Uluslararası İleri Doğa Bilimleri ve Mühendislik Araştırmaları Dergisi Sayı 8, S. 29-35, 10, 2024 © Telif hakkı IJANSER'e aittir **Araştırma Makalesi**



International Journal of Advanced Natural Sciences and Engineering Researches Volume 8, pp. 29-35, 10, 2024 Copyright © 2024 IJANSER **Research Article**

https://as-proceeding.com/index.php/ijanser ISSN:2980-0811

Impact of Elevated Temperature and Salt Solutions on Mechanical Strength of Geopolymer Concrete

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(Received: 03 November 2024, Accepted: 16 November 2024)

(3rd International Conference on Contemporary Academic Research ICCAR 2024, 10-11 November 2024)

ATIF/REFERENCE: Safi, S. A., Raza, A., Hemat, R. A., Selmi, A. & Rahmani, M. U. R. (2024). Impact of Elevated Temperature and Salt Solutions on Mechanical Strength of Geopolymer Concrete, *International Journal of Advanced Natural Sciences and Engineering Researches*, 8(10), 29-35.

Abstract – This study examines the durability of recycled aggregate geopolymer concrete (GC) made with metakaolin (MK) and fly ash (FA) under sulfate and salt exposure following high-temperature conditions. Five FA-based GC mixtures with varying MK contents were prepared, cured at 90°C for 72 hours, then exposed to temperatures ranging from 200-800°C. Afterward, samples were immersed in 5% sodium sulfate (SS) and 5% sodium chloride (SC) solutions for 30 days. MK improved compressive strength, with a 37.49% loss at 200°C and only 3.22% loss at 400°C + SS, while enhancing flexural strength. These results underscore MK's role in enhancing durability and mechanical properties.

Keywords – Geopolymer, Recycled Aggregates, Elevated Temperature, Compressive Strength, Flexural Strength.

I. INTRODUCTION

Cement production significantly contributes to global CO2 emissions [1]. Due to the environmental challenges, health risks, and resource depletion associated with cement, sustainable materials have gained attention [2]. Eco-friendly binders such as perlite, pumice, metakaolin (MK), volcanic ash, silica fume, and fly ash (FA) are widely used. Among these, FA and MK are notable precursors in producing durable geopolymer concrete (GC) [3-5]. Using Na2SO3 and NaOH enhances GC durability and strength due to high aluminosilicate dissolution, forming concentrated gels [6, 7]. Curing methods, duration, and temperature also affect GC strength, with high-temperature curing accelerating early-age strength development [8]. GC, formed through acidic or alkaline reactions with aluminosilicate materials, has been shown to outperform standard concrete in durability [9]. However, the performance of FA/MK-based GC under high temperatures and sodium chloride exposure remains less explored compared to conventional composites, requiring further analysis. This study examines FA-based GC with varying MK contents, first subjected to high temperatures, then exposed to sodium sulfate and sodium chloride solutions, assessing internal structural changes and mechanical properties.

II. MATERIALS AND METHOD

i. Materials

To produce the geopolymer concrete (GC), fly ash (FA) and metakaolin (MK) were used as supplementary cementitious materials, with their chemical compositions detailed in Table 1. The apparent densities of MK and FA were approximately 2.59 g/cm³ and 2.15 g/cm³, respectively. Class F FA was selected for its excellent reactivity with alkali, while MK was incorporated to enhance both thermal and chemical resistance. A fixed solution ratio was maintained by using an alkaline mixture of Na2SiO3 and NaOH, with the ratio set at 2.

Table 1. Composition of FA and MK.		
Component (% mass)	Fly ash	Metakaolin
Silica (SiO ₂)	57.94	53.07
Alumina (Al ₂ O ₃)	20.54	37.24
Iron oxide (Fe ₂ O ₃)	8.24	3.36
Calcium oxide (CaO)	5.23	0.98
Magnesium oxide (MgO)	3.16	1.65
Potassium oxide (K ₂ O)	1.48	1.29
Sodium oxide (Na ₂ O)	1.08	0.09
Titania (TiO ₂)	-	1.32
Sulfur trioxide (SO ₃)	0.16	-
Loss on ignition (LOI)	2.17	1.0

The recycled coarse aggregate (RCA), with a particle size of 4-19 mm and a dry density of 2.67 g/cm³, was used at a proportion of 25%, while fine aggregates, sized 0-4 mm with a density of 2.62 g/cm³, made up the remaining 75%. The RCA was sourced from crushed concrete cylinders used in commercial testing, aged less than one year. To achieve the desired workability, 2% of the total binder was supplemented with Sika ViscoCrete®, which contains naphthalene as its main component.

