Research Article

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Experimental Investigation of the Durability of Ambient-Cured Metakaolin-Based Geopolymer Concrete in Different Sustainable Environmental Conditions

M. Nanthini, R. Ganesan and V. Jaganathan*

Department of Civil Engineering, Saveetha School of Engineering, Saveetha Institute of Medical and Technical Sciences, Chennai, TN, India

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*jaganathanvenugopal.sse@saveetha.com

ABSTRACT

Geopolymer concrete (GPC) has received much attention among researchers in recent decades due to its increased durability properties. It helps reduce massive pollution and high energy consumption during cement production. Various cementitious materials having high alumino-silicate, such as, fly ash (FA), ground granulated blast furnace slag (GGBS), rice husk ash (RHA), and metakaolin (MK), are typically used for producing GPC. These materials are activated by an alkaline solution through a polymerization process. This study examined the durability properties of metakaolin-based GPC with fly ash and GGBS under ambient curing conditions (23 ± 20 ℃). The mechanical characteristics like compressive strength, splitting tensile strength, flexural strength, and durability properties like the sorptivity volume of permeable voids exposed to chloride, sulphate, and acid environments were studied and reported. Also, an effort has been made to establish a relationship among the mechanical properties. The test results show that better performance of GPC can be achieved with 40% replacement of MK with 10% FA and 50% GGBS under ambient curing than controlled concrete. The MK-GPC with a higher proportion of metakaolin, showed good resistance to environmental factors. It is a potential alternative to conventional cement concrete.

Keywords: Geopolymer; Water absorption; Sorptivity; Metakaolin; Fly ash; Ground Granulated Blast Furnace slag.

1. INTRODUCTION

Many engineers have been looking for a novel material to substitute the high CO_2-emitting Ordinary Portland Cement (OPC) manufacturing method due to concerns about harmful global warming. Subsequently, geopolymer has been suggested as a workable alternative due to its remarkable endurance, relatively good mechanical properties, and low carbon footprint (Albitara *et al.* 2017). The geopolymer manufacturing process is analogous to that of OPC, except that this process involves various curing stages and chemical processing with the aid of aluminium silicate and sodium hydroxide through the alkaline process (Almutairi *et al.* 2021). Different raw materials are obtained from industrial waste materials for producing geopolymer concrete (GPC), such as Ground Granulated Blast Furnace Slag (GGBS), Silica Fume (SF), Waste Glass Powder (WGS), and Fly Ash (FA), since the chemical activation is more important. It helped reduce waste disposal defects, reduce the greenhouse effect of CO_2 emission, and replace OPC in commercial applications.

In contrast to appearing more environmentally sustainable, GPC has been described as having comparable durability and mechanical properties to OPC (Vafaei *et al.* 2017). However, at the limits of cementitious materials processes, especially in exterior constructions, geopolymer thermal curing (at 60-1200 C for 24-48 hours) is often required during manufacturing. Therefore, many studies focused on using properly chosen alumino-silicate components to avoid the sluggish curing of cementitious materials (Nawaz *et al.* 2020).

Geopolymerization is the combination of aluminium (Al), and silicon (Si) natural source Metakaolin (MK) and various by-product like fly ash and GGBS, activated by alkaline activators, that form geopolymer binders (Ma *et al.* 2018; Morsy *et al.* 2014). The aggregated elements in OPC are bound together by cementitious materials, which act as a C-S-H. Metakaolin is one of the silicate mineral sources used most frequently in GPC. This MK is produced by calcining organic clay minerals (kaolin) at an optimized temperature. Sometimes, sodium silicate (Na_2SiO_3) or K_2SiO_3, KOH, and NaOH act as alkaline activating agents in cementitious material mixtures (Nazari *et al.* 2011). The calcium presence in GPC can prolong the strength of pozzolanic concrete, reduce strength development, and sometimes increase shrinkage at very shortages, mainly when using a high molar ratio of SiO_2/Al_2O_3 (Lothenbach

et al. 2011; Cho *et al.* 2017). Accelerating the ageing process using heat curing and increased calcium binding admixture is critical to compensate for the shortfall in geopolymer setting time (cement and GGBS) (Yusuf et al., 2020). According to a recent research work, adding alumino-silicate binders with a calcium source improves the structural properties of cementitious materials in the case of GPC (De and Sagoe, 2008).

