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Performance of Geopolymer Cconcrete Against Acid Attacks Using Taguchi Method

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ABSTRACT

The acid and sulfate resistance of geopolymer concrete generated from treated fly ash is the primary subject of this investigation. The major goal is to find out if geopolymer concrete can be used for structural requests in harsh climates. The study includes the replacement of 90% of regular Portland cement with fly ash. The effect of numerous issues on the performance of geopolymer concrete is studied, including molarities of alkali activator, sodium silicate/sodium hydroxide ratio, alkali activator/binder ratio, and binder content. All specimens are treated at 65$^{o C}$. After 28 days, specimens from each mix are put into a 10% concentration sulfuric acid bath for 8 weeks. The performance of the mixes was investigated using some mechanical properties. The outcomes show that geopolymer concrete has exceptional resistance to acid and sulfate attacks at all molarities tested compared to traditional concrete, with the 8-molarity mix suffering the least strength loss and the 12-molarity mix suffering the most. Overall, the results show that it is possible to make geopolymer concrete from treated fly ash that is strong and long-lasting enough for structural purposes, even in hostile chemical environments.

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1. Introduction

As cement manufacturing is one of the largest causes of CO2 emissions into the atmosphere, studies have sought to create less harmful concrete to the environment by replacing portions of cement with other elements. Concrete is the second-most plentiful resource on earth after water. Historically, Portland cement has been used to bind concrete. After the devastating fires in France, Davidovits (1991), presented the novel binder known as "geopolymer". It appeared useful for creating new non-flammable heat-resistant materials, so the geopolymer was the consequence of this study (Davidovits, 1991). Also, many studies have shown that using waste materials or common resources, for instance, fly ash, clays, kaolin, metakaolin, bottom ash, rice husk ash, and ground granulated blast slag, can reduce the quantity of cement that is consumed (Heah et al., 2012). This non-cementitious substance is known as a geopolymer (Shahedan et al., 2019). Because the novel concrete is environmentally friendly and provides the opportunity to activate many solid contaminants produced accidentally by industrial processes, researchers worldwide are interested in it (Joseph et al., 2019; Xu and Shi, 2018).

Olivia et al. (2011) studied the Taguchi method optimization of fly ash geopolymer combinations and researched the mechanical characteristics and durability of concrete generated from the ideal mixtures. The aggregate content influences, the ratio of alkaline solution to fly ash, the ratio of sodium silicate to sodium hydroxide, and the process of curing were all considered in nine different combinations. The control mix consisted of ordinary Portland Cement (OPC) concrete with a compressive strength of 55 MPa. T4, T7, and T10 were the three selected ideal mixes. According to the findings, 55 MPa of geopolymer concrete may be manufactured in 28 days. They produced reduced expansion and drying shrinkage, exhibited elastic modulus (14-28) % lower than those of the control mix of OPC, and had superior tensile and flexural strength.

The geopolymer's general composition includes aluminosilicate source materials such as fly ash or fly ash mixed with GGBFS, in addition to an alkaline activator such as a combination of sodium hydroxide (NaOH) and sodium silicate solution (Almufarji et al., 2019). When aluminosilicate-reactive materials are combined with robust alkaline solutions like sodium silicate (NA2SiO3) and sodium hydroxide (NaOH) or potassium silicate (K2SiO3) and potassium hydroxide (KOH), the combination can either be temperature-cured or cured at room temperature (Davidovits, 2008). The building industry is beginning to use geopolymer concrete as an alternative, green material. It is created when materials ironic in silica and alumina react with alkaline fluids.

The compressive strength of geopolymer concretes displays reductions in the strength of (11, 16, 10, and 18) % in 5% of Na2SO4 concentrations, respectively, when matched to controller specimens. This is the lowest value in discount strength when the geopolymer concretes are made of (8, 10, 12, and 14) Mole (Kumaravel and Girija, 2013). The findings of the sulfate assault show that GPC is more resistant to sulfate than OPC. When samples were submerged in a sulfate solution, neither the exterior nor interior appearance of the GPC samples showed any signs of fractures or flaws. On the other hand, after 90 days, OPC sample surfaces started to degrade (Ghanem et al., 2022). Also, Bakharev et al. (2003) investigated acid resistance as a desired attribute for structural materials used in the harsh environments of the chemical, mining, and mineral processing industries, among others, where AAS concrete of grade 40 has greater durability than (OPC) concrete of similar grade beneath acid attack.

