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Original Research Paper

An Evaluation of the Behavior of Metakaolin-Based Geopolymer Concrete Exposed to Magnesium Sulfate Attack Based on Non-destructive Testing Methods

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Abstract

The production of conventional Portland cement (OPC) entails significant consumption of primary raw materials and results in a substantial release of CO_2, contributing to greenhouse gas emissions and global warming. To address these environmental challenges, geopolymer concrete has emerged as an alternative that eliminates the need for cement. However, geopolymer concrete may be subjected to potential degradation when exposed to aggressive environments such as seawater, industrial waste, and areas with magnesium sulfate presence, which can affect its mechanical and durability properties. This study focused on evaluating the performance of metakaolin-based geopolymer concrete (MKGPC) exposed to magnesium sulfate attack based on non-destructive testing methods. The MKGPC was prepared using a binder ratio of 1:1.6:3.7 and a sodium hydroxide (NaOH) concentration of 16 molar, mixed with sodium silicate ($NaSiO_2$) at a ratio of 1:2.5. Concrete cubes of 100x100x100 mm were cast, allowed to set and harden for 24 hours, and then subjected to a temperature of 60°C for 24 hours before immersion in a 2.5% magnesium sulfate solution. Tests for compressive strength, ultrasonic pulse velocity, and rebound hammer were carried out at 7, 14, 28, and 56 days. Additionally, a water absorption test was conducted at 28 and 56 days. The experimental results revealed a slight increase in compressive strength of MKGPC samples exposed to magnesium sulfate attack at 7, 14, and 28 days, followed by a decrease of 13.09% at 56 days. Similarly, ultrasonic pulse velocity and rebound number exhibited initial increases at early ages but decreased at 56 days. The study established that prolonged exposure to 2.5% magnesium sulfate solution has a slight negative impact on the mechanical properties of MKGPC. Therefore, it should not be used for infrastructure in environments where the magnesium sulfate concentration is up to 2.5%.

Keywords: Aggressive Environment, Concrete, Geopolymer, Metakaolin, Magnesium Sulfate

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1. Introduction

Concrete, renowned for its affordability, exceptional endurance, and widespread availability of constituent materials, stands as the most utilized construction material globally (Shaikh, 2016). The infrastructure development is driving its demand continually upward (Prakash & Dave, 2013). The necessity for the utilization of Ordinary Portland cement (OPC) in construction arises as it serves as a binding agent in concrete production (Chowdhury *et al.*, 2021). OPC's prevalent usage in concrete production stems from its capacity to bind aggregates with water, yielding cement-based structures of varying sizes and shapes (Wong, 2022). However, OPC production leads to significant consumption of natural resources and energy, resulting in substantial carbon dioxide emissions, which present environmental challenges. Annually, the global OPC industry is responsible for emitting approximately 2.6 to 2.8 billion tons of greenhouse gases, constituting 5-9% of total man-made emissions (Prakash & Dave, 2013; Rajerajeswar *et al.*, 2014**)**. If considered as a standalone entity, the cement industry would rank third globally in greenhouse gas emissions, trailing only China and the United States of America (Nehdi & Yassine, 2020). Consequently, in response to the growing concerns over the socioeconomic impacts of carbon emissions and climate change, international bodies are increasingly advocating for carbon neutrality in the construction sector. This shift necessitates a transition away from carbon-intensive practices, such as the heavy reliance on OPC, toward the adoption of alternative, environmentally friendly cementitious materials. (Kaya *et al.*, 2022). Additionally, to achieve the targets set by the Paris Agreement, which aims to limit global warming to below 2°C by 2030 and net-zero carbon emissions by 2050, requires urgent reforms in the construction industry, including the reduction of OPC usage and the promotion of sustainable building materials (Matsimbe *et al.*, 2022; Bheel *et al.,* 2021).

