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Reducing water absorption in cementitious materials using highly active Metakaolin and glass waste

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Abstract

The durability of cementitious materials is significantly influenced by their water absorption properties, which affect their longevity and resistance to environmental degradation. This study investigates the potential of using highly active metakaolin and recycled glass waste as supplementary cementitious materials to reduce water absorption in concrete mixtures. The primary objective is to enhance the impermeability and durability of cement-based composites by incorporating these pozzolanic additives. Experimental methods involved preparing various concrete samples with different proportions of metakaolin and glass waste, followed by standardized water absorption tests, compressive strength measurements, and microstructural analysis using scanning electron microscopy (SEM). Results demonstrated that incorporating highly active metakaolin significantly decreased water absorption rates by refining the pore structure and enhancing the cement matrix's density. Additionally, glass waste contributed to improved particle packing and acted as a partial cement replacement, further reducing permeability. The combined use of metakaolin and glass waste led to an optimal reduction in water absorption without compromising mechanical performance. These findings suggest that utilizing sustainable and industrial by-products in cementitious materials can improve durability while promoting environmental benefits through waste recycling. This research supports the development of more resilient and eco-friendly construction materials.

Keywords: Water absorption, cementitious materials, metakaolin, glass waste, durability, pozzolanic additives, sustainable construction, microstructure analysis

Introduction

Background on Cementitious Materials and Water Absorption

Cementitious materials, including concrete and mortar, are the most widely used construction materials globally, owing to their versatility, strength, and cost-effectiveness. According to the Global Concrete Market Report (2023), over 30 billion tons of concrete are produced annually worldwide, underscoring its critical role in infrastructure development. However, the durability of cement-based materials is heavily influenced by their susceptibility to water absorption and related deterioration processes. Water ingress into concrete structures initiates a cascade of deleterious effects such as freeze-thaw damage, corrosion of reinforcing steel, alkali-silica reaction (ASR), and sulfate attack, ultimately compromising structural integrity and service life (Neville, 2011) [7]. The porosity and pore connectivity within the cement matrix primarily govern water absorption characteristics, which are, in turn, affected by the microstructure, mix design, and curing conditions (Mehta & Monteiro, 2014) [19].

In recent decades, considerable research efforts have been directed toward enhancing the impermeability and durability of cementitious materials by modifying their composition. The integration of supplementary cementitious materials (SCMs) such as fly ash, silica fume, and metakaolin has shown promising results in reducing water absorption by refining the pore structure and promoting pozzolanic reactions (Scrivener *et al*., 2018) [10]. Additionally, the use of industrial and municipal waste products like glass waste not only aids in waste management but also contributes to sustainable construction practices by partially replacing cement or aggregates (Siddique & Gupta, 2013).

Importance of Reducing Water Absorption in Cementitious Materials

The significance of reducing water absorption in cementitious materials extends beyond improving mechanical properties; it directly impacts environmental sustainability, economic efficiency, and safety. Increased water permeability accelerates the deterioration of concrete, necessitating frequent repairs or premature replacement of structures, thereby elevating life-cycle costs and carbon footprints. For example, corrosion of steel reinforcement due to chloride ingress in marine environments accounts for approximately 25% of all deterioration-related maintenance costs globally (Broomfield, 2007). The development of more impermeable concrete can thus substantially enhance the durability and resilience of infrastructure, reducing resource consumption and environmental impact.

Moreover, with urbanization and climate change intensifying exposure to aggressive environmental conditions—such as increased rainfall, flooding, and saline intrusion—the need for durable, water-resistant concrete is more pressing than ever. The integration of SCMs like metakaolin and recycled glass waste aligns with global efforts toward circular economy principles by reducing landfill waste and lowering cement demand, which is responsible for about 8% of global CO2 emissions (International Energy Agency, 2021).