ii. Specimen Preparation and Testing

Five distinct mix designs with varying metakaolin (MK) proportions were made including 0%, 20%, 40%, 60%, and 80% MK by mass of fly ash (FA). The blends are labeled as F-100, FMK-80, FMK-60, FMK-40, and FMK-20, indicating 100%, 80%, 60%, 40%, and 20% FA content, respectively. The total binder content of FA and MK was 600 kg/m³. Due to the exothermic nature of the reaction, the NaOH solution was prepared 24 hours prior to casting the geopolymer concrete. Raw materials and NaOH were mixed for 2 minutes, followed by Na₂SiO₃ for an additional 2 minutes, and finally, 2% additives and aggregates were mixed for another 2 minutes.

III. RESULTS AND DISCUSSION

i. Compressive Strength

Figure 1 shows the compressive strength (CS) of specimens exposed to heat and sodium sulfate (SS). As the metakaolin (MK) content increased, all blends exhibited a significant rise in CS. This enhancement can be attributed to the refinement and densification of the microstructure due to the increased presence of MK-sourced calcined material. Rich in CaO, MK provides highly soluble calcium, which directly contributes to higher CS. A study [10] highlighted the role of C-A-S-H gel in reducing void volume, resulting in a denser microstructure compared to C-S-H and N-A-S-H gels in FA-based geopolymer concrete with MK.



Figure 1. CS values of specimens for elevated temperatures subjected to SS and comparison with 28-days CS

With the addition of MK, a hybrid gel system with enhanced features was observed in the matrix, and the presence of soluble calcium further improved mechanical strength by generating additional C-A-S-H gel. Consequently, increased temperature led to a reduction in compressive strength (CS) across all test blends, but MK-rich geopolymer concrete (GC) showed relatively less CS loss at high temperatures. This is attributed to the reduced voids in MK-containing geopolymers, which also lowers permeability, porosity, and shrinkage. Blends heated to around 200°C, including FMK-20, FMK-40, FMK-60, FMK-80, and F-100, showed CS losses of 11.65%, 13.65%, 16.58%, 18.59%, and 37.49%, respectively. At higher temperatures and after exposure to 5% sodium sulfate (SS), the specimens only exhibited a reduction in strength. Compared to conventional concrete, GC's response to sulfate attack differs due to the varied reaction products, with GC generally showing less degradation and scaling. For instance, sulfate exposure results in less damage and minor scaling compared to traditional concrete [11]. Additionally, GC subjected to sulfate solutions showed improved strength over samples exposed to only water, with strength increasing with higher sodium-sulfur concentrations [12]. After 200°C and SS, the CS reductions for FMK-20, FMK-40, FMK-60, FMK-80, and F-100 were 5.39%, 3.93%, 3.48%, 6.48%, and 8.33%, respectively, mainly due to microcracking and shrinkage. Beyond 400°C and SS, these reductions were mitigated, with improvements of 4.31%, 7.62%, 8.35%, 6.27%, and 3.22%, respectively.

At 800°C, CS significantly decreased due to hydration effects, which also affected C-A-S-H stability, making the matrix more susceptible to deterioration similar to C–S–H [13]. Compared to other blends with only fly ash, incorporating FA/MK compromises thermal stability, resulting in significant strength degradation at high temperatures [14]. The strength gains after 600°C and 800°C exposure were 0.39%, 0.75%, 0.69%, 1.86%, and 2.65% for FMK-20, FMK-40, FMK-60, FMK-80, and F-100, respectively. FMK-20 specimens achieved peak strengths of 34.28 MPa and 23.97 MPa after exposure to 600°C and 800°C, respectively. Continuous sodium sulfate crystallization and hydration in GC slightly reduced porosity. The behavior of the ratio at temperatures near 600°C and 800°C showed variability, adding complexity to the heterogeneity of the geopolymer mortars.

Figure 2 illustrates the CS variations of test specimens subjected to sodium chloride (SC) after heating at 200°C, 400°C, 600°C, and 800°C. For instance, the CS of FMK-20, FMK-40, FMK-60, FMK-80, and FS100 blends increased by 2.43%, 5.39%, 1.97%, 4.49%, and 3.28% after 400°C and SC, and by 3.25%, 1.53%, 0.29%, 1.01%, and 0.17% after 200°C and SC, respectively. Higher MK proportions led to a notable decline in CS loss, with 40% and 20% MK blends showing about a 10% drop in 28-day CS after 200°C, although other blends experienced further CS reductions.