The mechanism of degradation comprises C-S-H decalcification and the creation of the soluble salt calcium acetate. Slag paste was shown to be more resistant to degradation of acid solution than OPC paste. The most common mutual activators used in the creation of geopolymers are hydroxide of sodium, silicates of sodium, hydroxide of potassium, and silicates of potassium. According to a study by A. Palomo et al. in 1999, the type of solution used to activate fly ash plays a decisive role in the reaction's growth. The study found that reactions occur at a faster rate when potassium silicates or sodium silicates are used as activators compared to hydroxide-based activators. This suggests that the choice of activator solution significantly influences the reaction kinetics and overall performance of fly ash-based materials (Palomo et al., 1999).

2. Program of experimental

2.1. Materials

In this experiment, fly ash class (F) and OPC are employed to describe two types of binders. As shown in Table 1, the essential proportions have been used. Normal-weight fines passed sieve no. 4, and crushed coarse aggregate (19) mm was used to produce the mixes. Table 2 shows the aggregates' physical characteristics. Solutions of sodium silicates (Na2SiO3) and sodium hydroxide (NaOH) are utilized by way of the alkali activator. The sodium silicate chemical composition is (SiO2 = 32.5%, Na2O = 13.4%, and water = 54% in liquid form).

Table 1. Chemical composition of FA & OPC through X-ray.

Oxides %	FA	OPC
Sio2	47.67	21.34
Al2o3	27.73	5.43
Fe2o3	18.42	2.326
Cao	5.11	60.3
So3	0.34	1.83
C3A	42.38	0.26
MgO	2.65	3.88
Free Cao	….	...
Cl-	…	....
LOI	3.71	2.18

Table 2. (Crushed coarse and fine) aggregates Physical properties.

Property	Crushed Coarse aggregate	Fine aggregate
Dry sp. Gravity.	2.61	2.59
S.S.D. sp. Gravity.	2.64	2.65
Absorption capacity (%)	1.2	2.91

2.2. Design of mix, molding, and sample curing

The research program focuses on producing FA-based geopolymer concrete using just one type of typical aggregate and an alkaline activator with varied molarity. In this operation, fifteen geopolymer concrete mixes were prepared with three mixes of normal concrete. Table 3 provides the parameters of geopolymer mixes designed by the Taguchi method. Moreover, Table 4 provides information on parameters for GPC mixes that were designed using the Taguchi method in the Minitab software program.

Table 5 provides information on the prepared mix amounts. The binder is composed of 90% FA and 10 % cement for all GPC mixtures. OPC was employed to achieve early hardening so that the mold could be removed in 24 hours.

Table 3. Illustrates the parameters of GPC mixes designed by the Taguchi method.

M *	B (%) *	SS/SH *	AA/B *
8	0.15	1	0.3
10	0.175	1.5	0.35
12	0.2	2	0.4
-	0.225	2.5	0.45
-	0.25	3	0.5

* (M) = molarity, (B) = binder content, (SS/SH) = sodium silicate/sodium hydroxide ratio, (AA/B) = alkali activator/binder ratio.

Table 4. Shows the parameters of mixes that were designed by the Taguchi method.

M	B (%)	SS/SH	AA/B
8	0.15	1	0.3
8	0.175	1.5	0.35
8	0.2	2	0.4
8	0.225	2.5	0.45
8	0.25	3	0.5
10	0.15	1.5	0.4
10	0.175	2	0.45
10	0.2	2.5	0.5
10	0.225	3	0.3
10	0.25	1	0.35
12	0.15	2	0.5
12	0.175	2.5	0.3
12	0.2	3	0.35
12	0.225	1	0.4
12	0.25	1.5	0.45

In distilled water, NaOH pellets were dissolved for 24 hours before use, depending on the molarity and concentration required. Both crushed coarse and fine aggregates were prepared in a saturated surface dry state (SSD) before use. The mixing process has a considerable influence on the creation of geopolymer concrete. The produced alkaline activator was added to the solid constituents after the dry components (aggregates and binders) had been well mixed in the mixing pan. To attain more homogeneity, the mixture was wet-mixed for an additional 3 minutes.