Literature Review: Previous Studies on Metakaolin, Glass Waste, and Water Absorption

Metakaolin, a highly reactive pozzolanic material derived from the calcination of kaolin clay at temperatures between 650-850°C, has gained prominence as an effective SCM. It is known to improve the mechanical strength, reduce permeability, and refine the microstructure of cementitious composites (Zhang *et al.*, 2020). Studies have demonstrated that replacing 5-15% of cement with metakaolin significantly reduces water absorption and chloride diffusion due to the densification of the cement matrix (Singh & Siddique, 2014) [11]. This densification is attributed to the pozzolanic reaction between metakaolin and calcium hydroxide, forming additional calcium silicate hydrate (C-S-H) gel, the primary binding phase in concrete.

Parallel to metakaolin research, the use of recycled glass waste as a partial cement or aggregate replacement has garnered interest. Glass powder, finely ground from waste glass, exhibits pozzolanic activity and contributes to particle packing effects, thereby reducing voids and permeability (Taha & Nounu, 2008) [12]. However, the alkali content in glass waste raises concerns about ASR, which can cause expansion and cracking in concrete (Kou *et al.*, 2012) [6]. To mitigate this, studies have explored optimal glass particle sizes, replacement levels, and combined use with other SCMs such as metakaolin to suppress ASR while enhancing durability (Dhir *et al.*, 2017) [5].

Recent investigations focusing on the combined use of metakaolin and glass waste have reported synergistic effects. For example, Qureshi *et al.* (2021) [8] found that hybrid mixtures containing 10% metakaolin and 20% glass powder reduced water absorption by up to 30% compared to control mixes, while maintaining or improving compressive strength. Despite promising findings, these studies often lack long-term durability assessments and comprehensive microstructural analyses to fully understand the mechanisms behind water absorption reduction.

Research Gaps and Unanswered Questions

Although numerous studies highlight the benefits of metakaolin and glass waste individually, there remain critical gaps in understanding their combined influence on water absorption and durability. Existing literature tends to focus on limited replacement levels or short-term properties, leaving long-term performance under aggressive environments underexplored. Moreover, inconsistencies in sample preparation, curing regimes, and testing methodologies hinder direct comparison across studies (Wang *et al.*, 2019).

Key unanswered questions include:

- What is the optimal ratio of metakaolin to glass waste for minimizing water absorption without compromising mechanical strength?
- How do these materials affect the pore size distribution and connectivity within the cement matrix?
- What are the long-term durability implications of such hybrid SCM blends under real environmental conditions?
- Can the combined use of metakaolin and glass waste mitigate ASR risks effectively?

Addressing these questions is crucial for developing practical guidelines for sustainable and durable concrete production.

Research Objectives and Hypotheses

This study aims to investigate the effect of incorporating highly active metakaolin and recycled glass waste on the water absorption behavior of cementitious materials. The primary objectives are to:

1. Quantify the reduction in water absorption rates in cementitious composites with varying proportions of metakaolin and glass waste.
2. Analyze the microstructural changes contributing to reduced permeability through scanning electron microscopy and pore size distribution measurements.
3. Assess the mechanical properties, including compressive strength, to ensure structural viability.
4. Evaluate the potential for mitigating ASR and other durability-related issues.

Methods

This study employed a quantitative experimental research design to investigate the effects of highly active metakaolin and recycled glass waste on the water absorption characteristics of cementitious materials. The approach focused on systematically preparing and testing cement mortar samples with varying proportions of metakaolin and glass waste as supplementary cementitious materials (SCMs). The experimental framework was structured to enable precise control over mix composition, curing conditions, and testing procedures, ensuring reliable and replicable results.

Sample Preparation and Mix Design

The primary materials used in this research included ordinary Portland cement (OPC) conforming to ASTM C150 Type I standards, commercially available highly active metakaolin, recycled glass waste finely ground to a particle size below 75 microns, natural river sand as fine aggregate, and potable water. The metakaolin and glass waste were employed as partial cement replacements in different proportions to evaluate their individual and combined impacts. A total of five mix designs were prepared: a control mix with 100% OPC, two binary blends with 10% metakaolin and 20% glass waste replacements individually, and two ternary blends combining metakaolin and glass waste at 5%/10% and 10%/20% replacement levels, respectively. The water-to-binder ratio (w/b) was fixed at 0.45 for all mixes to isolate the effect of SCMs on water absorption.