Table 1. Chemical properties of binders

S. No.	Chemical compound	GGBS	Fly Ash	Metakaolin
1	SiO_2 (%)	37.18	48	50.7
2	Al_2O_3 (%)	13.87	29	45.8
3	Fe_2O_3 (%)	1.11	6.23	0.35
4	CaO (%)	36.79	10.75	0.39
5	MgO (%)	8.71	2.36	0.08
6	Na_2O (%)	0.25	0.39	0.58
7	K_2O (%)	0.29	0.55	0.05
8	SO_3 (%)	0.39	1.11	Nil
9	TiO_2 (%)	Nil	Nil	1.25
10	Loss on ignition	1.41	1.61	0.86

Low calcium and calcium-free alkaline activation materials receive better durability in aggressive atmospheres in sulphate and acidic solutions (Wanga *et al.* 2020; Ismail *et al.* 2013). This material can include portlandite and calcium ($Ca_4(AlO_2)_6SO_4$) compounds that are not formed throughout the polymerization reaction but in the form of Sodium aluminosilicate hydrate gel with its enhanced protection against sulphuric acid (Temuujin *et al.* 2009). Compared to sodium, aluminosilicate hydrate gel, high in calcium, typically generates calcium-containing compounds, including gel and C-(A)-S-H gel. These gels and hydrates are less resistant to acid and sulphate than Sodium aluminosilicate hydrate gel (Džunuzović *et al.* 2017). Natural resources of aluminosilicate such as high calcium (Class F) industrial waste are the foundation for many contemporary cementitious materials investigations. However, there is a lack of information on MK-GPC-containing calcium compounds regarding their mechanical strength and concerns surrounding their lifespan durability. Therefore, a detailed study on MK-based geopolymers is needed. Previous experimental studies clearly show that MK-GPC combination was limited, and the effect of mixtures with different design parameters and alkaline-treated GPC was not reported.

This study aimed to produce GPC using the Na_2SiO_3 and 14 M NaOH mixed with 0% to 50% replacement level of Metakaolin (MK) with fly ash and 50% GGBS cured under ambient temperature. The mechanical characteristics of each mixed proportions were determined, including splitting tensile, flexural, and compressive strength. To evaluate the effects of porosity and permeability of GPC constituted under ambient curing, tests for sorptivity and volume of voids were performed. The resistance against chloride, sulphate, and acidic environments for exposure periods of 30, 90, and 180 days was studied.

2. MATERIALS AND METHODS

2.1 Materials

Fly ash, MK, GGBS, fine aggregate, coarse aggregate, and sulfonated naphthalene formaldehyde-based superplasticizer were used in this study to produce MK-GPC. Metakaolin is a clay mineral obtained from heating the clay mineral kaolin at a controlled temperature of 600°C - 700°C. GGBS is an industrial byproduct that's generated from the production of iron. GGBS, FA, and MK binders are rich in silica and alumina and exhibit pozzolanic properties. Their chemical properties are given in Table 1. This study used river sand as a fine aggregate, and coarse aggregates were procured from a local supplier. Their physical properties are given in Table 2. The alkaline liquid used in this investigation was a mixture of sodium (NaOH) and sodium silicate (Na_2SiO_3).

Table 2 Physical properties of aggregates

S. No.	Physical properties	Fine Aggregate	Coarse Aggregate
1	Specific gravity	2.5	2.8
2	Water absorption	5.7%	3.4%
3	Bulk density (compacted state)	1640 kg/m^3	1654 kg/m^3
4	Bulk density (loose state)	1427 kg/m^3	1459 kg/m^3
5	Fineness modulus	2.2	5.5
6	Percentage finer than 75µ	1.84%	-

2.2 Mix Proportion and Methodology

The mix proportion adopted in this study was based on the absolute volume method. Six mix proportions were considered in which the binder material GGBS and fly ash were adopted 50% each. The mix combinations and their compositions are given in Table 3. The alkaline solution:binder ratio was fixed as 0.3. The sodium silicate to sodium hydroxide ratio was 2.5. For all the combinations, a superplasticizer of 1.5% of the binder was used. The sodium hydroxide (14 M) was prepared by dissolving 560 g of NaOH pellets in distilled water to make up 1 litre of solution, and it was combined with sodium silicate before 24 hours of manufacturing GPC. Mechanical strength characteristics like compressive strength, splitting tensile strength, and flexural strength

were determined after the concrete was cast. Then, they were allowed to cure at the ambient temperature (IS516 (Part 1/Sec 1): 2021; IS 5816: 1999). The porosity was assessed using the sorptivity test, and permeability was assessed by volume of voids test (ASTM 1585–20; ASTM C642-21).