One of the most viable options for adopting environmentally friendly materials is either reducing the use of OPC in concrete or employing alternative materials, such as geopolymer concrete (Prakash & Dave, 2013). Geopolymer concrete, which is produced by eliminating or minimizing OPC use, represents a sustainable solution supporting recycling efforts (Herwani *et al.,* 2018; Gupta *et al.,* 2020). It is formed through the reaction between alumino-silicate materials and alkaline agents, which yields a semi-crystalline amorphous substance after curing (Castillo *et. al.,* 2021). Metakaolin, being rich in silica and alumina, serves as a prevalent source material for geopolymer production (Jena *et. al.,* 2019). When combined with alkali activators, metakaolin or byproducts such as fly ash yield a geopolymer binder (Abiodun et. al., 2022).

Previous research efforts have explored the viability of metakaolin-based geopolymer concrete by investigating various mix design parameters. A study conducted by Albidah *et al.* (2021) revealed promising compressive strength values at 28 days, with different sodium silicate to NaOH ratios yielding strengths of 57.6 to 58.5 MPa. However, higher ratios led to a reduction of compressive strength. Similarly, a study on the influence of coarse aggregate size conducted by Fazil *et al.* (2021) established that metakaolin-based geopolymer concrete exhibited diminishing compressive strength as aggregate size increased. Although compressive strength is one of the key parameters for durability assessment, it does not fully assess the viability of alternative materials (Shetty, 2005).

During service life, concrete structures may likely experience some form of deterioration. One of the most severe forms of deterioration is sulfate attack (Chakkor et *al.,* 2021). Sulfates, notably magnesium sulfate, pose significant risks, leading to the expansion, spalling, and softening of concrete (Shetty, 2005). Conventionally, the quality of concrete structures exposed to sulfate attack is determined by removing a sample from the structure via destructive means such as coring (Cumming, 2004). This may leave flaws that can propagate failure in the future. Deterioration from a sulfate attack can compromise the load-bearing capacity of concrete structures, which creates a demand for a reliable nondestructive testing (NDT) technique that can detect the onset and severity of degradation in concrete structures (Khumtorna & Chotickai, 2025).

Therefore, this study examined the performance of metakaolin-based geopolymer concrete (MKGPC) subjected to magnesium sulfate attack using nondestructive testing (NDT) methods. The investigation focused on assessing the effects of moderate sulfate exposure on MKGPC and establishing a correlation between destructive testing (compressive strength) and nondestructive methods (ultrasonic pulse velocity and rebound hammer). The established relationship provides a basis for evaluating the extent to which NDT techniques can be effectively employed to assess the behavior and durability of MKGPC under moderate magnesium sulfate attack.

2. Materials and Experimental Procedure

2.1 Materials

i. Metakaolin

A sample of kaolinite clay was obtained from the Kankara Local Government Area of Katsina State, Nigeria. The clay underwent a calcination process in an electric furnace at 800°C for 90 minutes to transform it into metakaolin. Subsequently, the material was passed through a 45-micrometer sieve to eliminate larger particles. The oxides of the metakaolin utilized in the study are presented in Table 1. The analysis revealed that the metakaolin consisted of 57.95% SiO_2, 36.51% Al_2O_3, and 2.03% Fe_2O_3. This SX indicates that the combined proportion of SiO_2, Al_2O_3, and Fe_2O_3 exceeded 70%. Thus, the metakaolin met the requirement outlined in NBR 12653, which specifies that the combined proportion of SiO_2, Al_2O_3, and Fe_2O_3 should not be less than 70%.

ii. **Alkaline Solution**

The alkaline solution was produced by dissolving solid sodium hydroxide (NaOH) in water, resulting in a solution with a 16-molar concentration of NaOH. The NaOH solution was then combined with sodium silicate (Na_2SiO_3) and left at room temperature for 24 hours before being used. The experimental ratio of NaOH to Na_2SiO_3 used was 1:2.5.