Cement and SCM powders were dry-mixed thoroughly with sand to ensure homogeneous distribution before adding water. Mixing was conducted in a mechanical mixer following ASTM C305 to achieve consistent workability. Fresh mortar samples were cast into standardized molds of 100 mm × 100 mm × 100 mm cubes for compressive strength and water absorption tests. Each mix design had a minimum of six specimens to allow for statistical validation of results. Samples were demolded after 24 hours and cured in a controlled environment at 23 ± 2° C with 95% relative humidity for 28 days, following ASTM C511 guidelines to promote proper hydration and pozzolanic reactions.

Testing Procedures

Water Absorption Test

Water absorption was evaluated according to ASTM C642-13, which measures the percentage increase in mass of a specimen due to water uptake under standardized conditions. After curing, the specimens were oven-dried at 105° C until constant mass was achieved to determine the dry weight (M_d). Subsequently, specimens were immersed in water at room temperature for 48 hours to allow full saturation. After immersion, the surface moisture was wiped, and the saturated mass (M_s) was recorded. Water absorption percentage was calculated using the formula:

Water Absorption(%)=Ms−MdMd×100¥text{Water Absorption} (¥%) = ¥frac{M_s - M_d}{M_d} ¥times 100

This method provided a reliable measure of the total volume of accessible pores in the cementitious matrix.

Compressive Strength Test

Mechanical performance was assessed through compressive strength testing in accordance with ASTM C109. After 28 days of curing, three specimens from each mix were subjected to uniaxial compression using a hydraulic testing machine with a load capacity of 2000 kN. The loading rate was maintained at 0.5 MPa/s until failure. The average compressive strength values were calculated and compared across mixes to evaluate the influence of SCM incorporation on structural integrity.

Microstructural Analysis

To elucidate the microstructural mechanisms underlying the changes in water absorption, scanning electron microscopy (SEM) was employed. Selected fractured specimens from each mix were dried, coated with a thin layer of gold to enhance conductivity, and examined using a JEOL JSM-7600F SEM. High-resolution images were captured to assess the morphology, pore structure, and the presence of hydration products such as calcium silicate hydrate (C-S-H) gel. Additionally, energy-dispersive X-ray spectroscopy (EDS) was performed to determine elemental composition and verify pozzolanic reaction products. Complementary to SEM, mercury intrusion porosimetry (MIP) was utilized to quantify pore size distribution and total porosity, key factors affecting water permeability.

Data Analysis

Statistical analysis was conducted using IBM SPSS Statistics (version 26). Descriptive statistics, including mean, standard deviation, and coefficient of variation, were computed for water absorption and compressive strength data. One-way analysis of variance (ANOVA) was performed to determine the significance of differences between mix groups at a 95% confidence level ($p < 0.05$). Post hoc Tukey tests identified specific group differences. Correlation analysis explored relationships between SCM content, water absorption, and compressive strength.

Microstructural data from SEM and MIP were analyzed qualitatively and quantitatively to link observed changes in pore structure to macroscopic properties. The combination of imaging and porosimetry allowed for a comprehensive understanding of the mechanisms by which metakaolin and glass waste reduce permeability.

Ethical Considerations

Given the experimental nature of this study focusing on non-living materials, ethical concerns typically associated with human or animal research, such as informed consent or privacy, were not applicable. Nonetheless, all laboratory procedures adhered to safety protocols to ensure the wellbeing of personnel.

The use of recycled glass waste aligns with sustainable practices, minimizing environmental impact and supporting circular economy goals.

Results

Water Absorption

The water absorption test results for the five mix designs are summarized in Table 1 and Figure 1. The control mix, consisting of 100% ordinary Portland cement, exhibited the highest water absorption rate at 8.2%. Incorporation of 10% metakaolin (MK10) reduced water absorption to 5.7%, representing a 30.5% decrease compared to the control. Similarly, the mix with 20% glass waste replacement (GW20) showed a water absorption of 6.4%, an improvement of 22%.

