

Degradation mechanisms of bio-enhanced geopolymers for acid-resistant sewer infrastructure

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Abstract

The long-term deterioration of sewer infrastructure in acidic environments presents a significant challenge for water utilities, as traditional cementitious materials tend to degrade rapidly when exposed to biogenic and industrial acids. To address this, this study investigates the durability of fly ash-based geopolymer concrete (GPC) reinforced with *Shewanella* bacteria, subjected to controlled sulphuric acid (H_2SO_4) immersions at concentrations of 0%, 3%, 5%, and 10% (by volume) over a duration of 60 days. A comprehensive experimental framework, including pH monitoring, mass change evaluation, capillary absorption tests, chloride migration analysis, permeability measurements, SEM/EDS, XRF, XRD, and FTIR spectroscopy, was employed to characterise chemical, mineralogical, and microstructural modifications. Results show that bio-modified GPC exhibited consistently improved performance under mild to moderate acidity, associated with enhanced Ca retention, reduced transport coefficients, and partial pore refinement, supporting a biomineralisation hypothesis pending direct bacterial activity verification. A clear mechanistic threshold emerged at 5% acid concentration, where bio-GPC demonstrated the greatest stabilisation against gel depolymerisation through secondary mineral precipitation. At 10% acidity, both mixes underwent advanced degradation, and the benefits of biomodification were largely overwhelmed, with both systems showing dominant gypsum formation, severe gel dissolution, and substantially increased permeability, indicating that extreme acid concentrations exceed the stabilisation capacity of the bio-modified matrix. These lab-scale findings are consistent with microbial biomineralisation enhancing acid resistance within the pH ranges typical of sewer environments (3–5% H_2SO_4), supporting further investigation of bio-enhanced geopolymers as candidate sustainable materials for wastewater infrastructure, pending validation under dynamic field conditions.

Keywords *Shewanella oneidensis* · Ambient-cured geopolymers · Direct inclusion · Sulphuric acid · Self-healing · Bio-modification

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Introduction

Rapid urbanisation and population growth increase the demand for sustainable infrastructure. Wastewater systems are vulnerable to contamination and corrosion, reducing their lifespan. Despite advantages, challenges in material selection exist due to cost, installation complexity, and performance under extreme conditions¹. Understanding environmental, operational, and performance factors is crucial for choosing appropriate materials. By leveraging each material properties, which engineers can increase durability and safety, promoting sustainable solutions². The construction industry is undergoing a significant transformation, driven by the need for sustainable practices to mitigate environmental impacts, particularly in the context of cement production. Cement is a critical component of infrastructure development worldwide, with global production reaching approximately 4.1 billion tonnes in 2022³. However, this production contributes to roughly 6% of global greenhouse gas emissions, primarily due to the energy-intensive processes involved in producing clinker^{4,5}. As the demand for durable and resilient infrastructure grows, the development of innovative materials that reduce environmental footprints has become essential.

One promising approach is the use of geopolymers, a zero-cement concrete, which incorporates fly ash, an industrial by-product from coal combustion, as a partial substitute for traditional cement⁶. Geopolymers produced from fly ash offer a viable, low-carbon alternative due to their aluminosilicate gel network, low permeability, and strong resistance to chemical attack. However, their long-term behaviour in highly aggressive sewer environments remains insufficiently characterised, particularly regarding the interactions between acid exposure, gel stability, and calcium mobilisation. Parallel advances in self-healing concrete have shown that microbial-induced mineralisation can enhance durability by promoting CaCO_3 precipitation, refining pore structures, and stabilising degradation products. Most studies, however, focus on *Bacillus* species, with limited exploration of other microorganisms. The integration of fly ash not only enhances the mechanical properties of concrete but also addresses the pressing issue of waste management by utilising a material that would otherwise contribute to landfill waste⁷.

Self-healing concrete, with the incorporation of *Shewanella*, a type of bacteria, as a healing agent, can further improve the longevity and durability of sewer systems, such as pipes and manholes, by autonomously sealing cracks through microbial-induced calcite precipitation^{8,9}. *Shewanella oneidensis* presents a compelling candidate for geopolymer modification due to its facultative anaerobic metabolism, iron-reducing capacity, and tolerance of low-oxygen and low-pH conditions, properties that make it uniquely suited to sewer environments relative to obligate aerobes such as *Bacillus* species. The biomineralisation mechanism and species selection rationale are detailed in the Literature Review section. This self-healing mechanism is particularly valuable in environments exposed to harsh conditions, including acidic conditions, which can lead to significant degradation over time¹⁰.

This is the first study to systematically evaluate the material performance of *Shewanella Oneidensis*-modified fly ash geopolymer concrete across multiple sulphuric acid concentrations (0–10%), integrating chemical, microstructural, and transport analyses to develop an evidence-based mechanistic interpretation of bio-enhanced acid resistance in a sewer-relevant context; the confirmed novelty lies in the observed durability differentials, with the biomineralisation mechanism proposed on the basis of mineralogical and chemical evidence. This study addresses this research gap by evaluating the degradation mechanisms of fly ash-based GPC with and without *Shewanella* exposure to H_2SO_4 concentrations of 0%, 3%, 5%, and 10% by volume. This range was selected to span conditions from neutral reference through mild-to-moderate acidity representative of biogenic sulphuric acid corrosion in sewer crowns, where pH typically ranges from 0.5 to 4, equivalent approximately to 1–10% H_2SO_4 ^{11,12}, to a severe limit condition, consistent with the accelerated test parameters established by Madraszewski et al. (2022)¹³ and Chen et al. (2021)¹⁴. The proposed study focuses on parameters such as mass loss, electrical resistivity, and water permeability over a specified duration. The pH levels of the samples and the acidic solution were monitored daily, and chloride migration tests were carried out after 60 days in accordance with the “NT Build 492” standard. Lastly, microscopic analyses such as SEM/EDX and XRF were performed to compare the samples’ properties before and after testing. If validated at field scale, these findings could contribute to the

development of sustainable construction materials aligned with SDG 11 and SDG 12¹⁵, though translation from controlled laboratory conditions to real sewer environments requires further investigation of dynamic acid generation, biofilm interactions, and long-term bacterial viability.

Literature review

Extensive research demonstrates that geopolymers generally outperform Portland cement (OPC) in acidic environments due to their low-calcium, aluminosilicate structure, which resists acid-induced decalcification. Studies investigating fly ash-based geopolymers report significantly lower mass loss and improved mechanical stability under sulphuric, acetic, and hydrochloric acid exposure¹⁶. However, research specifically addressing geopolymer behaviour in sewer-simulated environments remains limited, particularly regarding microbially influenced corrosion (MIC). Most existing studies employ accelerated testing using mineral acids, which do not fully capture the complex interplay of microbial activity, acidity, and transport processes.

Self-healing approaches incorporating bacteria such as *Bacillus subtilis* and *Sporosarcina pasteurii* have been shown to enhance crack closure, calcium carbonate precipitation, and chloride resistance^{8,17}. However, the use of *Shewanella* in geopolymer systems has been minimally investigated, despite its ecological relevance to sewer environments and its unique metabolic capabilities, including iron reduction and survival under anaerobic, low-pH conditions. The potential of *Shewanella*, known for its unique ability to thrive in anaerobic environments, as a healing agent in geopolymer concrete remains an area ripe for exploration. The biomineralisation mechanism of *Shewanella* operates through three coupled pathways. First, under the anaerobic conditions that develop as the geopolymer matrix densifies during curing, *Shewanella* utilises Fe(III) as a terminal electron acceptor in dissimilatory iron reduction, oxidising organic carbon from the growth medium while generating localised alkalinity that elevates CO_3^{2-} availability^{18,19}. The Fe_2O_3 content of the fly ash matrix (5.53 wt%, Table 2) provides a sustained in-situ Fe(III) source for this pathway, a metabolic resource unavailable to obligate aerobes such as *Bacillus* species. Second, the elevated local CO_3^{2-} concentration combines with Ca^{2+} released from partial gel decalcification to exceed the calcite solubility product ($K_{\text{sp}} \approx 3.3 \times 10^{-9}$), driving CaCO_3 precipitation on bacterial cell surfaces and within adjacent pore space; Lou et al. (2021)²⁰ demonstrated this mechanism directly in a closely related species, *Shewanella putrefaciens*, showing protective biomineral film formation through surface mineralisation. This metabolic profile stands in direct contrast to the *Bacillus*-based MICP systems that dominate the bacterial concrete literature: *Bacillus subtilis* and *Sporosarcina pasteurii* are obligate aerobes reliant on ureolytic activity that becomes severely constrained at pH below 7 and under the oxygen-depleted conditions characteristic of active sewer crown microenvironments¹⁷; at the pH range of 0.5–4 typical of biogenic sewer corrosion zones, *Bacillus* species lose both viability and ureolytic function, whereas *Shewanella oneidensis* maintains metabolic activity through Fe(III) reduction independently of oxygen and pH^{18,19}, making it uniquely suited among candidate bacterial concrete organisms for sewer-specific applications. Third, extracellular polymeric substances (EPS) produced by *Shewanella* bind Ca^{2+} ions and act as organic nucleation templates, concentrating precipitation at pore throats rather than distributing it diffusely, thereby producing the targeted pore-sealing effect reflected in the 35–60% permeability reductions observed in bio-GPC. Understanding how *Shewanella* can enhance the self-healing properties of geopolymer concrete could lead to significant advancements in sustainable construction practices. Sewer environments often expose materials to corrosive substances, including sulphuric acid and hydrogen sulphide, necessitating further studies to ascertain the effectiveness of this combination in preventing corrosion and maintaining structural integrity^{9,11}. Most existing research predominantly addresses traditional concrete mixes, which have shown varying degrees of success in maintaining integrity under acidic exposure¹⁰. The absence of targeted studies on geopolymer concrete in this context underscores the need for further investigation into its resilience against such aggressive environments.

The inherent properties of geopolymer concrete, such as lower porosity and higher resistance to chemical attacks, when combined with the self-healing capabilities of bacteria, could effectively prevent corrosion and

maintain structural integrity⁸. This synergy has the potential to revolutionise the construction of sewer systems, making them more resilient to harsh conditions. Therefore, the exploration of these interactions is crucial for understanding the full potential of geopolymer concrete in practical applications. Existing research indicates that traditional concrete exhibits a decrease in mechanical properties with increased acid exposure; however, the comparative performance of geopolymer concrete in this regard remains to be established²¹. While some studies indicate that bacterial self-healing can enhance durability, the specific interactions of these materials in a sewer environment are not yet fully understood²². The long-term performance of such combinations under real-world conditions remains to be established, highlighting the need for field studies and practical testing²³.