iii. **Fine Aggregate**

The experiment utilized sharp river sand sourced from Zaria, Kaduna State, Nigeria, with a particle size of 4.75 mm.

iv. **Coarse Aggregate**

The experiment utilized coarse aggregate sourced from a quarry in Zaria, Kaduna State, Nigeria. The aggregate was obtained from crushed gravel with a particle size of 20 mm.

v. **Water**

The experiment employed purified water suitable for drinking purposes.

vi. **Magnesium Sulfate**

Water was mixed with magnesium sulfate salt to create a solution with a 2.5% magnesium sulfate concentration, simulating moderate exposure to the corrosive effects of magnesium sulfate. The preparation of the solution adhered to the procedure adopted by Neville (2004).

Table 1. Oxides of Metakaolin

Element	Concentration (%)
MgO	0.348%
Al_2O_3	36.509%
SiO_2	57.948%
P_2O_5	0.072%
SO_3	0.246%
Cl	0.015%
K_2O	1.420%
CaO	0.289%
TiO_2	1.058%
Cr_2O_3	0.006%
Mn_2O_3	0.038%
Fe_2O_3	2.033%
ZnO	0.007%
SrO	0.011%

2.2 Methods

2.2.1 Concrete Mix Proportioning

Since there is no standardized mix design procedure for geopolymer concrete, a series of trial MKGPC cubes was prepared using different material proportions. After curing for seven days, the cubes were tested for compressive strength, and the mix proportion that produced the optimum strength was selected for the final experimental program. The proportions of the materials are presented in **Table 2**.

2.2.2 Preparation of Samples

The metakaolin, fine, and coarse aggregates were thoroughly mixed, after which an alkaline solution was introduced and mixed vigorously for approximately 10 minutes until a consistent color was achieved, as depicted in Plate I. The freshly mixed MKGPC was then cast into a 100×100×100 mm steel mould in two layers, each layer was compacted using a poker vibrator, as illustrated in Plate II.

Table 2. Mix Proportions of Material for the Production of MKGPC

S/no	Material	Quantity (Kg/m^3)
1	Metakaolin	319
2	Fine aggregate	506
3	Coarse aggregate	1182
4	Sodium silicate	169
5	Sodium hydroxide	73
6	Water	3.19

Plate I: Mixing of MKGC

Plate II: Compaction of MKGC

Twenty-four hours after casting, the MKGC cubes were demolded, sealed in polyethylene sheets, and subjected to heat curing in an electric oven at 60 °C for 24 hours in compliance with the procedure outlined by Siciliano *et al.* (2024), as illustrated in Plate III. This curing regime was adopted to facilitate optimal geopolymerization of the MKGPC. The cubes were unwrapped and left to cool to room temperature in the laboratory, as shown in Plate IV. Subsequently, the MKGPC cubes were submerged in a 2.5% magnesium sulfate ($MgSO_4$) solution. At 7, 14, 28, and 56 days, the cubes were tested for compressive strength, ultrasonic pulse velocity, and rebound hammer tests, following the guidelines provided by (BS EN 12390-3:2009), (BS EN 12504-4:2004), and (BS EN 12504-2: 2001), respectively. Additionally, a water absorption test was conducted at 28 and 56 days, following the procedure outlined by (BS 1881-122:1983).

Plate III: MKGPC wrapped and placed in oven

Plate IV: MKGPC removed from the oven

3. Results and Discussion

3.1 Workability

Table 3 depicts the workability of the MKGPC mix, with a slump of 12mm indicating very low workability. This may be due to the molarity of the NaOH solution used in the experiment. Reddy (2010) made a similar observation, establishing that the workability of a geopolymer concrete mix decreases with higher molar concentrations of NaOH solution. This implies that MKGPC produced with a higher NaOH molar concentration will be difficult to mix or place in a formwork. Therefore, the utilization of water-reducing admixture may be required to improve the workability of the MKGPC mix.