Compressive strength has been determined by casting and testing cube specimens of (100 × 100) mm. The molds were sealed after the geopolymer concrete was formed to prevent the specimens from evaporating and deforming within 24 hours. The geopolymer concrete was kept at room temperature until it was tested after being heat-cured in an oven at 65 $^{o\,C}$ for 24 hours.

Before testing, the geopolymer concrete samples had been completely immersed in 10 % sulfuric acid (H2SO4) for eight weeks. The exposure period was constant for each specimen, and the duration was determined based on prior research by Çevik et al. (2018) and Somiyadevi et al. (2019). Moreover, there is no standard test technique for determining geopolymer concrete's durability to acid and sulfate attacks, but ASTM C267-01 (2001) conditioned the steps to studying the polymer concrete's chemical resistance.

Table 5. Generated Mixes proportions.

Mix no.	FA	OPC	sand	Crushed gravel	W/C	SS	SH	M	Extra water %
	kg/m3								
C 1	…	330	708.4	1062.6	0.4	…	…	…	…
NC 2	…	385	672.1	1008.15	0.4	…	…	…	…
NC 3	…	440	633.6	950.4	0.4	…	…	…	…
GP1	297	33	708.4	1062.6	…	49.5	49.5	8	7
GP2	346.5	38.5	672.1	1008.15	…	80.85	53.9	8	7
GP3	396	44	633.6	950.4	…	117.33	58.66	8	7
GP4	445.5	49.5	592.9	889.35	…	159.10	63.64	8	7
GP5	495	55	550	825	…	206.25	68.75	8	7
GP6	297	33	695.2	1042.8		79.2	52.8	10	7
GP7	346.5	38.5	656.7	985.05	…	115.5	57.75	10	7
GP8	396	44	616	924	…	157.1429	62.85714	10	7
GP9	445.5	49.5	622.6	933.9		111.375	37.125	10	7
GP10	495	55	583	874.5	…	96.25	96.25	10	7
GP11	297	33	682	1023	…	110	55	12	7
GP12	346.5	38.5	679.8	1019.7	…	82.5	33	12	7
GP13	396	44	642.4	963.6	…	115.5	38.5	12	7
GP14	445.5	49.5	602.8	904.2	…	99	99	12	7
GP15	495	55	561	841.5	…	148.5	99	12	7

3. Outcomes and discussion

3.1. Visual appearances

Geopolymer specimens' visual appearance shows no evidence of massive. The specimens' color afterward dipping was the same as their color before being exposed to sulfuric acid, and because the quantity of CaO is low, there is very minor surface deterioration and softening on the specimens' surfaces. The softening and deterioration intensified as the dipping duration lengthened and the sulfuric acid concentration increased.

The color of specimens changed from grey to white in the visual appearance of regular concrete; also, the external surface of specimens deteriorated more due to formations of gypsum and ettringites that caused harm to all structures of the matrix. Figure 1 displays the visual deterioration of geopolymer and normal concrete samples before and after 8 weeks' exposure to sulfuric acid.

a. GPC (before) **b. GPC (after)**

a. NC (before) **b. NC (after)**

Fig. 1. Visual deterioration of GP and normal concrete specimens, (a) before and (b) after acid exposure.

3.2. Mass changes after chemical exposure

The geopolymer and normal concrete mass were determined using cubes after being subjected to 10% sulfuric acid for 8 weeks. Whenever exposed to the sulfuric acid solution, the specimens release sulfates into the geopolymer concrete and react with calcium hydroxide products, such as calcium hydroxide and free calcium hydroxide, to produce calcium sulfa-aluminates and calcium sulfates. Also, with the use of OPC, more calcium sulfa-aluminates and calcium sulfates will be generated, which will lead to the density deterioration of geopolymer concrete. On the other hand, the sulfate ions can react with calcium hydroxide to generate gypsum, affecting geopolymer concrete's volume and mechanical characteristics.