Table 1 provides a comprehensive overview of various studies conducted on geopolymer concrete and its performance in acidic environments, particularly focusing on self-healing characteristics and resistance to corrosive agents. Several studies, such as those by Guo et al. (2023) and Zhao et al. (2024), investigate the self-healing capabilities of geopolymer composites, revealing that fly ash-based engineered geopolymer composites can recover significant tensile strength and strain after being pre-damaged^{24,25}. This incorporation of self-healing materials and bacteria could enhance the longevity and durability of these composites in harsh conditions.

Furthermore, multiple studies, including those by Yang et al. (2021) and Ariyadasa et al. (2024), assess the resistance of various geopolymer composites to sulphuric acid and other corrosive substances^{28,33}. The results indicate that fly ash-based geopolymer concrete exhibits superior performance compared to OPC, with significantly lower mass loss and better mechanical strength retention under acidic conditions. The studies also explore the microstructural evolution of geopolymer materials when subjected to these environments; for instance, low-calcium fly ash geopolymer mortar demonstrated minimal mass loss and retained mechanical strength even after prolonged exposure to highly acidic conditions, as previously reported by Ariyadasa et al. (2025)³⁵.

A critical distinction exists between accelerated laboratory acid immersion and real sewer corrosion conditions that must be considered when interpreting results from studies in Table 1, including the present work. Laboratory tests employ static mineral acid at fixed concentrations, whereas biogenic sulphuric acid in sewers is generated continuously by sulphur-oxidising bacteria, primarily *Acidithiobacillus thiooxidans*, creating dynamic pH gradients, localised corrosion hotspots, and fluctuating acid concentrations that vary with temperature, humidity, and sewage composition^{13,14}. Real sewer environments additionally involve complex microbial communities including sulphate-reducing bacteria, iron-oxidising bacteria, and neutrophilic heterotrophs that interact with concrete surfaces through biofilm formation, producing heterogeneous corrosion patterns absent from mineral acid immersion tests¹¹. Wet-dry cycling in sewer crowns further imposes salt crystallisation stresses and periodic desiccation that accelerate surface scaling beyond what constant immersion conditions replicate. Grengg et al. (2020)¹¹ directly compared laboratory mineral acid exposure with in-situ sewer performance, finding that dynamic MIC conditions produced more heterogeneous and faster degradation than static mineral acid tests predicted. These distinctions mean that laboratory acid immersion results, including those presented here, should be interpreted as establishing chemical acid resistance under controlled conditions, providing a necessary performance baseline, rather than as direct predictions of field service life. Field validation through bioreactor-simulated MIC exposure and pilot-scale sewer trials remains essential before practical implementation can be recommended.

The results summarised in Table 1 indicate that geopolymer concrete has significant potential for use in sewer environments, primarily due to its superior resistance to acidic conditions and its ability to maintain mechanical properties under stress. While existing studies have examined the self-healing properties of other bacterial species, the specific interactions and benefits of *Shewanella* in the context of geopolymer concrete remain largely unexplored. This literature gap motivates the present study, which offers the first detailed investigation of *Shewanella*-modified fly ash geopolymers across multiple sulphuric acid concentrations (0–10%), integrating chemical, microstructural, and transport analyses to develop a mechanistic understanding of acid-induced degradation. This gap in knowledge highlights the need for additional research to evaluate the effectiveness of this combination in preventing corrosion and maintaining structural integrity in sewer systems.

Table 1 Summary of work done and key findings on geopolymer concrete/self-healing composites under acidic conditions.

Materials/objective	Test condition	Mechanical/Microstructural properties
Self-healing behaviour of fly ash/slag-based engineered geopolymer composites	Pre-damaged strains subjected to self-healing in air, water, and NaOH/Ca (OH) ₂ solutions	<ul style="list-style-type: none"> - Retained strain hardening and multi-cracking behaviour - Healing products: C(N)-A-S-H gel and CaCO₃ - Significant self-healing in saturated Ca(OH)₂ and 7% NaOH solutions; new damage in high NaOH concentrations²⁵
Self-healing performance of engineered geopolymer composites subjected to sodium sulphate	Exposed to sodium sulphate (Na ₂ SO ₄) solution and wet-dry cycles	<ul style="list-style-type: none"> - Higher Na₂SO₄ concentration promotes self-healing in fine cracks - Self-healing products include C-(A)-S-H gels and CaCO₃²⁶
Resistance of soda residue-fly ash based geopolymer mortar to acid and sulphate environments	Exposed to 5% HCl and 5% Na ₂ SO ₄ environments	<ul style="list-style-type: none"> - Superior resistance of SR-FFA-GEO mortar to 5% HCl and 5% Na₂SO₄ compared to cement mortar - Long-term mechanical strengths evaluated up to 360 days²⁷
Self-healing properties and mechanism of the cracked fly ash-based engineered geopolymer composites (FA-EGC)	Evaluated in different environments (air, wet-dry cycles, 20 °C water, 40 °C water)	<ul style="list-style-type: none"> - Increased temperature and presence of water enhance self-healing performance - N-A-S-H gels identified as primary self-healing products in air; (N/C)-A-S-H gels in water environments²⁴
Resistance to sulphuric acid corrosion of geopolymer concrete based on different binding materials and alkali concentrations	Immersed in sulfuric acid solution with pH=1 for 6 days	<ul style="list-style-type: none"> - Compressive strength increased due to gypsum presence in fly ash-based GPC - Metakaolin-based GPC failed after immersion - Best performance observed in GPC with high-calcium fly ash and 12 M NaOH²⁸
Performance of FA-based geopolymer concretes exposed to acetic and sulphuric acids	Exposed to 1 M sulfuric acid and acetic acid for 360 days	<ul style="list-style-type: none"> - Alkali-activated concretes showed better performance than OPC - Residual strengths: 2 MPa (OPC), 10 MPa (FA/OPC), 19 MPa (FA/GBFS) in H₂SO₄ - Gypsum formation observed in H₂SO₄ exposure²⁹
Macro and microstructural evolution of low-calcium fly ash-based geopolymer mortar exposed to sulphuric acid corrosion	Immersed in 0.5 pH sulfuric acid at 40 °C for 31 days	<ul style="list-style-type: none"> - Mass loss of OPC: 49% - Mass loss of FAGP: 6% (42% lower than OPC) - Indicates calcium-bearing hydrates in OPC are susceptible to acid corrosion³⁰
Long-term durability of iron-rich geopolymer concrete in sulphate, acidic and peat environments	Immersed in 1% and 3% sulfuric acid, 5% Na ₂ SO ₄ , 5% MgSO ₄ , and simulated peat solution for 18 months	<ul style="list-style-type: none"> - Strength increased in Na₂SO₄ by 8.9% (12 months) and 6.3% (18 months) - Decreased strength in MgSO₄ by 11% (12 months) and 19.8% (18 months) - In 1% and 3% H₂SO₄: Strength loss of 1.2% and 4.5%, and mass loss of 2% and 3%, respectively - Simulated peat: 1.25% strength increase (12 months), 11.3% decrease (18 months)³¹
Durability of calcium-rich municipal solid waste incineration fly ash-based geopolymer to sulphate and sulfuric acid	Immersed in water, 3% Na ₂ SO ₄ , 1% H ₂ SO ₄ , and 3% H ₂ SO ₄ for 112 days	<ul style="list-style-type: none"> - Na₂SO₄ showed minimal erosion due to inert CaCO₃ - H₂SO₄ caused decalcification and dealumination, forming gypsum and cracks - Solidification efficiency of Pb and Zn reduced under H₂SO₄, while Cu and Cr remained above 70% efficiency³²
Bond performance of fly ash-based geopolymer mortar in simulated concrete sewer substrate	Slant shear testing on Rough-Dry, Rough-Saturated, Smooth-Dry, and Smooth-Saturated substrates	<ul style="list-style-type: none"> - Bond strength: 14 MPa (outperformed OPC and P-GP) - Acid resistance preserved bond integrity - No delamination observed under line load testing; OPC and P-GP delaminated at 20% ultimate load³³
Resistance of alkali-activated slag mixed with wastewater towards biogenic sulfuric acid attack	Immersed in sulfur oxidizing fungi (<i>Aspergillus Niger</i>) for up to 180 days	<ul style="list-style-type: none"> - Ni and Cr wastewater reduced fungal growth and sulfuric acid production - Lowest leachability observed for Ni and Cr samples - Compressive strength regression reduced significantly³⁴
Degradation mechanisms of low-calcium fly ash-based geopolymer mortar in simulated aggressive sewer conditions	Immersed in sulphuric acid solutions (pH 0.5, 1, and 4) at 40 °C for up to 3000 h	<ul style="list-style-type: none"> - FAGP retained 30% mechanical strength after 3000 h at pH 0.5 - OPC failed after 2000 h exposure - Ion leaching and pore creation observed³⁵
Impact of original aggregate and attached mortar types of recycled aggregates on sulfuric acid resistance of geopolymer recycled concrete	Immersed in sulfuric acid solution (5% concentration) for 91 days	<ul style="list-style-type: none"> - Quartz-based RA exhibited superior acid resistance - Compressive strength and mass loss ratio minimally affected (1.7% and 5.8%) - Limestone-based RA degraded more quickly³⁶

Materials and methods

Fly ash-based geopolymer concrete was produced using Class F fly ash, activated with a sodium silicate/12 M NaOH mixture. Two formulations were prepared:

- (1) a control mix without bioadditives,
- (2) a bio-modified mix incorporating *Shewanella Oneidensis* at 10^8 CFU/mL, added directly during mixing.

Cylindrical specimens were cast and cured at 30 °C for 7 days.

Specimens were immersed in 0%, 3%, 5%, and 10% H₂SO₄ at 30 °C and 100% humidity for 60 days. pH of both the solution and mortar interior was monitored regularly. Mass variation was recorded every 4 days. Capillary absorption followed BS EN 1015-18, chloride migration followed NT Build 492, and water permeability was assessed using pre-cracked discs under constant hydraulic head. Post-exposure microstructural characterisation included:

- SEM/EDSX to assess gel morphology and elemental distribution,
- XRF to quantify oxide composition changes,
- XRD to identify crystalline phases formed after acid attack, and.
- FTIR to assess aluminosilicate network depolymerisation and carbonate formation.

These combined methods, detailed below, allowed quantitative and mechanistic evaluation of acid-induced degradation.

Binders and additive materials

Class F fly ash (FA) served as the sole aluminosilicate source for producing GPC mortar, activated by a mixture of sodium silicate and sodium hydroxide (NaOH) solution and its chemical composition was analysed via X-ray fluorescence (XRF), and phase patterns were analysed via XRD. The results are presented in Table 2; Fig. 1, respectively. Geopolymer concrete performance was accessed in acidic environments together with their self-healing capabilities.