Table 3. Slump of Metakaolin Geopolymer Concrete Mix

Slump Value	Slump range	Workability
12mm	0-25	Very low

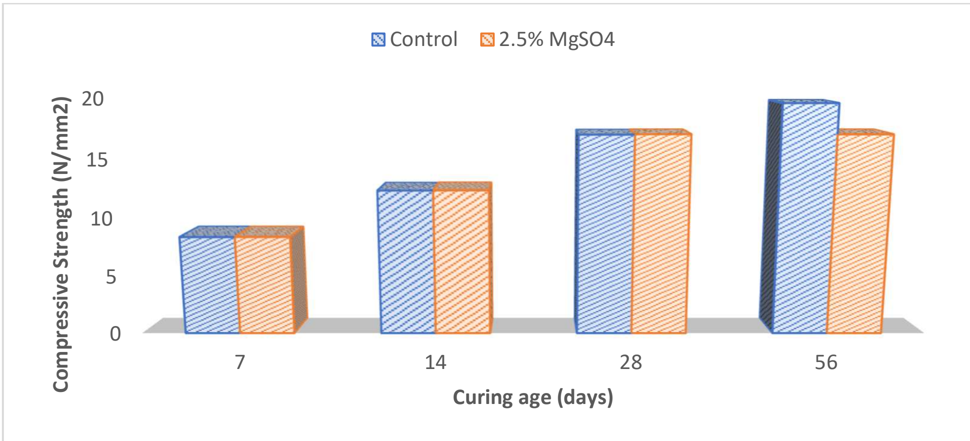

Figure 1. Compressive Strength of Metakaolin Geopolymer Concrete Samples

3.2 Compressive Strength

Figure 1 illustrates the compressive strength of MKGPC samples kept at room temperature (control samples) alongside those exposed to magnesium sulfate attack. It's noticeable that the compressive strength of samples subjected to magnesium sulfate slightly increased from 7 to 28 days, but began to decline by 56 days. At 28 days, the control sample achieved a compressive strength of 16.97N/mm², while the samples exposed to magnesium sulfate achieved 17N/mm², indicating a 0.18% increase in compressive strength compared to the control. However, at 56 days, the control samples reached a compressive strength of 19.55 N/mm², while the samples exposed to magnesium sulfate attained a compressive strength of 16.99 N/mm², indicating a 13.09% decrease in compressive strength compared to the control.

The improvement in compressive strength of MKGPC samples exposed to magnesium sulfate from 7 to 28 days may be due to the migration of magnesium sulfate salt into the microstructure of the MKGPC, filling voids and thereby enhancing compressive strength. However, the subsequent decrease in compressive strength at 56 days could be linked to the formation of gypsum and ettringite, accompanied by expansion in MKGPC and subsequent cracking.

Magnesium sulfate attack on cementitious materials involves a sequence of chemical reactions that lead to severe degradation. Initially, magnesium sulfate reacts with calcium hydroxide to produce gypsum and a surface layer of magnesium hydroxide (brucite). Although the brucite layer may temporarily reduce permeability, sulfate ions continue to diffuse inward, forming ettringite in zones of higher pH (Whittaker & Black, 2015). Magnesium sulfate attack on concrete occurs through a process involving both the sulfate (SO_4^{2-}) and magnesium (Mg^{2+}) ions. Unlike sodium sulfate, which mainly produces expansive ettringite, magnesium sulfate generates gypsum ($CaSO_4{\cdot}2H_2O$) and brucite ($Mg(OH)_2$) as primary reaction products. The Mg^{2+} ions react with calcium hydroxide, forming insoluble brucite, which destabilizes the calcium silicate hydrate (C–S–H) gel and causes severe decalcification (Zhang *et al.*, 2024). This degradation converts C–S–H into non-cementitious magnesium silicate hydrate (M–S–H), resulting in a loss of binding ability, surface softening, and structural disintegration.