Figure 2 illustrates the alteration in mass of geopolymer and normal concrete specimens after sulfuric acid solution exposure at 10% concentration.

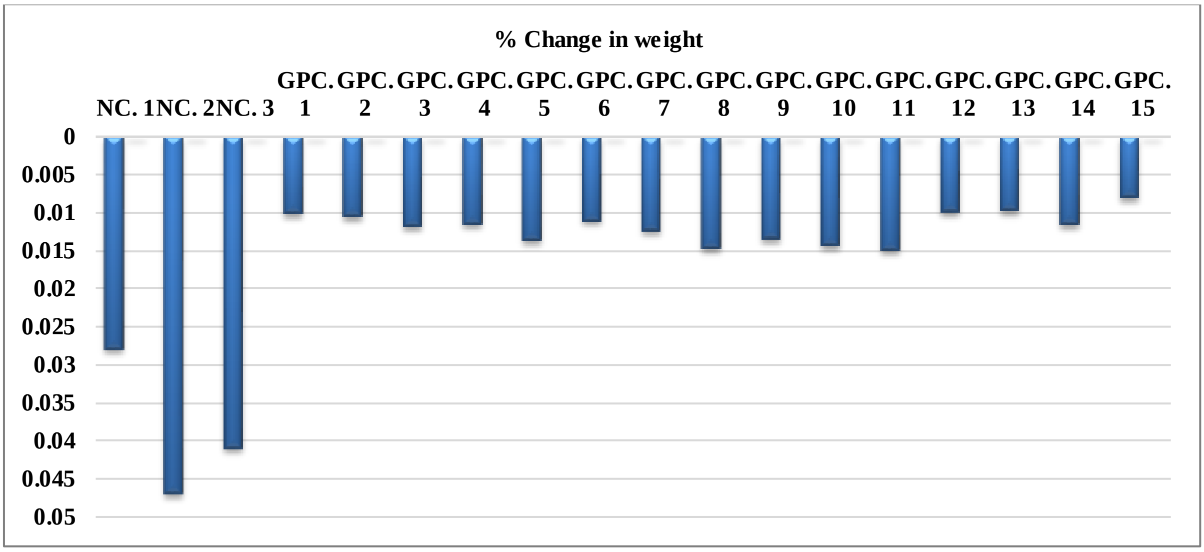

Fig. 2. Change in the mass of normal and geopolymer concrete specimens after exposition to sulfuric acid solution by 10 % concentration.

3.3. Compressive strength

Three cubic samples are averaged to provide the specified compressive strength for each mix. Like how cement hydrates, the geo-polymerization process results in an exothermic reaction and hydrolysis of geopolymer concrete (Wongpa et al., 2010). Results show that geopolymer concrete and normal concrete heat-cured at an early age achieve maximum compressive strength growth in comparison to compressive strength after 28 days because heat curing accelerates internal reactions and causes the strength to develop earlier, as displayed in Figure 3.

Also, the test outcomes showed that the compressive strength increased by way of the weight of the binder increasing for normal and geopolymer concrete, where the compressive strength of normal concrete mixtures improved from 34 MPA to 46 MPA at 28 days as the binder content increased. also, the compressive strength of geopolymer concrete mixtures of 8 molarity improved from 31.845 MPA to 38.855 MPA at the age of 28 days; and for mixtures of 10 molarity, the compressive strength improved from 27.656 MPA to 45.2 MPA at the age of 28 days, as shown in Table 6.

As displayed in Figure 3, the outcomes of mixes indicate a modest strength increase after 28 days versus early strength values due to thermal curing that increases the hydration of geopolymer formation. The compressive strength deteriorated after exposing the geopolymer and normal specimens to a 10% sulfuric acid concentration, as shown in Table 6. After 8 weeks of exposure to a 10% concentration of sulfuric acid, the compressive strength of geopolymer concrete began to decrease with higher concentrations of sulfuric acid solution. The findings indicate that the strength decrease was significant, reaching nearly half the strength of samples that were not exposed to sulfuric acid.