Cylindrical mortar specimens measuring 50 mm in diameter and 150 mm in height were cured at 30 °C for 7 days, followed by 21 days of ambient laboratory curing, giving a total pre-immersion curing period of 28 days. These conditions were selected to ensure proper curing and strength development prior to testing, based on previous research recommendations³⁷. It is acknowledged that this protocol may not achieve the maximum strength potential of the mix; the reported acid resistance performance, therefore, represents a conservative baseline, and a more fully developed matrix may exhibit further enhanced durability. The NaOH pellets, 98% pure and supplied by Thermo Scientific, were dissolved in distilled water to prepare the NaOH solution, which was made a day before testing. A 12 M NaOH solution was prepared in advance. Additionally, sodium silicate was purchased from Fisher Scientific. The coarse aggregate, sized 10–20 mm, was supplied by Specialist Aggregate and Travis Perkins in the UK, while fine sand for the aggregate was obtained from Tarmac Plant, UK.

Sample preparation

The mixing process started with a 5-minute dry blend of FA, coarse aggregate, and sand in a low-speed mixer. This was followed by a 3-minute mixing period during which the alkaline activator was gradually added to produce a uniform geopolymer mortar. To prepare bio samples, 200 ml of the bioproduct was added and mixed for an additional 2 min. The study utilised a strain from the *Shewanella* genus, known for its ability to respire iron and

Table 2 Chemical compositions of fly ash (wt%).

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	P ₂ O ₅	SO ₃	TiO ₂	CaO	K ₂ O	MgO	SrO	BaO
56.3	20.8	5.53	0.836	0.578	0.915	3.33	1.99	1.53	0.16	0.154

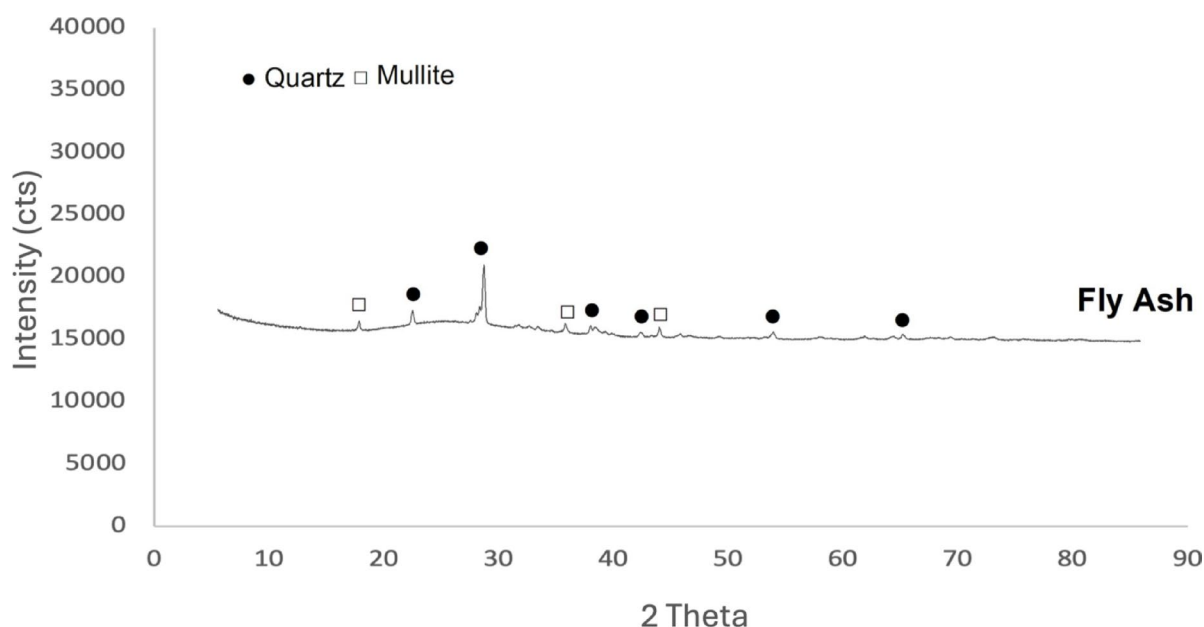


Fig. 1 XRD patterns of fly ash used in geopolymer samples.

withstand fluctuating low pH environments. The *Shewanella Oneidensis* strain was inoculated into autoclaved Tryptic Soy Broth (TSB), which was cooled before use. The bacterial culture was grown in a shaking incubator for 24 h at 30 °C and 200 rpm; serial dilution confirmed a concentration of 10^8 CFU/ml, selected based on prior literature demonstrating optimal mineralisation activity at this inoculation level^{8,9}. Alkaline activation at pH > 12 presents a viability challenge during mixing; the rationale for partial bacterial survival through the curing period, including TSB buffering, matrix pH equilibration, and Fe(III) availability as an anaerobic electron acceptor, is detailed in the Literature Review^{18,19}. The TSB growth medium introduces organic components, enzymatic digests of casein and soybean meal, sodium chloride, and glucose, that may in principle influence geopolymerisation kinetics or contribute to early carbonate formation through alkaline decomposition; however, the similar XRD amorphous hump positions and FTIR Si-O-T band characteristics between bio and control specimens prior to acid exposure (Figs. 2 and 3; Table 6) indicate that TSB addition did not substantially alter the primary geopolymer matrix chemistry at the volume used. A TSB-only control is recommended in future work to formally quantify the medium contribution independently of bacterial effects. Direct CFU enumeration at multiple curing ages was not conducted in this study and is recommended as a priority for future work. The ratios of FA to activator and NaOH to sodium silicate used were 2.0 and 2.5, respectively. The concrete mix design for six 100 mm x 100 mm cubes and ten 100 mm x 150 mm cylinders for the geopolymer is shown in Table 3.

Testing procedures

Since there is no standard method for simulating aggressive sewer conditions, previous approaches were adopted. Commonly reported values include a pH below 1 in sulphuric acid medium with a relative humidity (RH) of at least 98%, typically at temperatures above 30 °C^{13,14}. The concentrations of 0%, 3%, 5%, and 10% H₂SO₄ were selected to span the range from neutral (reference) through mild-to-moderate acidity, representative of biogenic sulphuric acid corrosion in sewer crowns (typically pH 0.5–4), to a severe limit condition^{13,14}. The study's methodology comprises immersing GPC samples in 0, 3, 5, and 10% sulphuric acid at 30 °C and 100% humidity to simulate sewer conditions. Specimens were cured for 28 days prior to immersion; acid exposure therefore ran from day 28 to day 60, representing a 32-day immersion period within the total 60-day experimental duration. Visual examination of all specimens was conducted at day 28 (before immersion) and day 60 (after exposure);

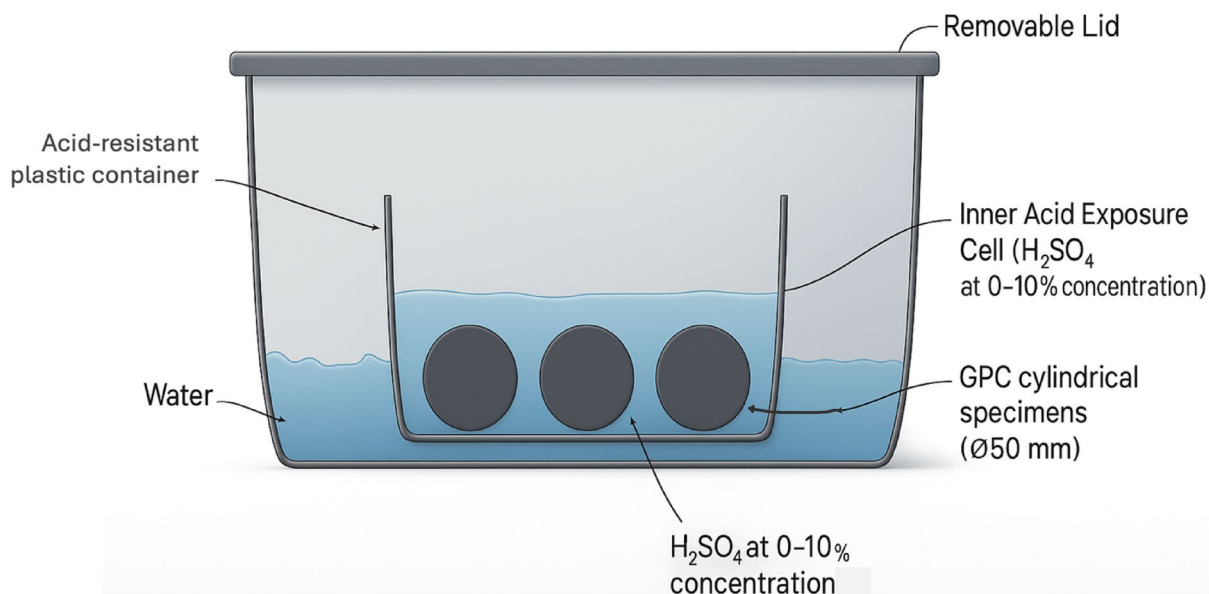


Fig. 2 XRD patterns of bio and control samples at 60 days after acid immersion with different concentrations.