Table 3. Mix proportions of Geopolymer and OPC

Mix ID	Binders			Alkaline Solution/Binder	Alkaline Solution		Aggregate Ratio		SP %
	GGBS	FA	MK		NaOH	$NaSiO_3$/NaOH	Fine Aggregate	Coarse Aggregate	
MK-00	50%	50%	0%	0.3	14M	2.5	40%	60%	1.5
MK-10	50%	40%	10%	0.3	14M	2.5	40%	60%	1.5
MK-20	50%	30%	20%	0.3	14M	2.5	40%	60%	1.5
MK-30	50%	20%	30%	0.3	14M	2.5	40%	60%	1.5
MK-40	50%	10%	40%	0.3	14M	2.5	40%	60%	1.5
MK-50	50%	0%	50%	0.3	14M	2.5	40%	60%	1.5

Table 4. Comparison of measured and predicted values

Mix ID	Splitting Tensile Strength			Flexural Strength		
	Measured MPa	Predicted MPa	Error %	Measured MPa	Predicted MPa	Error %
MK-00	6.40	5.84	8.68	4.30	3.98	7.43
MK-10	6.70	6.66	0.53	4.60	4.57	0.60
MK-20	6.80	6.83	-0.47	4.70	4.69	0.15
MK-30	7.00	7.02	-0.21	4.80	4.83	-0.53
MK-40	7.30	7.25	0.62	5.10	5.00	2.00
MK-50	7.10	7.13	-0.49	4.80	4.91	-2.33

3. RESULTS AND DISCUSSION

3.1 Mechanical Properties

The compression strength was measured using a 150 mm concrete cube specimen in compliance with IS 516:2021; splitting tensile strength was measured using a cylindrical specimen with a 150 mm diameter and 300 mm length in compliance with IS 5816:1999, and flexural strength was measured using a beam specimen in compliance with IS 516:2021.

The maximum compressive strength was attained at 40% MK replacement and decreased with further replacement level. The compressive strength varied from 44.64 N/mm2 to 61.2 N/mm2. This range shows that the compressive strength increased with the incremental replacement level of MK. This is because MK has a lower size percentage than GGBS, which leads to increase in particle packing density. In the case of splitting tensile strength and flexural strength, the maximum value was observed at 40% MK replacement, which indicates that the replacement of MK with GGBS increases the mechanical property by up to 40%. The mechanical properties for various replacement levels and the graph representing the strength incremental level are shown in Fig. 1.

An effort has been made to establish a relationship between the mechanical properties of compressive strength (fc) with splitting tensile strength (fct) and compressive strength (fc) with flexural strength (fcr). The plot showing the relationship between compressive strength (fc) and splitting tensile strength (fct) is depicted in Fig. 2 and the regression equation thus obtained is:

$$f_{ct} = 1.1919\sqrt{f_c} - 1.9931 \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

where f_c, f_{ct} is in N/mm^2

The relationship between compressive strength (f_c) and flexural strength (f_{cr}) is plotted in Fig. 3, and the regression equation thus obtained is:

$$f_{cr} = 0.86\sqrt{fc} - 1.6744 \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad (2)$$