This decrease in strength can be credited to the reaction between sulfate ions from H2SO4 and the alkali activators (NAOH and NA2SO3) present in the geopolymer concrete, which results in a new creation of sodium sulfate (NA2SO4) that deteriorates the external surface of the geopolymer concrete as well as a deformed internal composition of the matrix, both of which negatively affect the geopolymer concrete's performance about properties like compressive strength loss and durability, among others.

Table 6 illustrates the lowest and greatest degradation for geopolymer specimens was 16% and 47% at age 28 days for mixes GPC 3 and GPC 14, respectively. whereas, the loss of strength is affected by zeolite production and the depolymerization of Geo-polymeric products (Bakharev, 2005). Due alumina-silicate bonds breaking during intense sulfuric acid exposure, this was thought to be the cause of the decrease in strength (Mehta and Siddique, 2017).

For normal concrete specimens, the maximum degradation in compressive strength reached 62% for NC 2, and the minimum degradation reached 60% for NC 1, as shown in Table 6. The degradation of compressive strength is due to the high content of calcium in ordinary Portland cement, which causes the gypsum crystals and ettringite to lead to the deterioration of specimens.

Figure 4 illustrates the parameters that affect the compressive strength after exposure to sulfuric acid for GPC (drawn by the Minitab software program).

Table 6. Illustrates the outcomes of compressive strength before and after exposure to sulfuric acid involving percent of reduction.

Mix no.	**Compressive strength MPa**			**Percent of reduction %**
	7 days	**28 days**	**Exposed to Acid**	
NC 1	27.6	34.4	13.7	0.60
NC 2	28.9	38.6	14.6	0.62
NC 3	39.6	46.3	19.5	0.57
GP1	21.6	29.2	21.2	0.27
GP2	24.9	31.8	22.2	0.30
GP3	29.6	36.9	30.9	0.16
GP4	30.9	37.2	24.8	0.33
GP5	32.0	38.8	24.5	0.36
GP6	21.7	27.6	19.6	0.28
GP7	26.0	37.7	27.8	0.26
GP8	24.3	36.4	24.1	0.33
GP9	30.2	29.4	22.5	0.23
GP10	33.6	45.2	28.9	0.36
GP11	27.3	30.6	23.7	0.22
GP12	28.2	35.6	19.5	0.45
GP13	29.6	37.1	21.9	0.41
GP14	30.7	38.0	20.0	0.47
GP15	33.5	39.6	21.7	0.45

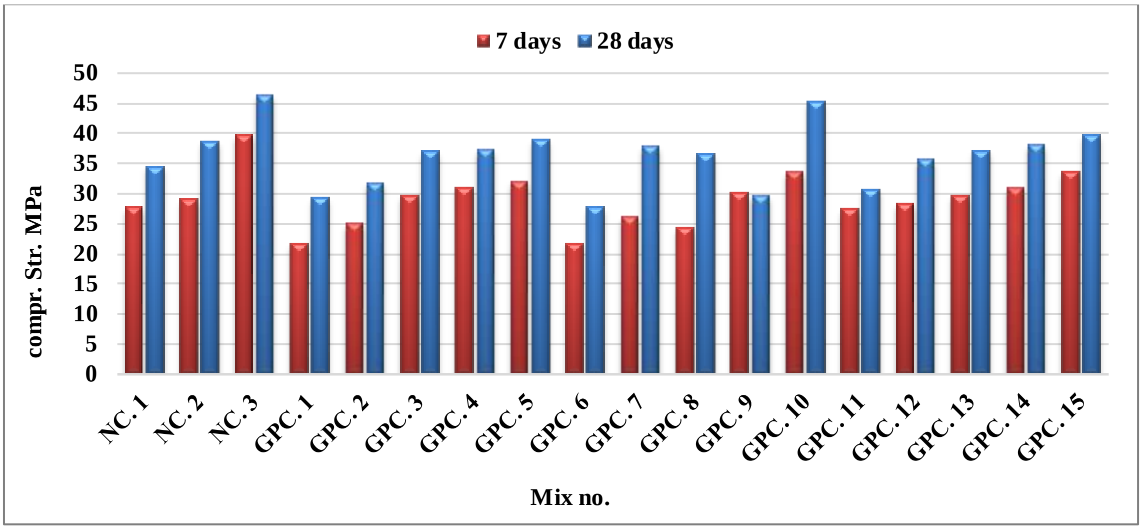

Fig. 3. Strength development with age.