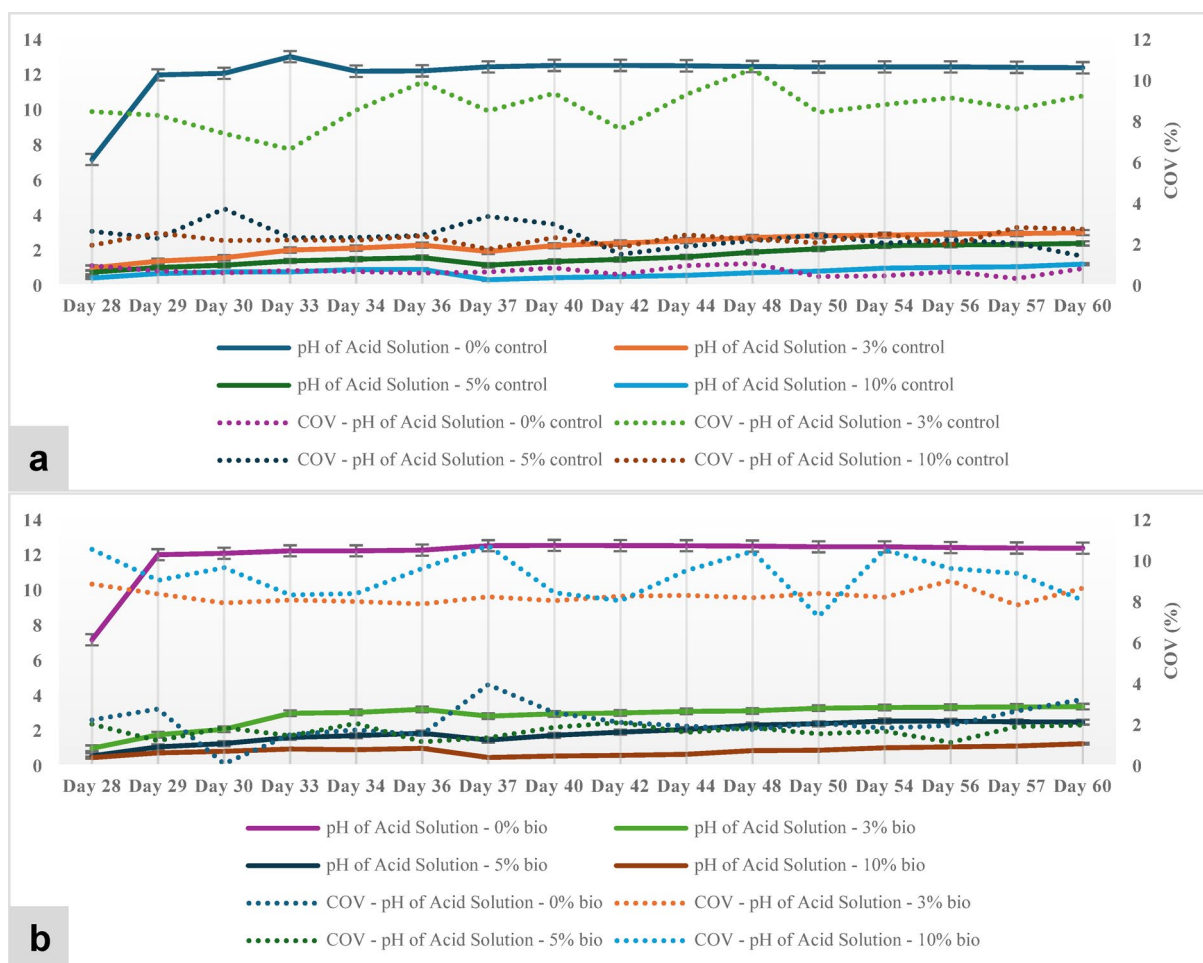


Fig. 3 XRD patterns of bio and control samples at 60 days without acid immersion.

Table 3 Mix proportion of geopolymer concrete.

Component	Kg
Fly Ash	15.27
Sand	13.75
Aggregate	20.62
NaOH	2.18
Sodium Silicate solution	5.46

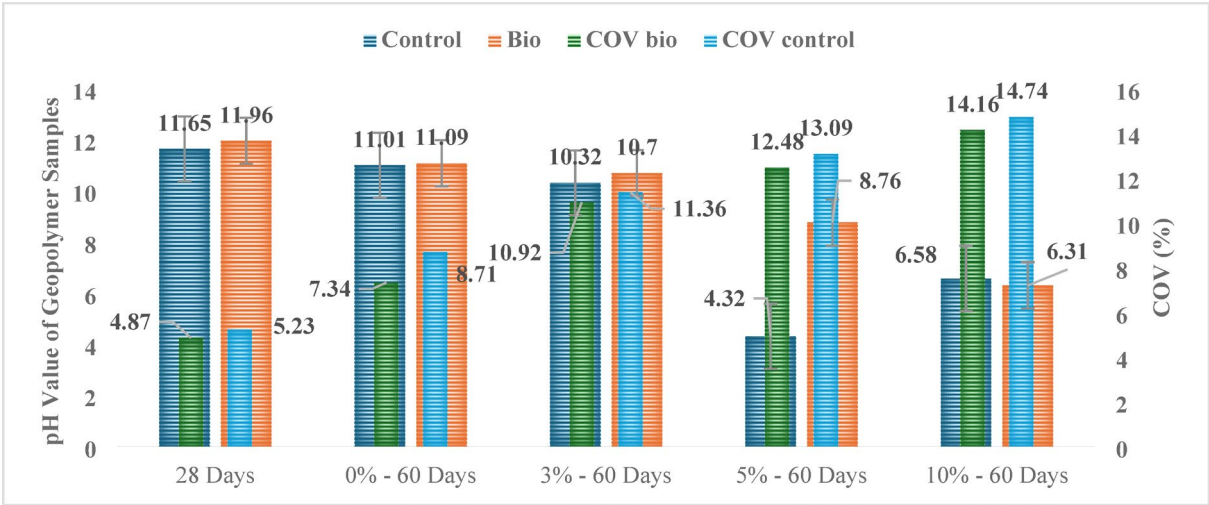


Fig. 4 Sewer simulation exposure box.

photographs for all conditions are provided in the Supplementary Materials. Three 100 mm x 150 mm samples of each type are prepared (Fig. 4): (1) control samples (without bio), 2) bio samples.

The pH of the exposure solution (H_2SO_4 acid) was regularly measured with a calibrated benchtop Plus pH Meter (ETI Ltd., UK) to track material degradation. The rate of pH change was used to assess acid consumption across various mortar types. pH levels of the mortar samples were specifically checked at 60 days to understand the acid’s impact. Moreover, changes in sample mass, after immersion in acid, were recorded every 4 days using a CKE 6K0.02 precision balance (KERN & SOHN GmbH, Germany) to evaluate the degree of material degradation caused by acid exposure. Three replicates were tested per condition across all experiments. Results are reported as means; variability was within $\pm 5\%$ for transport tests and ± 0.2 pH units for pH measurements, confirming acceptable reproducibility. To assess data consistency, the Coefficient of Variation (COV) was calculated for all quantitative datasets and is presented as error indicators on all relevant figures. All COV values fall below 20%, confirming acceptable experimental reproducibility across conditions.

Water absorption via capillary was conducted following BS EN 1015-18:2002 standards. Cylinder samples measuring 50 mm thick and 100 mm in diameter were used. Three samples of each type were oven-dried at 30 °c until their mass change was no more than 0.1%. Subsequently, they were submerged in 10 mm of water within a sealed container to maintain a consistent hydrothermal environment. The samples’ masses were measured at 0, 5, 15, 30, 60, 120, and 180 min, then at 21 and 24 h, and subsequently at daily intervals up to 28 days or until water absorption stabilised.

The non-steady state chloride migration test was performed in accordance with NT BUILD 492 standards. Three cylindrical samples, each 50 mm thick and 100 mm in diameter, were prepared. The samples were pre-conditioned by vacuum for three hours and then immersed in calcium hydroxide for 18 ± 2 h. Afterwards, they were sealed in rubber sleeves, exposing only the top and bottom surfaces. The top surface faced an anolyte solution (0.3 M NaOH), while the bottom contacted a catholyte solution (10% sodium chloride). A voltage supply started at 30 volts, with the initial current for each sample recorded. The voltage was then adjusted based on these

initial readings and maintained for six hours. Post-test, the samples were fractured with a hydraulic compression machine, and chloride penetration was evaluated using silver nitrate spray. The chloride migration coefficient, D_{nssm} ($\times 10^{-12} \text{ m}^2/\text{s}$), was calculated and compared among the samples.

Water permeability test was conducted using three pre-cracked 100 mm x 50 mm disks of each type were used, sealed on their sides to prevent water leakage through cracks. Crack development was analysed via optical microscopy, measuring widths with a crack meter calibrated for accuracy. Measurements at three evenly spaced points along each surface were averaged; when multiple cracks occurred, their widths were summed. If a pore was present, measurements were taken at the nearest non-pore site. Polychloride vinyl (PVC) tubes (100 mm diameter, 250 mm height) were attached to the disks, with the bottom surface wrapped in PVC tape after sealing to prevent water escape. The setup involved filling the tubes with a 200 mm water column, then removing the tape at time zero to start the test, recording water head reduction at intervals, and calculating total flow over 30 min. Flow follows Darcy's law, with the permeability coefficient calculated by Eq. (1)^{38,39}.

$$K = Ql/Ah \quad (1)$$

where Q is the flow rate through the specimen, l is its thickness, A is the cross-sectional area of the concrete specimen, and h is the hydraulic head drop across the specimen.

The electrical resistivity was measured using a Resipod Wenner four-probe meter. Cylindrical samples were used, with three readings taken from each to calculate the average. For material characterisation, a scanning electron microscope (SEM) analysed fragments from post-compressive strength tests, focusing on GPC morphology. These fragments were carefully cut and coated with platinum. Two sections, called the inside and outside fragments, were examined. The elemental composition was determined with EDS, analysing six spots on each fragment. The crushed fragments were ground into a fine powder, and the chemical composition of each mix was analysed using a Rigaku NEX CG II X-ray fluorescence (XRF) spectrometer. For each mix, three samples were tested, and their average values were reported. Additionally, phase analysis of raw FA and geopolymers samples was performed with a Rigaku MiniFlex 600 (Rigaku Corporation, Tokyo, Japan) powder X-ray diffractometer (XRD). The samples were finely ground and scanned from 5° to 80° at a rate of 0.01° per second. The IRAffinity-1S series SHIMADZU FT-IR spectrophotometer, operating over a spectral range of 500 cm^{-1} to 4000 cm^{-1} , was used to analyse changes in the functional groups of mortar samples exposed to acid corrosion.

Results and discussion

Results are interpreted within the biomineralisation framework described in the Literature Review, secondary CaCO_3 precipitation retaining Ca, occluding pore throats, and buffering internal pH, supported by converging evidence from six independent techniques, none of which produced results inconsistent with the proposed mechanism. Together, these processes converge at 5% acid exposure, where the available evidence is consistent with a competition between two mineralogical pathways defining the threshold: at 3–5% H_2SO_4 , the data suggest sulphate activity remains insufficient to drive preferential gypsum ($\text{CaSO}_4 \cdot 2 \text{H}_2\text{O}$) precipitation, with the stabilising calcite pathway appearing to dominate; at 10%, the pattern of XRD peaks, XRF SO_3 trends, and SEM micro-cracking is consistent with SO_4^{2-} activity redirecting Ca^{2+} toward expansive gypsum crystallisation rather than CaCO_3 formation. This interpretation is proposed on the basis of converging qualitative evidence; quantitative phase analysis through Rietveld refinement or explicit thermodynamic modelling would be required to establish this mechanism with greater confidence. The findings are thoroughly examined in the subsequent discussion.