Previous studies have reported trends consistent with the compressive strength behavior observed in this investigation. Elyammany *et al*. (2018) documented a similar pattern in metakaolin geopolymer concrete (MKGPC), where an initial strength gain was followed by strength deterioration attributed to micro-cracking caused by the formation of gypsum and ettringite. Similarly, Zang *et al.* (2020) found that increasing magnesium sulfate concentrations led to a decrease in compressive strength, highlighting the detrimental effects of sulfate attack on MKGPC durability. These findings support the hypothesis that while early-stage sulfate exposure may temporarily enhance strength by filling micro-voids, prolonged exposure promotes expansive chemical reactions that compromise the concrete's structural integrity.

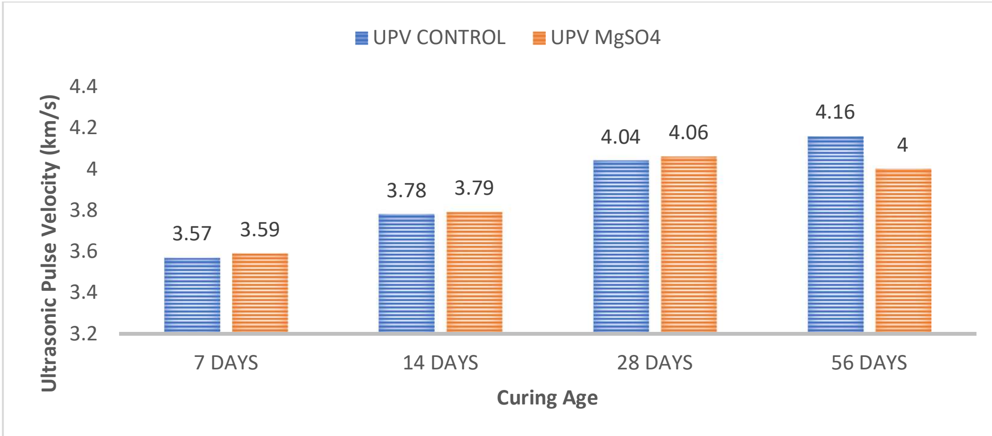

Figure 2. Ultrasonic Pulse Velocity of Metakaolin Geopolymer Concrete Samples

Table 4. IAEA Rating of Ultrasonic Pulse Velocity for Concrete (km/s)

Age (days)	Control Sample	2.5% $MgSO_4$	Remarks
7	3.57	3.59	Good
14	3.78	3.79	Good
28	4.04	4.06	Good
56	4.16	4.0	Good

3.3 Ultrasonic Pulse Velocity

Figure 2 depicts the ultrasonic pulse velocity of both control MKGPC and samples subjected to magnesium sulfate attack. The ultrasonic pulse velocity of samples exposed to magnesium sulfate showed a slight increase in ultrasonic pulse velocity from 7 to 28 days compared to the control samples. However, by 56 days, the ultrasonic pulse velocity started to decline. At 28 days, samples exposed to magnesium sulfate achieved an ultrasonic pulse velocity of 4.06 km/s, while the control samples attained 4.04 km/s, representing a 0.5% increase in ultrasonic pulse velocity for the samples exposed to magnesium sulfate. By 56 days, the control samples reached an ultrasonic pulse velocity of 4.16 km/s, whereas samples exposed to magnesium sulfate attained 4.0 km/s, representing a 3.85% decrease in ultrasonic pulse velocity compared to the control.

The ultrasonic pulse velocity (UPV) results in this study align with prior research on the durability assessment of MKGPC under sulfate attack. Similar to the compressive strength trend, the UPV of samples exposed to magnesium sulfate exhibited a slight increase between 7 and 28 days, likely due to the penetration of sulfate solution filling microstructural voids and enhancing density. However, after 56 days, a noticeable decline in UPV was observed, which corresponds with the formation of micro-cracks caused by expansive products such as gypsum and ettringite. This behavior concurs with the findings of Gosh et al. (2018), who reported that microstructural imperfections and voids slow wave propagation, leading to lower ultrasonic pulse velocities. Despite this decline, according to the International Atomic Energy Agency (IAEA) rating presented in **Table 4**, all MKGPC samples remained classified as being in good condition throughout the curing period.