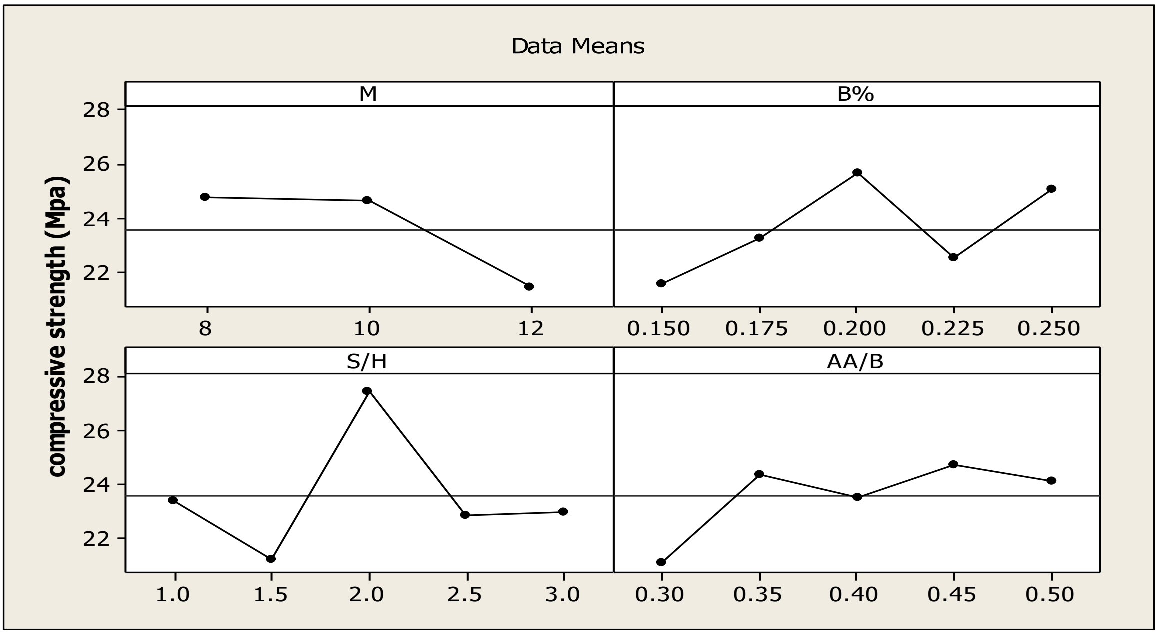

Fig. 4. Illustrates the effect of sulfuric acid on the compressive strength of GPC.

4. Conclusions

Geopolymer concrete against sulfuric acid attacks was studied. The mixtures were ready using different parameters and exposed to sulfuric acid at a concentration of 10%. The following notes concluded:

- ✓ The outcomes gained of GPC compressive strength at 7 days in the range of (21–33) MPa, whereas the compressive strength at 28 days in the range of (27–39) MPa.
- ✓ The optimum parameters ratio that gives minimum strength reduction after exposure to sulfuric acid for GPC was M = 8, B = 20%, SS/SH = 2, and AA/B = 0.45 when studying each factor separately.

- ✓ The visual screening of the geopolymer specimens exposed to 10% sulfuric acid revealed different geopolymer concrete specimens' surface deterioration with no cracking marks and no color change, demonstrating that the GPC has good resistance to acid and sulfate attack and greater stability.
- ✓ The alteration in compressive strength in the geopolymer specimens is in range (16-47) %. Whereas in the normal concrete specimens was (57 – 62) %.
- ✓ The maximum and minimum mass loss in geopolymer specimens after 10% acid exposure for 8 weeks was (1.5 and 0.81) % for GP 11 and GP 15, respectively.
- ✓ The maximum and minimum mass loss in normal specimens after 10% acid exposure for 8 weeks was (4.7 and 2.8) % for NC 2 and NC 1, respectively.

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