COV values for all reported quantitative datasets are presented as error indicators on the corresponding figures. All values fall below 20%, confirming that the observed trends reflect genuine material behaviour rather than experimental scatter, and supporting confidence in the reported differences between bio-modified and control specimens. For the key transport property comparisons, permeability reduction of 35–60% at 5% acid, D_{nssm}

differential of $20.25 \times 10^{-12} \text{ m}^2/\text{s}$ at 10% acid, and capillary absorption differences across all concentrations, the magnitude of observed differences substantially exceeds the within-condition variability indicated by COV values, providing practical confidence in the reported trends. Formal ANOVA testing was not performed given $n=3$ per condition; this is acknowledged as a limitation, and a larger replicate series is recommended in future work to provide formal statistical confirmation.

Materials properties

Variation of pH in GPC samples and acid solution and associated concrete mass variation

Figure 5 presents the variation in pH levels of (a) sulphuric acid solutions for control samples during the study period, (b) sulphuric acid solutions for bio samples during the study period. Figure 6 shows the variation in pH levels of GPC samples at 28 (before acid immersion) and 60 (after acid immersion) days of age for 0–10% acid exposures. Figure 7 shows the mass loss of GPC samples from 28 to 60 days, during 0, 3, 5 and 10% acid exposure for (a) control samples, (b) bio samples.

Figure 5 demonstrates that the solution pH increased over time across all sample mixtures, confirming the process of acid neutralisation via alkali release from the geopolymer matrix. At 0% exposure, a rapid rise in alkalinity was observed due to unrestricted leaching of OH^- and Na^+/K^+ ions. When subjected to 3–5% acid exposure, the pH increase was more gradual, indicating controlled gel dissolution rather than severe matrix degradation. Importantly, bio-GPC maintained a higher solution pH compared to the control at 3–5% exposure, suggesting an enhanced buffering capacity⁴⁰. This improved pH retention in bio samples is consistent with decreased permeability and chloride migration, and is proposed to result from limited gel dissolution and secondary Ca-rich precipitation, as suggested by XRF and EDS analyses discussed later. At a 10% acid concentration, the pH increase was minimal for both systems, indicating that the acidic conditions overwhelmed the matrix's neutralisation capacity and reduced the advantages of biomodification.

Figure 6 demonstrates that all samples maintained an alkaline pH after 60 days; however, the retention of pH was significantly influenced by acid concentration and mixture type. Notably, the bio-GPC samples exhibited higher internal pH levels at 3–5% acid exposure, with a marked increase at 5% (8.76 compared to 4.32 for the control), indicating enhanced buffering capacity under moderate acidity. This improved pH stability is attributable to decreased gel dissolution and secondary Ca-rich precipitation, as corroborated by XRF and EDS analyses, which mitigated structural degradation and acid penetration.

At 10% acid exposure, the control specimen shows a higher internal pH (6.58) than at 5% (4.32), which is not indicative of better performance but reflects a mechanistically distinct degradation regime: the severity of attack at 10% drives rapid near-complete dissolution of the outer gel layer, producing a dense gypsum-enriched surface crust, confirmed by dominant gypsum XRD peaks and CaO of 26.9 wt% (Table 4), that partially impedes further acid ingress into the specimen interior, temporarily isolating it and producing an apparently higher internal pH. This interpretation is supported by the simultaneously highest permeability, capillary absorption, and SEM microcracking at 10% control, confirming advanced overall degradation despite the higher interior pH reading. Bio-GPC, by contrast, shows a monotonic pH reduction from 8.76 at 5% to 6.31 at 10%, reflecting genuine progressive buffering capacity reduction rather than interior isolation, consistent with a chemically more uniform matrix maintained by calcite precipitation, preventing preferential gypsum crust formation.

Figure 7 demonstrates that control specimens experienced significant mass gain at 0% and 3% exposure, primarily due to water absorption. In contrast, the 5% exposure showed minimal change, suggesting decreased sorption and the initiation of acid-induced gel dissolution. At 10% exposure, continued mass increase indicates simultaneous leaching and absorption processes. Overall, the control matrix exhibits higher water uptake and diminished stability under acidic conditions⁴¹. Conversely, bio-modified samples consistently displayed lower mass variations across all exposure levels, confirming reduced water uptake and leaching. Notably, at 5% acid exposure, bio-GPC exhibited a controlled mass decrease rather than gain, reflecting a net balance between slow

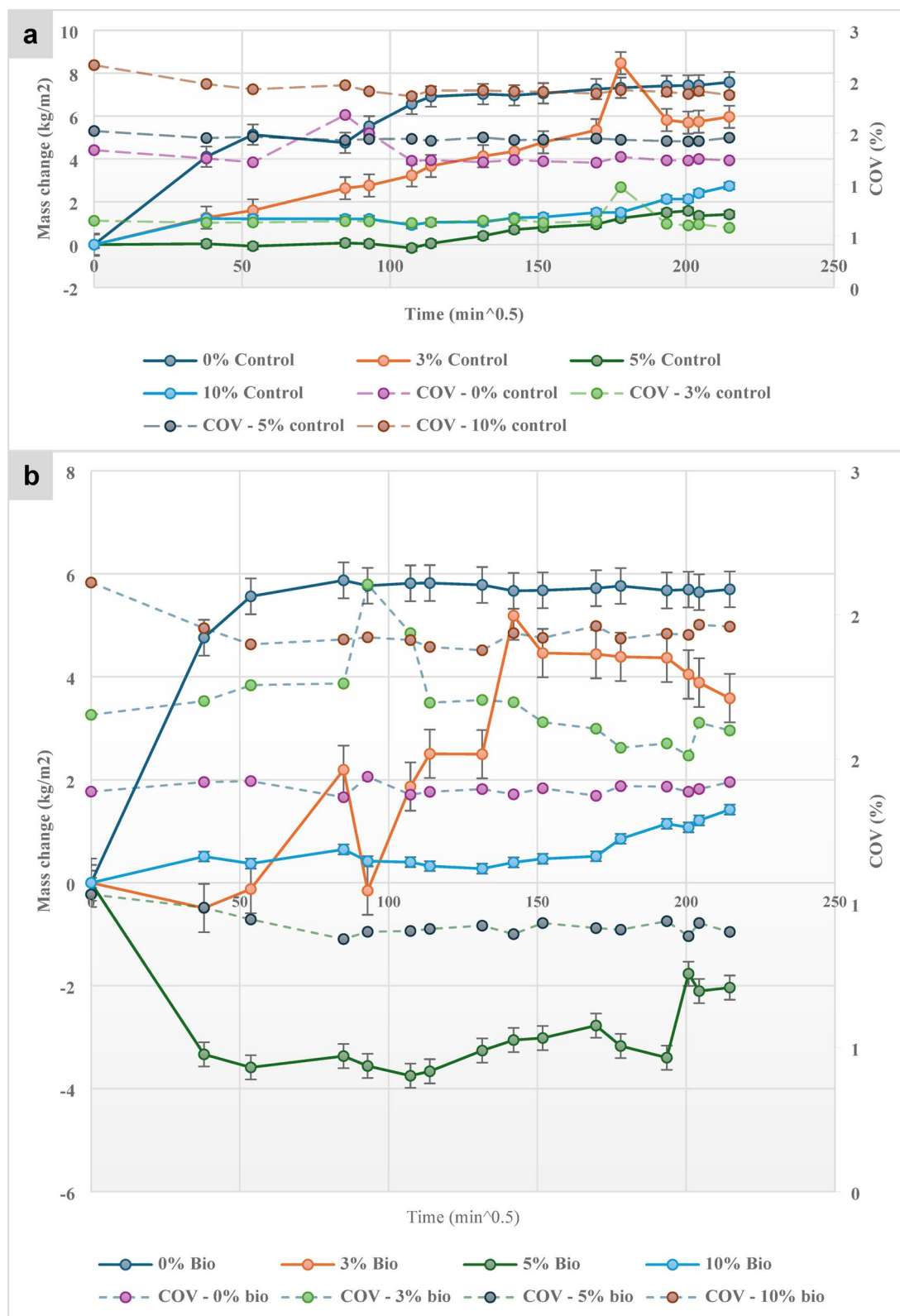


Fig. 5 Variation in pH levels of sulphuric acid solutions for: (a) *control* samples during the study period, (b) *bio* samples during the study period.

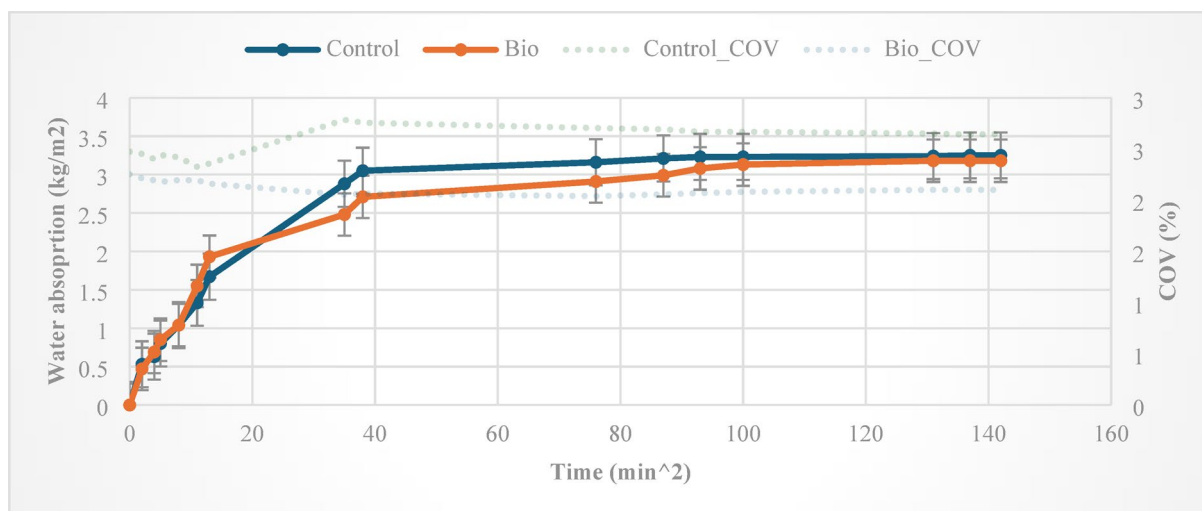


Fig. 6 Variation in pH levels of GPC samples at 28 (before acid immersion) and 60 (after acid immersion) days of age for 0–10% acid exposures.

gel dissolution and partial mass recovery through secondary CaCO_3 precipitation, the latter confirmed by XRD calcite reflections and EDS Ca enrichment at this concentration.