3.4 Schmidt Rebound Hammer

Figure 3 illustrates the Rebound number for both control MKGPC samples and those exposed to magnesium sulfate degradation. The rebound number of both control and exposed samples increased from 7 to 28 days, however, by 56 days, the rebound number of the MKGPC samples subjected to sulfate attack began to decline.

At 7 days, the control MKGPC samples exhibited a rebound number of 10.5, while the MKGPC samples exposed to magnesium sulfate attained a rebound number of 11, representing a 4.5% increase in rebound number for the samples exposed to magnesium sulfate attack. However, by 56 days, the control MKGPC samples had a rebound number of 25, whereas the samples exposed to magnesium sulfate attack reached a rebound number of 20, indicating a 20% reduction for samples exposed to 2.5% magnesium sulfate attack.

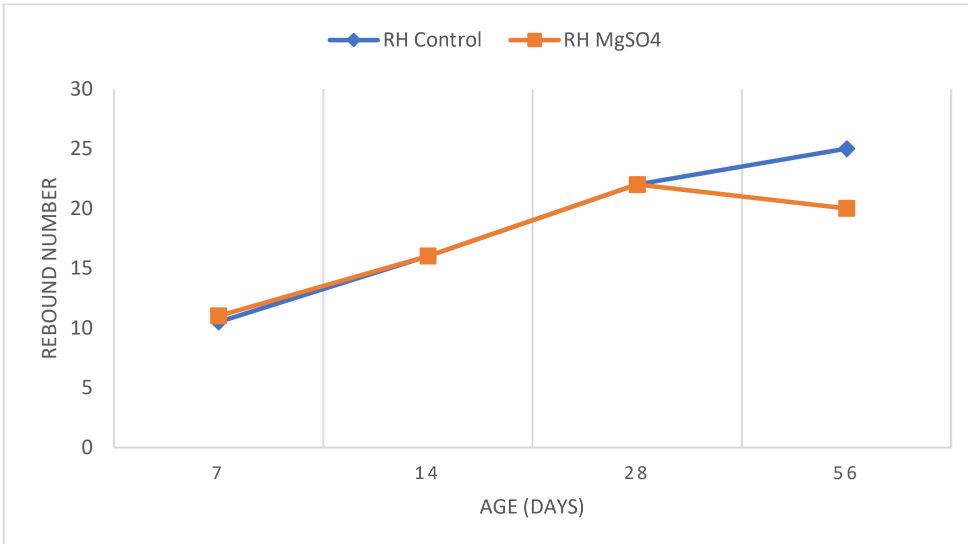

Figure 3. Schmidt Rebound Number of Metakaolin Geopolymer Concrete Samples

The rebound number results for MKGPC under magnesium sulfate attack exhibit trends consistent with compressive strength and ultrasonic pulse velocity observations. Both control and exposed samples showed increased rebound numbers from 7 to 28 days, likely due to sulfate diffusion filling microstructural voids and enhancing surface density. Additionally, the formation of white magnesium sulfate precipitates on the surface may contribute to increased surface hardness, further elevating rebound values. By 56 days, however, the rebound number of samples subjected to sulfate attack decreased by 20% compared to controls, reflecting the onset of micro-cracking and degradation. These findings align with Noori *et al.* (2021), who reported that fillers originating from source materials can increase the surface hardness of geopolymer concrete, thereby influencing the rebound number value.