where f_c, f_{cr} are in N/mm^2

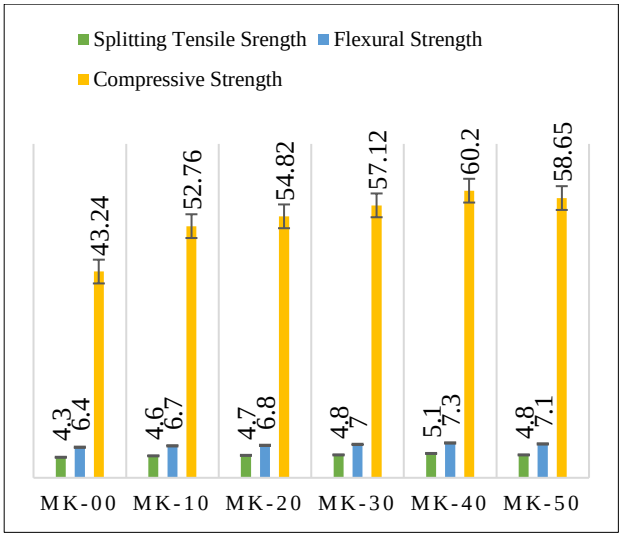

Fig. 1: Mechanical strength of MK-GPC

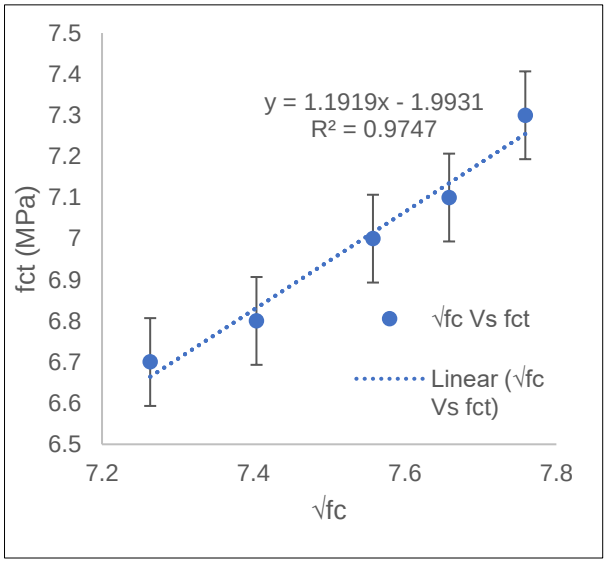

Fig. 2: Relationship between $\sqrt{f_c}$ Vs f_{ct}

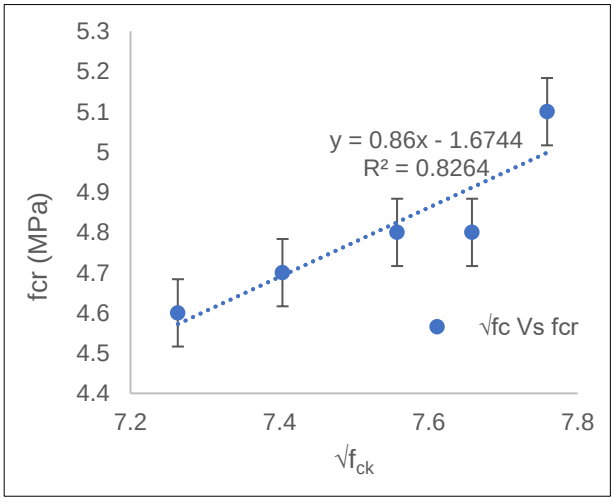

Fig. 3: Relationship between $\sqrt{f_c}$ Vs f_{cr}

The predicted values of splitting tensile strength using equation (1) were compared with the experimental values for different mix proportions are given in Table 4, and also the predicted values of flexural strength using equation (2) were compared with the experimental values for different mix proportions. The predicted error was about 8.68% in the case of splitting tensile strength and 7.43% in the case of flexural strength.

3.2 Durability Properties

3.2.1 Sorptivity

The rate of water ingress in concrete occurs due to absorption by capillary action through the pores in the concrete. The sorptivity test was conducted following ASTM C 1585. The 28-day ambient cured concrete specimen of diameter 100 mm and 50 mm in height was used to determine the absorption (I) in mm, and the test results obtained are shown in Fig. 4. The absorption varied from 0.65 mm to 1.47 mm; the minimum absorption was observed at MK-40, with a 40% MK replacement with GGBS. The capillary suction of fluids was more in the 0% MK mix proportion. With a further increase in MK replacement levels, the pores were reduced since particle sizes of MK were lesser than GGBS.

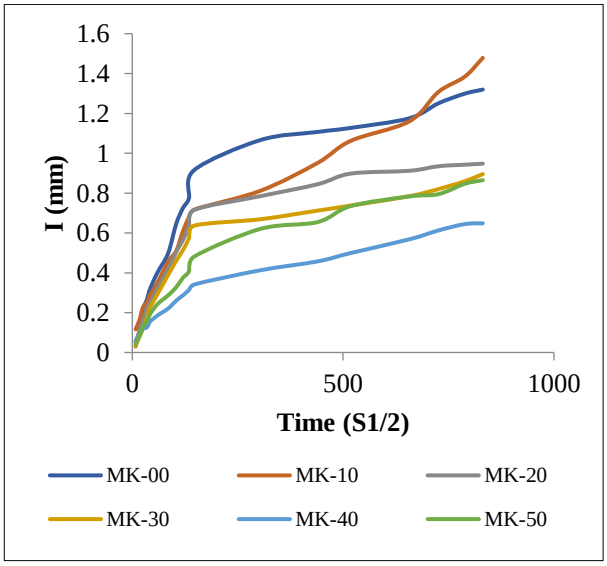

Fig. 4: Sorptivity of MK-GPC

The initial (rate of water absorption from 1 minute to 6 hours) and secondary (rate of water absorption from 1 day to 7 days) absorption rates were calculated from the sorptivity test results and plotted (Fig. 5). The slope of the best-fit line for both the initial and secondary absorption against the square root of time in seconds was used to determine the coefficients. In the case of initial absorption, the coefficient decreased from 0% MK replacement level to 40%, after which it started increasing. The coefficient at 40% was 0.0019 mm $s^{1/2}$

and, in the case of secondary absorption, it decreased with increasing replacement level of MK. A better value (0.0003 mm $s^{1/2}$) was obtained at 20% replacement, which indicates that all the pores were filled during initial absorption.