The net negative mass change indicates that dissolution slightly outweighs precipitation within the 60-day window, but at a substantially lower rate than the unrestricted acid uptake observed in control specimens. Whether this balance would shift toward stabilisation or accelerating mass loss over longer durations depends on the persistence of bacterial activity beyond the study period. Notably, EDS elemental maps at 180 days (Fig. 8) show that localised Ca enrichment in bio-GPC persists at this extended timepoint, confirming the durability of the early-age precipitation legacy, though this observation cannot establish whether bacterial activity continued beyond the immersion period; this is discussed further in the limitations section.

Water absorption via capillary

Figures 9 and 10 reveal that capillary uptake is heavily affected by prior acid exposure. The unexposed reference samples (Fig. 9) show significantly lower absorption ($2.3\text{--}2.5\text{ kg/m}^2$) compared to acid-immersed mixes, which reach $5\text{--}7\text{ kg/m}^2$. Both control and bio samples eventually reach similar saturation levels, suggesting that bacterial incorporation does not impair pore connectivity under non-acid conditions.

Figure 10 demonstrates that acid exposure markedly enhances capillary absorption, with the severity following the sequence $10\% > 5\% > 3\% > 0\%$, thereby corroborating the progressive dissolution of gel and coarsening of pores. Nonetheless, bio-GPC consistently exhibited lower capillary absorption than control specimens across all acid concentrations, with the most notable difference at 5% exposure, consistent with slower acid ingress and greater matrix stability in bio-modified samples under the static immersion conditions tested⁴². At 5%, control specimens show higher absorption attributable to pore opening and decalcification, while bio-GPC sustains lower uptake, a pattern consistent with pore refinement through secondary mineral precipitation, independently corroborated by the 35–60% permeability reduction and denser gel morphology observed in SEM. At 10% acid concentration, both formulations exhibit elevated absorption, indicating that severe acidity surpasses the protective capacity conferred by biomodification.

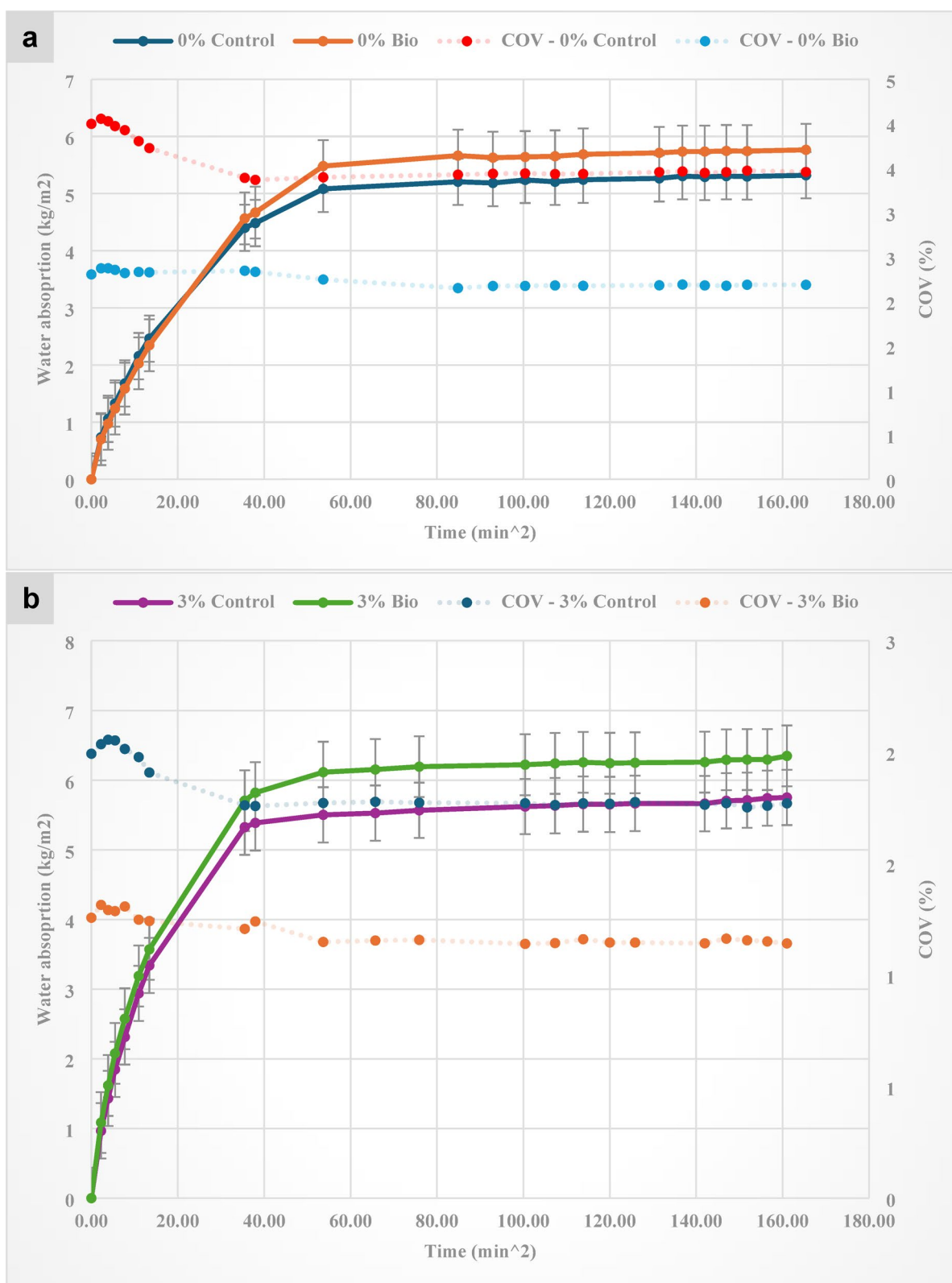


Fig. 7 Mass loss of GPC samples from 28–60 days, during 0, 3, 5 and 10% acid exposure for (a) control samples, (b) bio samples.