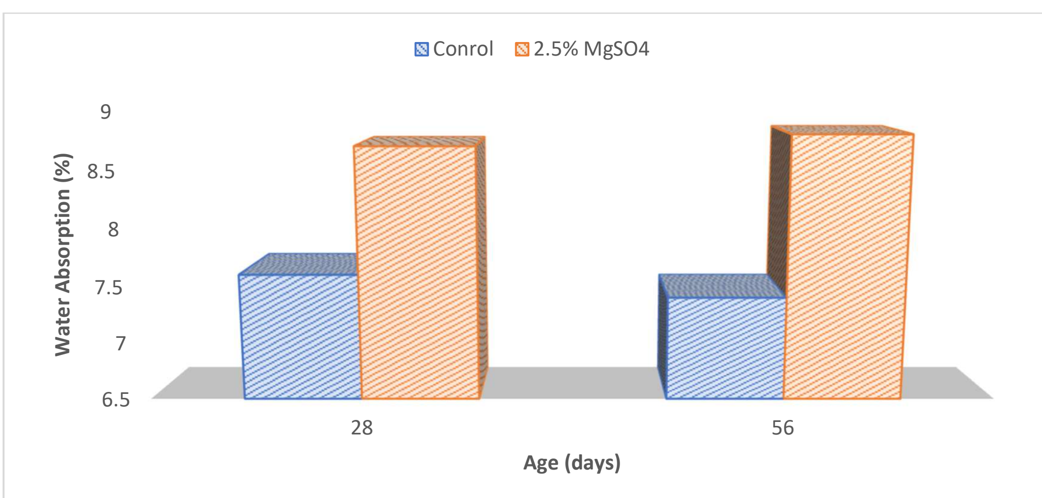

Figure 4. Water Absorption of Metakaolin Geopolymer Concrete Samples

3.5 Water Absorption

The water absorption test results, depicted in **Figure 4**, reveal different trends between control MKGPC samples and those exposed to magnesium sulfate attack. While the control samples exhibited a gradual decrease in water absorption over time, the samples subjected to sulfate attack showed a progressive increase, reaching 8.7% at 28 days and 8.8% at 56 days compared to 7.62% and 7.4% for the controls, respectively. This increase in water absorption is likely due to the development of internal micro-cracks caused by expansive sulfate reaction products such as gypsum and ettringite, which increased the porosity and permeability of the MKGPC samples exposed to 2.5% magnesium sulfate attack.

3.6 Correlation between Compressive Strength and Ultrasonic Pulse Velocity

Figure 5 demonstrates the correlation between compressive strength and ultrasonic pulse velocity of concrete samples exposed to magnesium sulfate deterioration. Notably, the coefficient of correlation (r^2) and adjusted r^2 were determined to be 0.98 and 0.94 respectively. This suggests a strong linear correlation between compressive strength and ultrasonic pulse velocity of MKGPC subjected to magnesium sulfate attack. Hence, it can be inferred that ultrasonic pulse velocity may be used for the prediction of the compressive strength of MKGPC exposed to magnesium sulfate attack, as indicated by Equation 1.

$$fc = 0.0512UPV + 3.1602 \quad (1)$$

Where:

fc = Compressive Strength

UPV = Ultrasonic Pulse Velocity

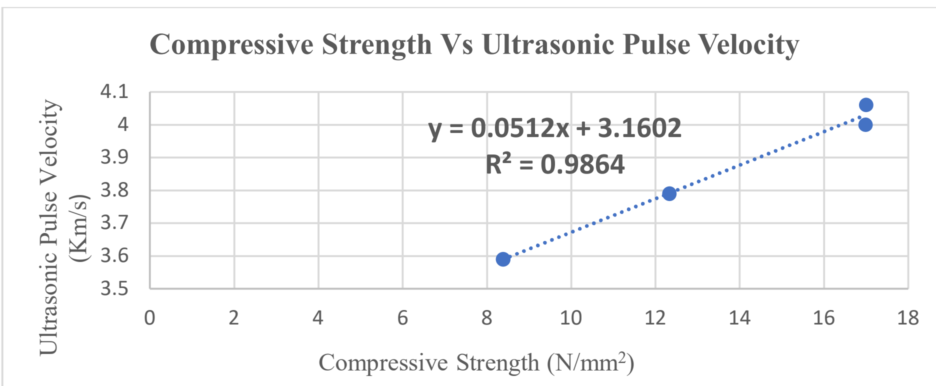

Figure 5. Correlation between Compressive Strength and Ultrasonic Pulse Velocity

3.7 Correlation between Compressive Strength and Rebound Number

Figure 6 illustrates the correlation between compressive strength and the rebound number of MKGPC samples exposed to magnesium sulfate deterioration. Notably, the coefficient of correlation (R^2) was calculated to be 0.97, while the adjusted R^2 was found to be 0.91. These values indicate a robust linear Correlation between compressive strength and the rebound number of MKGPC exposed to magnesium sulfate attack. Consequently, it can be inferred that the rebound number may be used for predicting the compressive strength of MKGPC subjected to magnesium sulfate attack, as presented by Equation 2.

$$fc = 1.1502RN + 1.521 \quad (2)$$

Where:

fc = Compressive Strength

RN = Rebound number

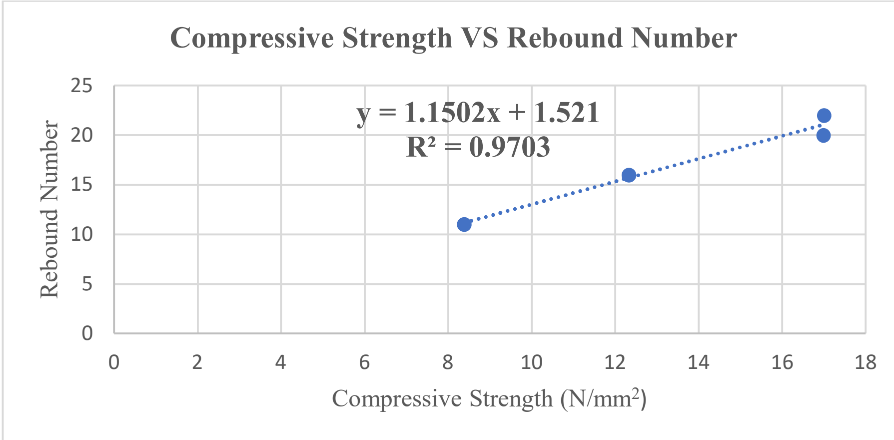

Figure 6. Correlation between Compressive Strength and Rebound Number

4. Conclusions

i. The MKGPC mix demonstrated very low workability, which could be attributed to the high molarity of the NaOH solution.
ii. Compressive strength of MKGPC samples exposed to 2.5% magnesium sulfate initially increased up to 28 days but declined significantly at 56 days due to sulfate-induced formation of gypsum and ettringite, causing micro-cracking.
iii. Ultrasonic pulse velocity and rebound number exhibited trends consistent with compressive strength, indicating initial microstructure densification followed by degradation after 2.5% magnesium sulfate exposure for 56 days.
iv. Water absorption increased in samples exposed to 2.5% magnesium sulfate, reflecting internal micro-crack development.
v. Strong linear correlations between compressive strength and both ultrasonic pulse velocity (r^2 = 0.98) and rebound number (r^2 = 0.97) indicate that these non-destructive tests are reliable predictors of compressive strength of MKGPC under 2.5% sulfate attack.

5. Recommendations

i. Further studies should be carried out by optimizing alkali activator concentration to enhance workability.
ii. Low-strength MKGPC should not be used for infrastructure in environments where magnesium sulfate concentration exceeds 2.5%
iii. Ultrasonic pulse velocity and rebound hammer tests could be used for routine, non-destructive monitoring of MKGPC exposed to magnesium sulfate environments.
iv. Other durability tests should be carried out to fully characterize the long-term magnesium sulfate resistance of MKGPC.

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