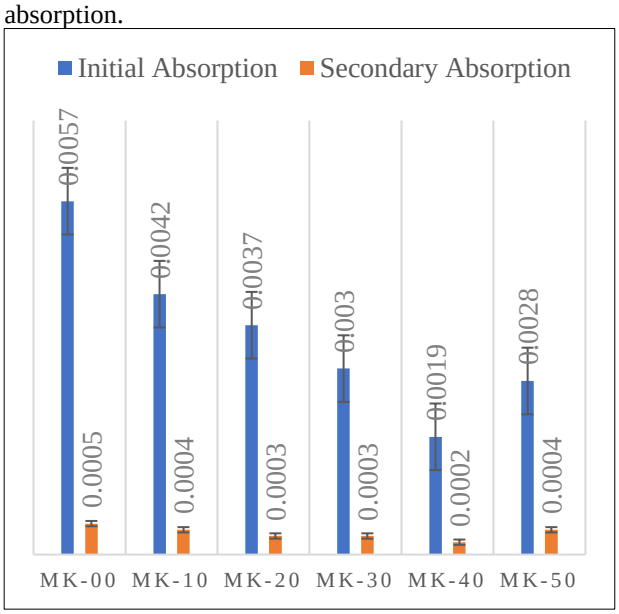

Fig. 5: Initial and secondary absorption of MK-GPC

3.2.2 Volume of Permeable Voids

According to ASTM C-642, a quantitative assessment of the apparent volume of permeable voids was carried out. The permeable voids (%) volume was calculated using an ambient-cured concrete specimen with a diameter of 100 mm and a height of 50 mm. The specimens were cured for 28 days. The apparent volume of permeable voids decreased with increasing replacement levels up to 40% replacement of MK with GGBS, after which an increase in MK increases the apparent volume of voids. The apparent volume of voids was in the range 17.97% to 11.87%, as presented in Fig. 6.

3.2.3 Resistance of MK-GPC Against Chloride Attack

Chloride salts have a deleterious effect on concrete. The efficiency of the MK-based GPC specimens when subjected to a chloride environment was determined by simulating a sodium chloride solution. A solution of 5% sodium chloride was prepared using deionised water. For each test interval (in days), the specimens were initially weighed and then fully submerged in the chloride solution for the required test duration. During testing, the samples were removed and cleaned with running tap water, surface moisture was wiped off, and air drying was allowed for half an hour. The specimens were inspected visually for any colour change, and the test solution was replaced when there was a colour change. The compressive strength was determined at 30-, 90- and 180-day immersion and the percentage of reduction in compressive strength and weight loss were calculated.

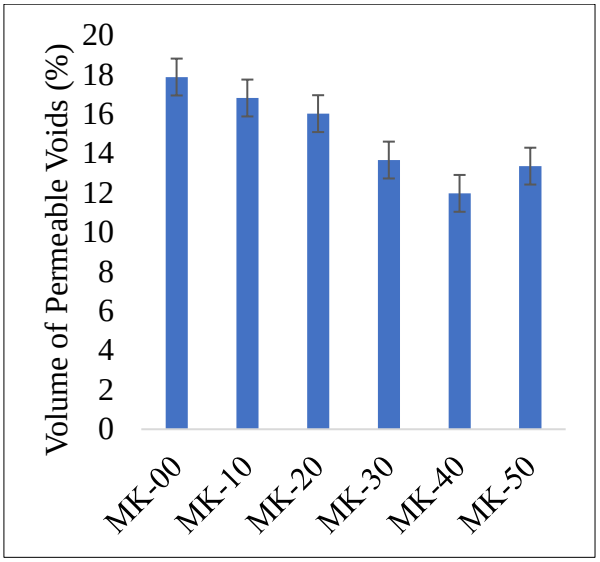

Fig. 6: Volume of voids (%) of MK-GPC

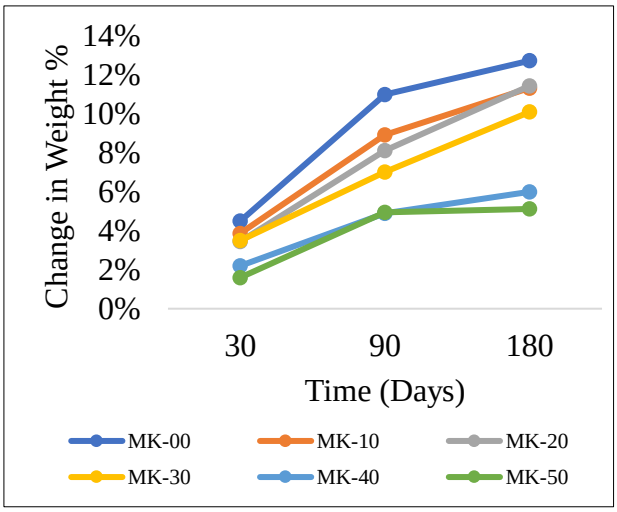

Fig. 7: Change in weight of MK-GPC exposed to Chloride environment

At 30, 90 and 180 days of exposure to a chloride environment, the visual inspection showed no distinct changes in the physical appearance of a specimen. However, the surface started degrading with time, although quantitative determination was impossible by simple visual inspection. The % increase in weight with increasing exposure time is displayed in Fig. 7. Weight gain in percentages ranged from 3.52% to 8.23%. Higher weight gain was observed with a lower replacement level of MK. Fig. 8 shows the change in compressive strength for different exposure periods in a chloride environment. After 30 and 90 days, there was an increase in compressive strength in the range 10.67% to 15.84% and 12.88% to 19.80%, respectively. At 180 days of