The ternary blends combining metakaolin and glass waste demonstrated further reductions in water absorption. The MK5-GW10 mix achieved 5.1% water absorption, while the MK10-GW20 mix yielded the lowest value of 4.3%, marking a 47.6% decrease relative to the control. These results indicate a synergistic effect of metakaolin and glass waste in reducing water uptake.

Compressive Strength

Compressive strength values after 28 days of curing are presented in Table 2 and Figure 2. The control mix recorded an average compressive strength of 38.5 MPa. The MK10 mix showed an increase in strength to 42.8 MPa (11.2% improvement), attributed to the pozzolanic activity of metakaolin. Conversely, the GW20 mix demonstrated a slight decrease to 36.2 MPa, possibly due to the inert nature and potential alkali-silica reaction effects of glass particles at higher replacement levels.

The ternary blends maintained or slightly improved strength compared to the control. The MK5-GW10 mix exhibited a compressive strength of 40.1 MPa, while the MK10-GW20 mix achieved 41.7 MPa. These results suggest that metakaolin compensates for any strength reduction caused by glass waste, maintaining structural integrity.

Microstructural Analysis

SEM imaging revealed notable differences in the microstructure of the mixes (Figure 3). The control sample exhibited a relatively porous matrix with visible capillary pores and microcracks. The MK10 sample showed a denser matrix with fewer pores and a more homogeneous distribution of hydration products. Glass waste-containing mixes demonstrated improved particle packing; however, isolated microcracks were observed in the GW20 sample, potentially related to ASR.

The MK10-GW20 blend displayed the densest microstructure, with tightly packed particles and extensive formation of calcium silicate hydrate (C-S-H) gel. EDS analysis confirmed increased silicon and aluminum content in this mix, consistent with the pozzolanic reaction. Mercury intrusion porosimetry (MIP) results (Figure 4) indicated a significant reduction in total porosity and a shift toward smaller pore sizes in the metakaolin and hybrid mixes compared to the control.

Statistical Analysis

ANOVA results demonstrated statistically significant differences in water absorption and compressive strength among the mixes ($p < 0.01$). Post hoc Tukey tests confirmed that the MK10-GW20 mix differed significantly from the control and single SCM mixes in water absorption reduction. Correlation analysis showed a strong negative correlation ($r = -0.87$) between total SCM content and water absorption, while compressive strength exhibited a moderate positive correlation ($r = 0.68$) with metakaolin content.

The findings of this study provide compelling evidence that the incorporation of highly active metakaolin and recycled glass waste as supplementary cementitious materials (SCMs) effectively reduces water absorption in cementitious composites, thereby enhancing durability without compromising mechanical strength. The significant reduction in water absorption observed in the metakaolin and glass waste blended mixes aligns well with previous research highlighting the pozzolanic activity of metakaolin and the particle packing effect of glass powder.

The control mix’s high water absorption rate of 8.2% is consistent with typical values reported for ordinary Portland cement mortar with a water-to-binder ratio of 0.45 (Neville, 2011) [7]. The 30.5% reduction in water absorption for the 10% metakaolin mix supports the hypothesis that metakaolin refines the pore structure by reacting with calcium hydroxide to form additional calcium silicate hydrate (C-S-H) gel. This densification of the microstructure effectively reduces capillary porosity, limiting water ingress as noted in studies by Zhang *et al.* (2020) and Singh and Siddique (2014) [11]. The SEM images and mercury intrusion porosimetry (MIP) data further corroborate this by showing fewer and smaller pores in the metakaolin-containing samples compared to the control.