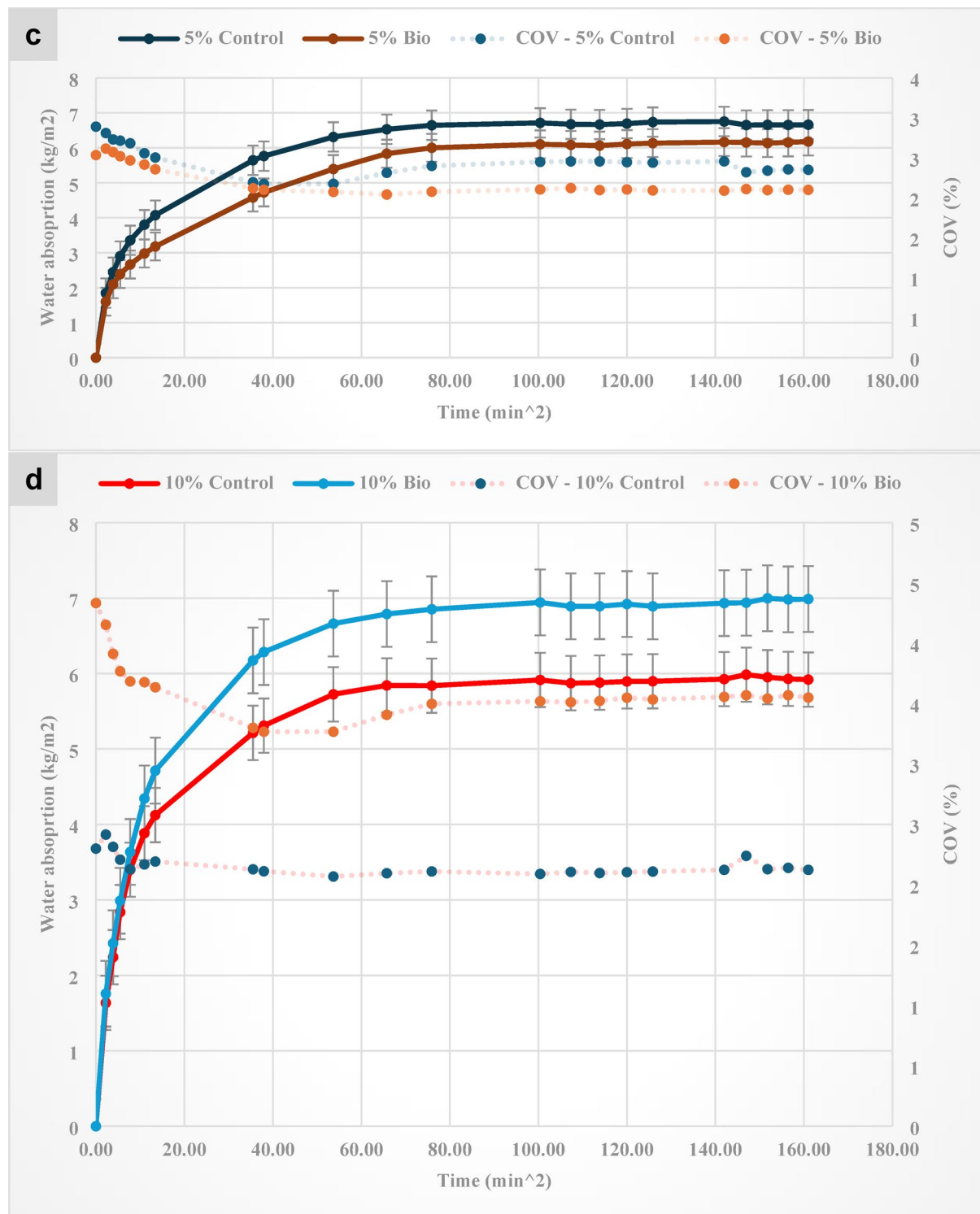


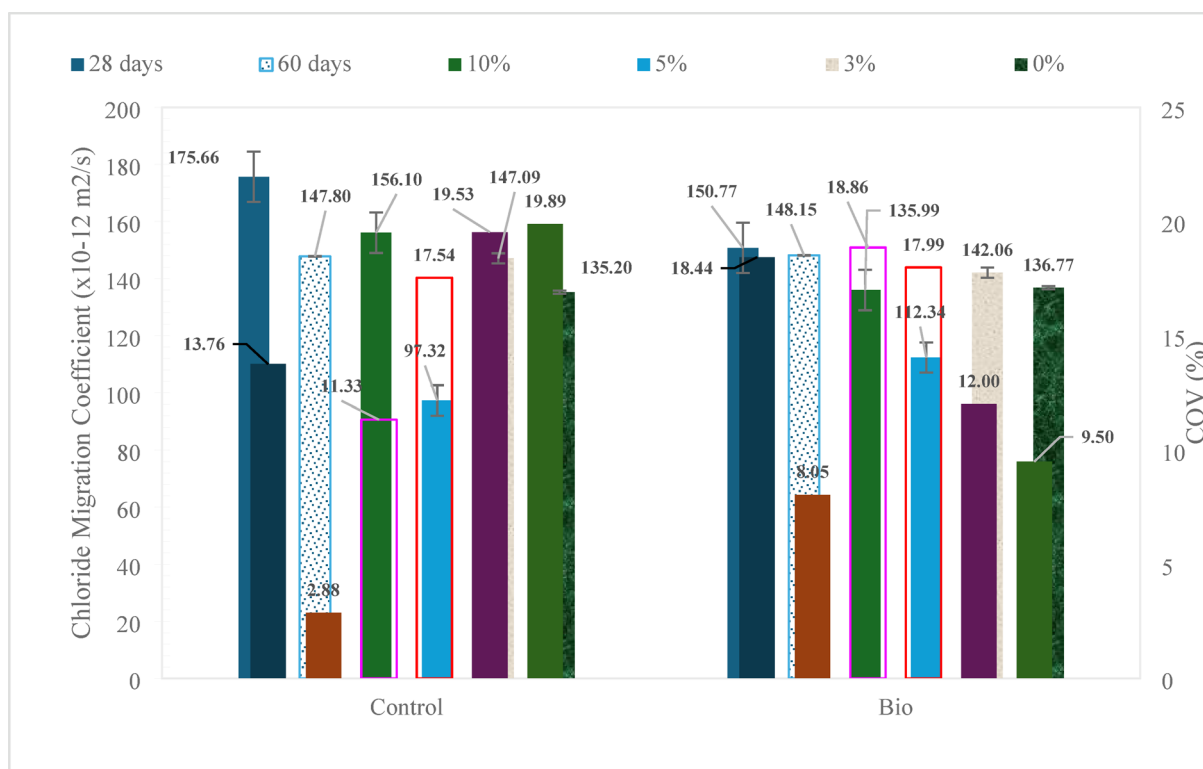
Fig. 7 (continued)

Chloride migration

Figure 11 illustrates that chloride migration is influenced by both curing age and acid exposure, with bio-modification generally enhancing transport resistance. At 28 days, bio-GPC demonstrates a lower D_{nssm} compared

Table 4 Chemical compositions of bio and control samples without acid immersion and with acid immersion at different concentrations (wt%).

Chemical Compositions	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	P ₂ O ₅	SO ₃	TiO ₂	CaO	K ₂ O	MgO	Na	Cl
Control (without acid exposure)	42.7	11.3	3.56	0.144	0.641	0.51	6.24	1.67	0.87	7.54	0.0088
Bio (without acid exposure)	41.7	11	3.5	0.152	0.609	0.518	6.69	1.59	0.925	8.12	0.012
0% - Control	46.2	11.4	2.95	0.302	0.146	0.544	9.45	1.33	0.865	6.65	0.0043
0% - Bio	38.2	10.2	3.4	0.169	0.221	0.493	14.6	1.38	0.922	5.91	0.0064
3% - Control	42.4	10.9	2.86	0.373	0.362	0.501	6.48	1.22	0.807	5.91	0.0094
3% - Bio	46	12.5	3.96	0.155	0.924	0.582	8.13	1.81	0.963	5.94	0.0092
5% - Control	48.2	11	3.02	0.330	2.83	0.594	9.62	1.25	0.754	4.65	0.0062
5% - Bio	42.5	11	3.55	0.193	1.38	0.551	14.5	1.56	0.955	5.42	0.0105
10% - Control	24.5	6.77	2.75	0.118	0.913	0.391	26.9	0.940	0.905	3.34	0.014
10% - Bio	46.3	8.82	3.42	0.105	9.19	0.572	10.7	1.4	0.596	2.52	0.0167

**Fig. 8** EDS elemental maps of geopolymer samples (at 180 days): (a) Control sample without acid exposure, (b) Bio sample without acid exposure, (c) Bio sample with 0% acid exposure, (d) Bio sample with 3% acid exposure, (e) Bio sample with 5% acid exposure, (f) Bio sample with 10% acid exposure.

to the control (150.77 vs. $175.66 \times 10^{-12} \text{ m}^2/\text{s}$) at neutral pH, which is aligned with a denser gel/ITZ observed via SEM and the higher CaO content from XRF, which suggests limited biomineral deposition or early pore refinement. By 60 days without acid exposure, both mixes attain similar values ($\sim 148 \times 10^{-12} \text{ m}^2/\text{s}$), suggesting that ongoing geopolymerisation and pore refinement reduce early differences. Following acid exposure, the 0% and 3% treatments yield intermediate coefficients (control: 136.77 and 147.09 ; bio: 136.77 and $142.06 \times 10^{-12} \text{ m}^2/\text{s}$), indicating limited additional densification beyond standard curing. At 5% acid, Dnssm reaches its lowest levels overall, though the control ($97.32 \times 10^{-12} \text{ m}^2/\text{s}$) records a marginally lower coefficient than bio-GPC ($112.34 \times 10^{-12} \text{ m}^2/\text{s}$). This apparent anomaly is explained by the greater gypsum formation in control samples

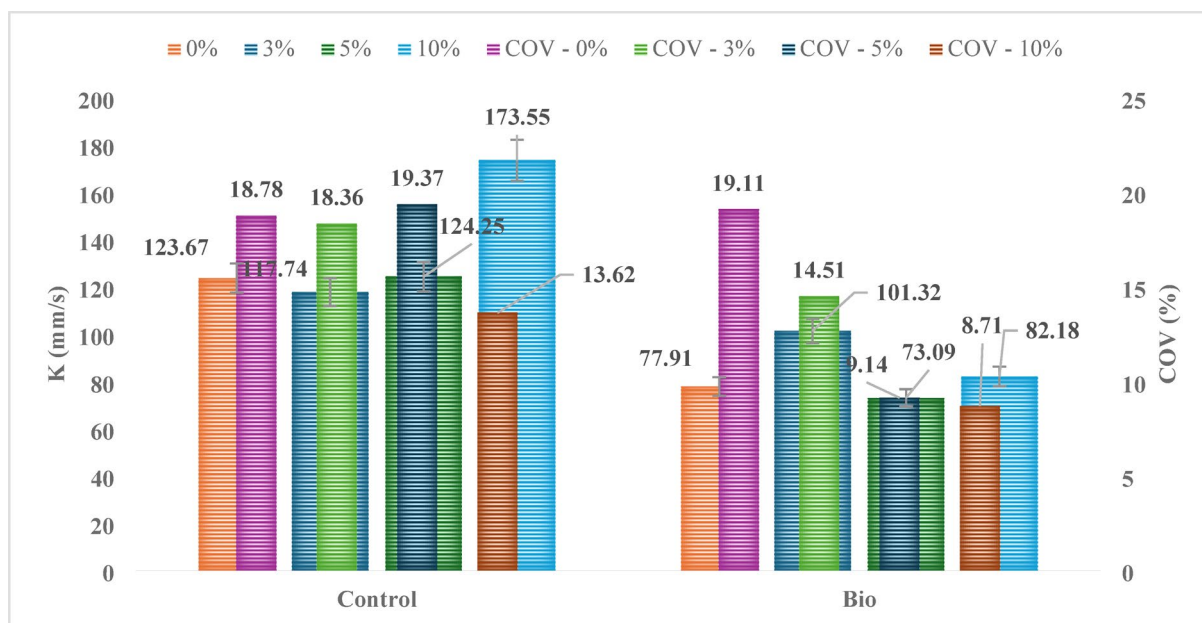


Fig. 9 Water absorption via capillary for GPC samples (at 60 days) without acid immersion.

at 5% (higher SO_3 at 2.83 vs. 1.38 wt%, Table 4; stronger gypsum XRD reflections, Fig. 2), which transiently occludes pore throats and reduces ionic pathways at the 60-day measurement point.

This temporary transport reduction does not reflect durable matrix stabilisation: gypsum is an expansive phase, and the same control specimens simultaneously exhibit higher permeability, higher capillary absorption, and significantly lower internal pH (4.32 vs. 8.76) than bio-GPC at 5%, indicators that better reflect long-term matrix integrity. Conversely, at 10%, Dnssm increases (control: 156.10; bio: $135.99 \times 10^{-12} \text{ m}^2/\text{s}$), reflecting acid-induced gel dissolution and pore coarsening, corroborated by increased permeability and SEM evidence of microcracking. The relatively narrow Dnssm range across 0–5% reflects the inherently low permeability of the fly ash geopolymer matrix. More broadly, gypsum pore-filling can transiently reduce ionic transport coefficients even as the matrix deteriorates, meaning Dnssm alone may be a misleading indicator at intermediate acid concentrations; permeability and capillary absorption, not subject to this artefact, provide more reliable durability indicators and should be interpreted alongside chloride migration.

Permeability coefficient

As explained by^{35,38}, the flow rate of water passing through a crack varies depending on the width of the crack and the height of water head. Therefore, a crack permeability coefficient is widely used by many researchers to normalise or generalise the permeability of materials. Figure 12 demonstrates that permeability increases with acid severity, corroborating the progressive dissolution of gel and pore coalescence. The marked increase at 10% exposure indicates extensive decalcification, increased microcrack connectivity, and structural disintegration. Conversely, exposures between 0% and 5% maintain relatively lower permeability, suggesting that the geopolymer matrix remains largely intact or is only partially densified.

