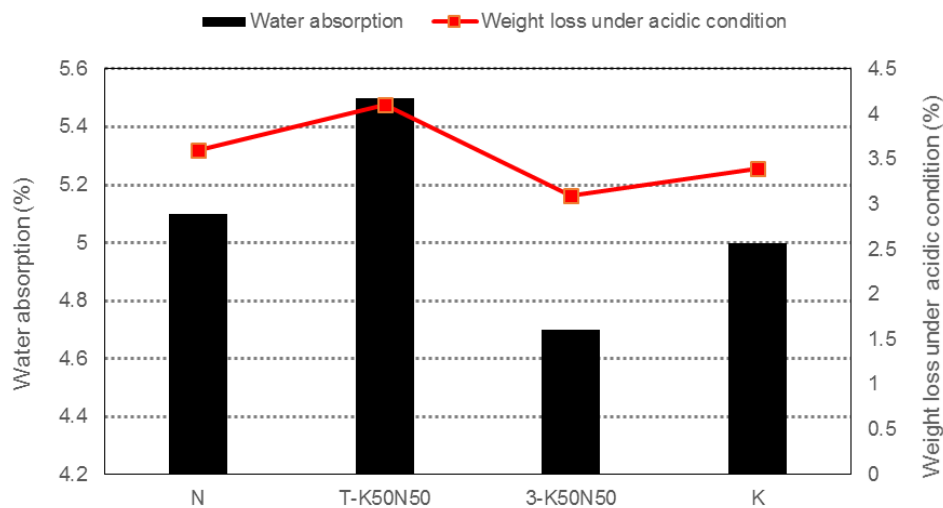


Figure 6. Water absorption and weight loss under acidic condition of N, T-K50N50, 3-K50N50 and K mixes.



4. Conclusion

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

1- 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 solutions.

2- Simultaneous application of NaOH and KOH with Na_2SiO_3 , as the alkaline solution, decreased compressive, tensile and flexural strengths of fly ash-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, 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 amongst other cases.

3- 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 condition.

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