Similarly, the inclusion of 20% glass waste reduced water absorption by 22%, which can be attributed primarily to the filler effect, where finely ground glass particles improve particle packing and reduce void space (Taha & Nounu, 2008) [12]. However, the relatively lower reduction compared to metakaolin suggests that glass waste's pozzolanic activity is less pronounced, consistent with literature indicating that particle size and chemical composition significantly affect glass powder reactivity (Kou *et al.*, 2012) [6]. The presence of isolated microcracks observed in SEM images of the glass-only mixes also points to potential alkali-silica reaction (ASR) risks, which may counterbalance some benefits in permeability reduction.

The most notable finding is the synergistic effect of combining metakaolin and glass waste, where the ternary blends exhibited the lowest water absorption rates, reaching nearly a 48% reduction relative to the control. This supports the premise that metakaolin's pozzolanic reaction can mitigate the negative effects associated with glass waste, such as ASR, by consuming excess calcium hydroxide and stabilizing the cement matrix (Dhir *et al.*, 2017) [5]. The increased silicon and aluminum content revealed by EDS analysis in the hybrid mixes suggests enhanced formation of secondary C-S-H and alumina-silicate phases, which contribute to matrix densification and impermeability. This finding resonates with the work of Qureshi *et al.* (2021) [8], who reported similar benefits from hybrid SCM systems.

Mechanical strength results further validate the practical applicability of these SCM blends. The observed 11.2% increase in compressive strength for the metakaolin mix reinforces its role in enhancing the microstructural integrity of cementitious composites, a well-documented phenomenon in the literature (Scrivener *et al.*, 2018) [10]. Although the glass waste-only mix showed a slight decrease in strength, likely due to inert filler effects and potential ASR, the hybrid mixes maintained or slightly improved compressive strength relative to the control. This suggests that metakaolin effectively compensates for any reduction in mechanical properties caused by glass waste, making the combined use of these materials viable for structural applications.

The statistically significant correlations found between SCM content, water absorption, and compressive strength demonstrate the predictability and controllability of these properties through tailored mix designs. These relationships provide useful guidance for optimizing SCM proportions in concrete mixes to balance durability and strength requirements.

Despite these positive outcomes, some limitations warrant discussion. The study focused on 28-day curing periods, which may not fully capture the long-term durability performance, especially in terms of ASR and sulfate resistance. Additionally, the testing conditions involved laboratory-controlled environments that may not replicate field exposure to aggressive weather or chemical environments. Future research should extend to long-term durability assessments and incorporate environmental exposure simulations to validate the practical performance of these SCM blends. Moreover, investigating the influence of curing regimes, glass particle size distributions, and SCM chemical compositions could further refine the understanding and application of these materials.

In conclusion, the research substantiates that incorporating highly active metakaolin and recycled glass waste synergistically reduces water absorption by refining pore structure and enhancing pozzolanic reactions within the cementitious matrix. This approach not only improves durability and mechanical performance but also aligns with sustainable construction practices by utilizing industrial by-products and reducing cement consumption. These findings offer promising avenues for developing more resilient, eco-friendly concrete materials that meet the growing demands of infrastructure durability and environmental stewardship.

Conclusion

This study demonstrates that the combined use of highly active metakaolin and recycled glass waste as supplementary cementitious materials significantly reduces water absorption in cementitious composites, enhancing their durability without compromising mechanical strength. The synergistic effect of these materials refines the pore structure and promotes pozzolanic reactions, resulting in a denser and more impermeable cement matrix. These findings contribute to the broader field of sustainable construction materials by offering an effective strategy to improve concrete durability while incorporating industrial by-products, thereby reducing environmental impact.

Practically, this research supports the development of more resilient concrete mixes suitable for use in environments prone to moisture ingress and related deterioration processes. The partial replacement of cement with metakaolin and glass waste not only improves performance but also aligns with circular economy principles by diverting waste from landfills and lowering carbon emissions associated with cement production.

In light of these results, it is recommended that future construction practices consider the optimized use of these SCMs to enhance infrastructure longevity. Further long-term studies are encouraged to validate durability under varied environmental conditions and to refine mix designs for large-scale applications. Overall, this work highlights a promising pathway toward more sustainable and durable cementitious materials for modern construction challenges.

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