exposure, the drop in compressive strength was observed in the range -11.45% to 14.10%. The hydration of Ca-Al-Silicates (CAS) from the dissolution of the CAS phase inhibited the growth of specimen strength. This is due to the presence of hydroxyl ion (OH-) from aqueous NaOH. The attachment (OH-) caused Al and Si to become pentavalent. Al-O-Al and Si-O-Si connections often become stronger (Yousuf et al., 2014).

3.2.4 Resistance of MK-GPC Against Sulphate Attack

Sulphate salts have a deleterious effect on concrete, which prevalently occur on soils around coastal zones. Calcium sulphates are formed when calcium hydroxide and sodium sulphates are combined. It has been suggested that adding pozzolans, such as fly ash, to concrete increases its resistance to sulphates as the curing process extends. This test focuses on determining the efficiency of MK-based GPC specimens when subjected to a sulphate environment by simulating a sodium sulphate solution and testing by determining the weight loss and the loss of compressive strength before and after immersing in the solution. The sodium sulphate solution was prepared to simulate the prevalent field conditions in the soil groundwater (Sanjuktha *et al*. 2015). A solution of 5% sodium sulphate was prepared to simulate the field conditions of underground structures exposed to soils with sulphates. The specimens were first weighed and then completely submerged in the sulphate solution for the necessary test period for each test interval (measured in days). The specimens were removed prior to testing, cleaned under running tap water, removed surface moisture, and left to air dry for half an hour. The specimens were inspected visually for any colour change, and the test solution was replaced when there was a change in the colour of the solution. The compressive strength was determined at 30-, 90- and 180-day immersion and the percentage of reduction in compressive strength and weight loss were calculated.

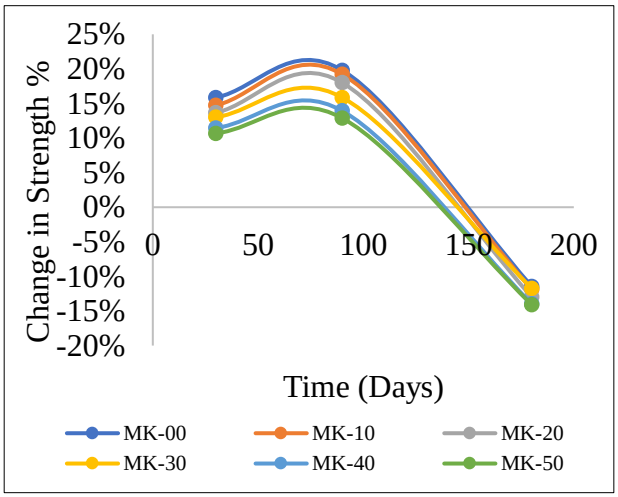

Fig. 8: Change in compressive strength of MK-GPC exposed to Chloride environment

During the 30 days and 90 days of exposure to a sulphate environment, the visual inspection shows no distinct changes in the physical appearance of the specimen. However, the crystallized salt formation was seen on the surface of the specimen, which was removed when the specimens were washed. The specimens remained stable and intact for 180 days, exposed to a sulphate environment. However, mild surface scaling was observed on the specimens with 30% - 50% MK replacement.

The % increase in weight with increasing exposure time is displayed in Fig. 9. The percentage of weight gain falls between 3.42% and 8.25%. Increased weight gain was seen when MK replacement levels were lower. Due to a more stable cross-linked alumina silicate polymer structure than the typical Portland cement hydration structure, the geopolymer mortars performed better in the sulphate attack (Vanchai *et al.* 2012). Fig. 10 shows the compressive strength change for different exposure periods in sulphate environments. The rise in compressive strength observed in the 30 and 90-day ranged from 10% to 15.33% and 12.29% to 19.33%, respectively. At 180 days of exposure, the drop in compressive strength was observed in the range -10.93% to 13.54%. Since the geopolymers containing MK are unable to produce sulphate-aluminate products such as, gypsum and ettringite through the dehydration process and reaction to sulphate solution, this may be caused by MK-GPC with low level of Ca+, which has a detrimental effect on sulphate resistance (Duan *et al*. 2015).