Figure 2. CS values of specimens for elevated temperatures subjected to SC and comparison with 28-days CS

Figure 2 shows that the compressive strength (CS) of all blends increased after exposure to both $600^{\circ}C + SC$ and $800^{\circ}C + SC$. The rate of increase was consistent across blends, except for the FMK-20 blend, which exhibited the highest CS. Several factors affect concrete's resistance to chloride environments, with the CaO ratio and the reactivity of raw materials being crucial. A slight increase in CaO proportion enhances the blend's reactivity and boosts CS, likely due to the formation of extensive N-A-C-H or C-A-S-H gels that reduce porosity and chloride penetration. This process minimizes ion diffusion within the geopolymer concrete structure. Additionally, the adsorption and binding properties of the concrete significantly impact the diffusion and transfer of chloride ions [15]. Overall, the durability applied showed minimal to no impact on the 30-day concrete characteristics after high temperature + SC exposure.

ii. Flexural Strength

Figure 3 depicts the flexural strength of specimens exposed to heat and sulfate solutions. Increasing the Increased metakaolin (MK) content resulted in substantial improvements in flexural strength across all blends, attributed to microstructural refinement and densification from the additional calcined MK material [9, 16]. Research emphasizes the role of C-A-S-H gel in enhancing microstructure density, making MK-blended fly ash (FA)-based geopolymers superior to traditional C-S-H and N-A-S-H gels [10]. The addition of MK created a hybrid gel system with advanced characteristics in the matrix, enhancing mechanical strength through increased C-A-S-H formation [17, 18]. However, high temperatures caused significant reductions in flexural strength by decomposing hydration products in MK-FA blends, though MK-rich specimens experienced less strength loss due to reduced voids and improved impermeability, porosity, and shrinkage resistance. For example, specimens FMK-20, FMK-40, FMK-60, FMK-80, and F-100 showed respective flexural strength losses of 14.23%, 12.86%, 17.47%, 19%, and 40.65% after heating to approximately 200°C.



Figure 3. Flexural strength values of specimens for elevated temperatures subjected to SS and comparison with 28-days strength

Comparatively, sulfate attack affects geopolymers differently than conventional concrete due to differences in reaction products and lower Ca content, which enhance sulfate resistance. Geopolymer concrete (GC) exposed to sulfate solutions showed minimal degradation and scaling compared to traditional concrete [11]. Studies revealed sodium sulfate crystal formation without expansive products, indicating inert interactions that are vital for stability. Strength in sodium sulfate solutions improved with higher concentrations, suggesting ongoing GC hydration and sulfate crystal formation.

After temperatures exceeding 400°C, MK-blended GC experienced thermal degradation affecting C-A-S-H stability, compromising strength more than FA-only blends [14]. However, MK-containing blends showed stability with minimal flexural strength reduction after 400°C and subsequent sulfate exposure. For instance, FMK-20, FMK-40, FMK-60, FMK-80, and F-100 blends exhibited flexural strength reductions of 5.94%, 5.78%, 5.16%, 5.98%, and 6.12% after 200°C followed by sulfate exposure. At 600°C and 800°C, these blends showed strength gains of 0.31% to 2.32%, attributed to sulfate ion penetration promoting crystal growth via geopolymerization. At 800°C, flexural strength reduction intensified due to hydration effects and compromised C-A-S-H stability, affecting overall matrix durability similar to C-S-H degradation [13]. Despite thermal challenges, MK-FA blends showed comparable outcomes post-400°C and sulfate exposure, highlighting the stabilizing effects of MK on GC microstructures [14].

Figures 4 illustrate flexural strength variations under simultaneous chloride exposure after tempering from 200°C to 800°C. Higher MK proportions correlated with reduced flexural strength loss, emphasizing enhanced reactivity and gel formation (N-A-C-H or C-A-S-H), which are crucial for mitigating chloride ingress and diffusion within GC structures. Improved durability was evident with minimal or no adverse impacts on 30-day concrete properties after high-temperature chloride exposure.



Figure 4. Flexural strength values of specimens for elevated temperatures subjected to SC and comparison with 28-days strength

IV. CONCLUSION

This study aimed to evaluate the influence of SC and SS on FA and MK-based GC features after exposure to elevated temperatures. MK's role in refining the microstructure and providing soluble CaO significantly boosts CS, with specimens showing up to 37.49% CS loss at 200°C. At higher temperatures, MK-rich geopolymers demonstrate superior resistance, with losses reduced to 3.22% at 400°C + SS. Similarly, flexural strength benefits from MK's inclusion, showing reductions of 40.65% at 200°C. These findings underscore MK's efficacy in mitigating thermal and chemical degradation, enhancing the durability and mechanical properties of geopolymers under harsh environmental conditions.

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