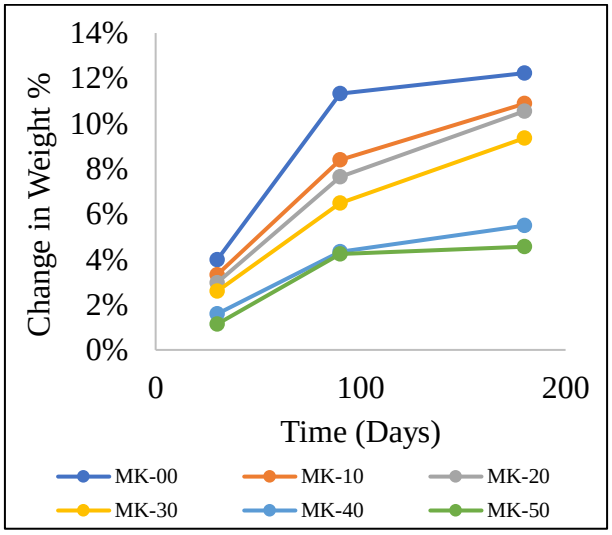

Fig. 9: Change in weight of MK-GPC exposed to sulphate environment

3.2.5 Resistance Properties of MK-GP Against Acid Attack

This test focuses on determining the efficiency of the MK-based GPC cured in ambient condition. The specimens were subjected to an acidic environment by simulating a sulphuric acid solution and testing the

weight loss and the loss of compressive strength before and after immersing in the solution. Sulphuric acid is an inorganic acid that may occur naturally in ground waters and soils and results from oxidative weathering of certain sulphide minerals like iron disulphides and pyrites. A solution of 5% sulphuric acid was made with 98% concentrated sulphuric acid (Sanjuktha *et al*. 2015; Siad *et al*. 2010). For each test interval (in days), the specimens were weighed and then fully immersed in the solution for the required test duration. At the time of testing, the specimens were removed and cleaned with running tap water and surface moisture was wiped off and allowed to air dry for 30 minutes. The specimens were inspected visually for any colour change, and the test solution was replaced when there was a change in the colour of the solution. The compressive strength was determined at 30-, 90- and 180-day immersion and the percentage of change in compressive strength and weight loss were calculated.

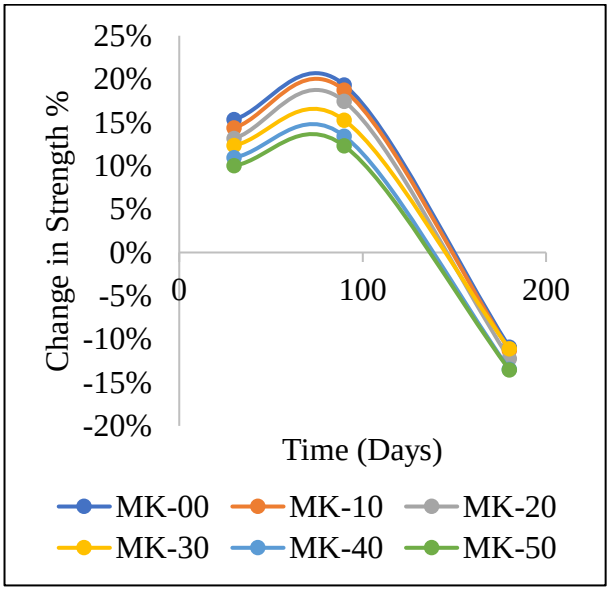

Fig. 10: Change in compressive strength of MK-GPC exposed to sulphate environment

At 30 days of exposure to an acidic environment, the visual inspection shows that the specimens did not undergo distinct changes in shape; a mild change in the colour of the specimens and disintegration of the edges were observed. At 90 days, the matrix dissolution was observed, and at 180 days, the coarse aggregate in the specimens was exposed, indicating the surface paste content was dissolute in the acidic environment. However, when the specimens were broken, the inner part in specimens was intact, and stability was observed.

The percentage change in weight and compressive strength is shown in Figs. 11 and 12, respectively. The weight of the specimen decreased as the time interval of the immersion period increased. In the case of an increase in the percentage of MK replacement, it is inverse. It is observed that a specimen with 0% has a higher percentage of weight loss compared to a higher replacement level of MK. In case of change in compressive strength, the same pattern is observed, decreased in compressive strength as time interval increases.

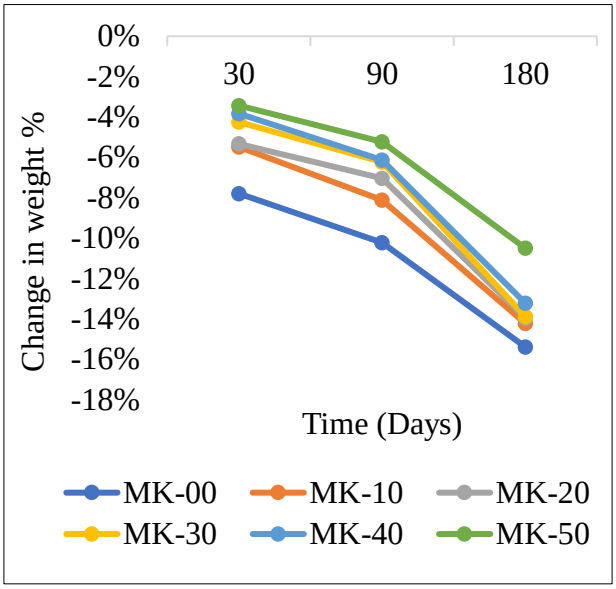

Fig. 11: Change in weight of MK-GPC exposed to acid environment

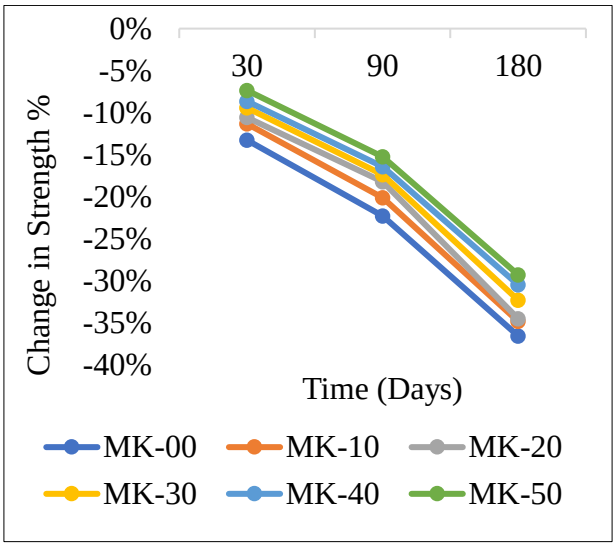

Fig. 12: Change in compressive strength of MK-GPC exposed to acid environment

A lesser percentage decrease in compressive strength was observed in the case of increased MK replacement level. During the 180-day immersion period, the test sample showed a decrease in the percentage of weight loss and compressive strength in the range 10.5% to 11.39% and 29.3% to 36.54%, respectively. The deterioration was due to the leaching of Al and Na from

the geopolymer structure in the presence of sulphuric acid. An increase in the SiO_2 / Al_2O_3 ratio after immersion probably led to deterioration and caused a reduction in strength (Vanchai *et al*. 2012; Bakharev, 2005).

4. CONCLUSION

The present investigation studied the mechanical characteristics like compressive strength, splitting tensile strength, flexural strength, sorptivity, volume of permeable voids, and durability of MK-based GPC under ambient curing conditions. The essential findings of the experimental results areMetakaolin with fly ash and GGBS produce better geopolymer concrete (GPC) under ambient curing conditions. The mechanical characteristics like compressive strength, splitting tensile strength and flexural strength were maximum at 40% metakaolin (MK) replacement with fly ash and 50% GGBS and increased at 27%, 15% and 12%, respectively.

1. Metakaolin with FA and GGBS produce better GPC under ambient curing conditions. The mechanical characteristics were maximum at 40% MK replacement with FA and 50% GGBS and increased at 27%, 15% and 12%, respectively.

2. The sorptivity showed better results at a 40% replacement level of MK with FA and 50% GGBS due to the matrix densification, reducing the pore system of the concrete specimens.

3. The apparent volume of voids was found to be low in the case of 40% replacement of MK with FA and 50% GGBS since fines of the MK compared to GGBS reduce the voids.

4. The resistance to chloride environment showed a strength gain of up to 90 days of exposure condition. A further increase in exposure conditions decreased the strength due to the hydration of Ca-Al-Silicates from the dissolution of the CAS phase.

5. The resistance to sulphate environment showed a strength gain of up to 90 days exposure condition and a further increase in exposure conditions decreased the strength since MK-based GPC was unable to produce sulphate-aluminate products such as, gypsum and ettringite through the dehydration process and reaction to sulphate solution.

6. As a result of the leaching of Al and Na from the geopolymer structure in the presence of sulfuric acid, the resistance to acid environments demonstrated a strength loss of up to 35%.

7. The strength, porosity and permeability properties were enhanced due to the replacement of MK with FA and 50% GGBS in GPC under ambient curing conditions.

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CONFLICT OF INTEREST

The authors declared no conflict of interest in this manuscript regarding publication.

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