Notably, bio-GPC exhibits a 35–60% reduction in permeability compared to the control across all concentrations, with the most significant improvement observed at 5% exposure. This finding is consistent with reduced capillary absorption and is corroborated by SEM analysis revealing a denser gel structure and refined pore networks. Unlike the chloride migration coefficient, which at 5% is transiently reduced in control samples by gypsum pore-filling, permeability reflects structural matrix integrity more reliably at this timepoint, as it is not confounded by the temporary densification effect of expansive sulphate phases. These findings confirm that biomodification

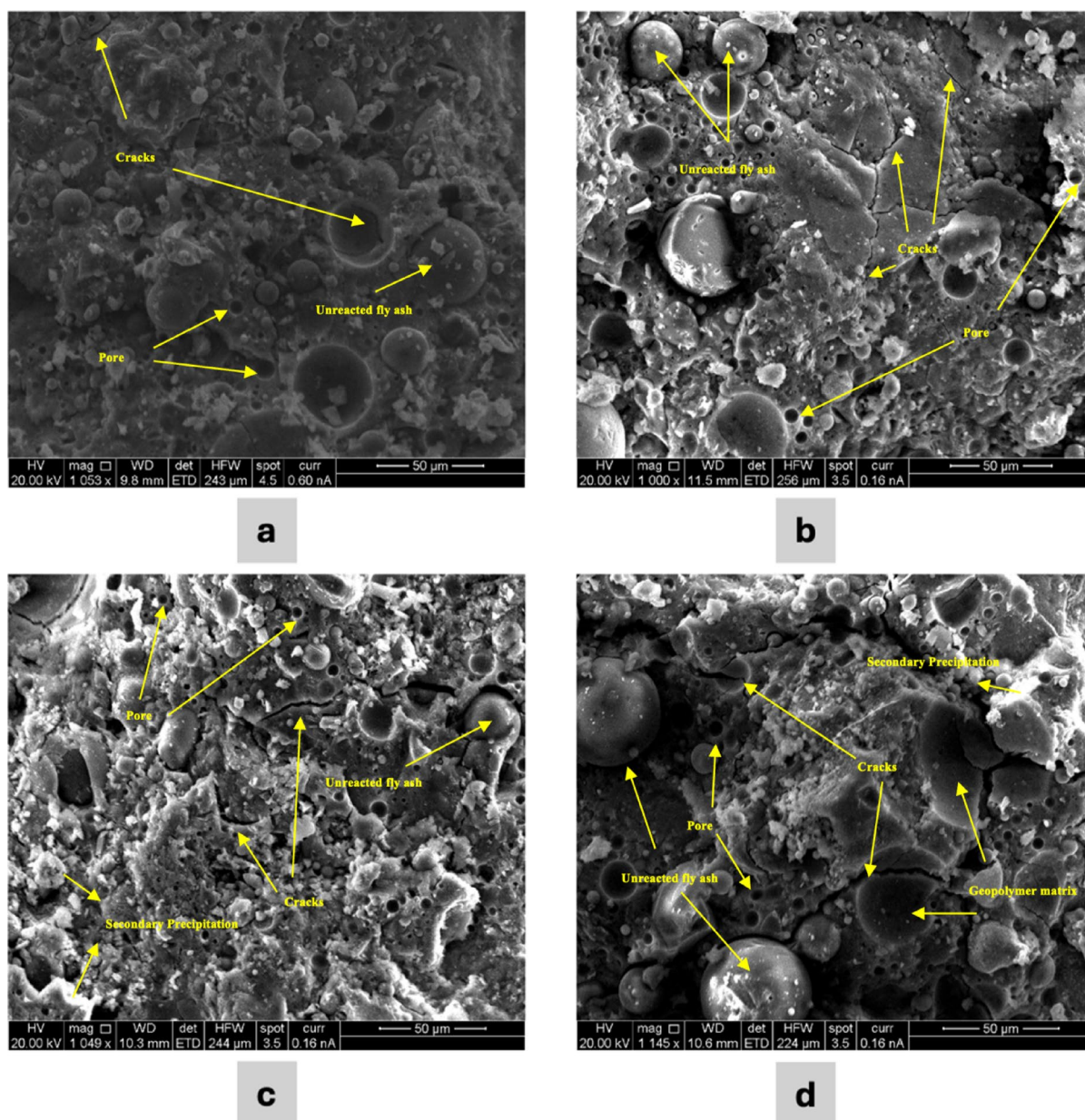
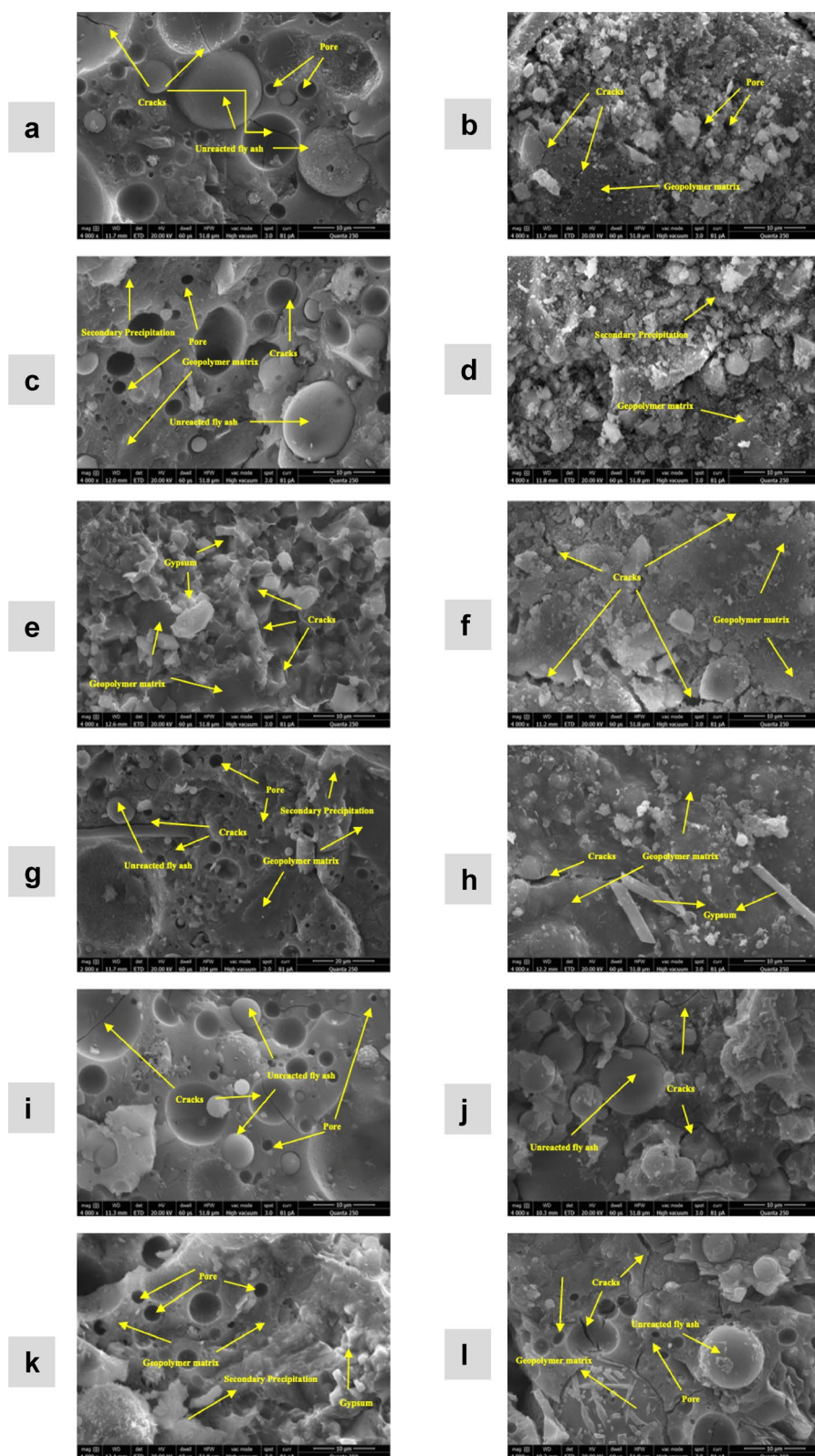


Fig. 10 Water absorption via capillary for GPC samples (at 60 days) immersed in acid, **(a)** 0% concentration, **(b)** 3% concentration, **(c)** 5% concentration, **(d)** 10% concentration.

enhances microstructural stability under mild to moderate acidity, consistent with threshold behaviour documented by Yi et al. (2011) and Shin et al. (2017)^{38,39} and with partial self-induced healing mechanisms reported by Lo Monte and Ferrara (2021) and Doostkami et al. (2023)^{44,45}, though whether these mechanisms would operate under dynamic sewer conditions was not investigated. The co-occurrence of lowest mass variation, lowest permeability, and competitive chloride migration in bio-GPC at 5% across three independent techniques strengthens confidence in the proposed mechanism.

Fig. 11 Chloride migration for GPC samples at 28 and 60 days (with/ without acid exposure) for different acid concentrations.



Materials microstructure

Table 4 illustrates that exposure to sulphuric acid induces distinct compositional variations within the geopolymer

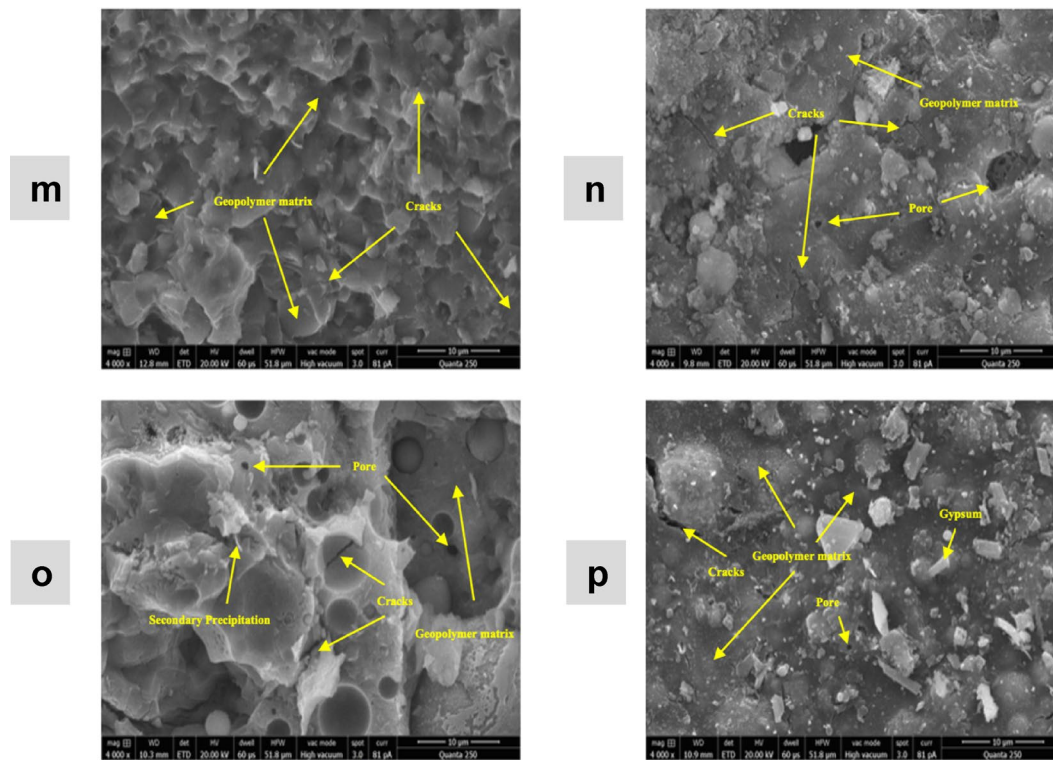


Fig. 11 (continued)

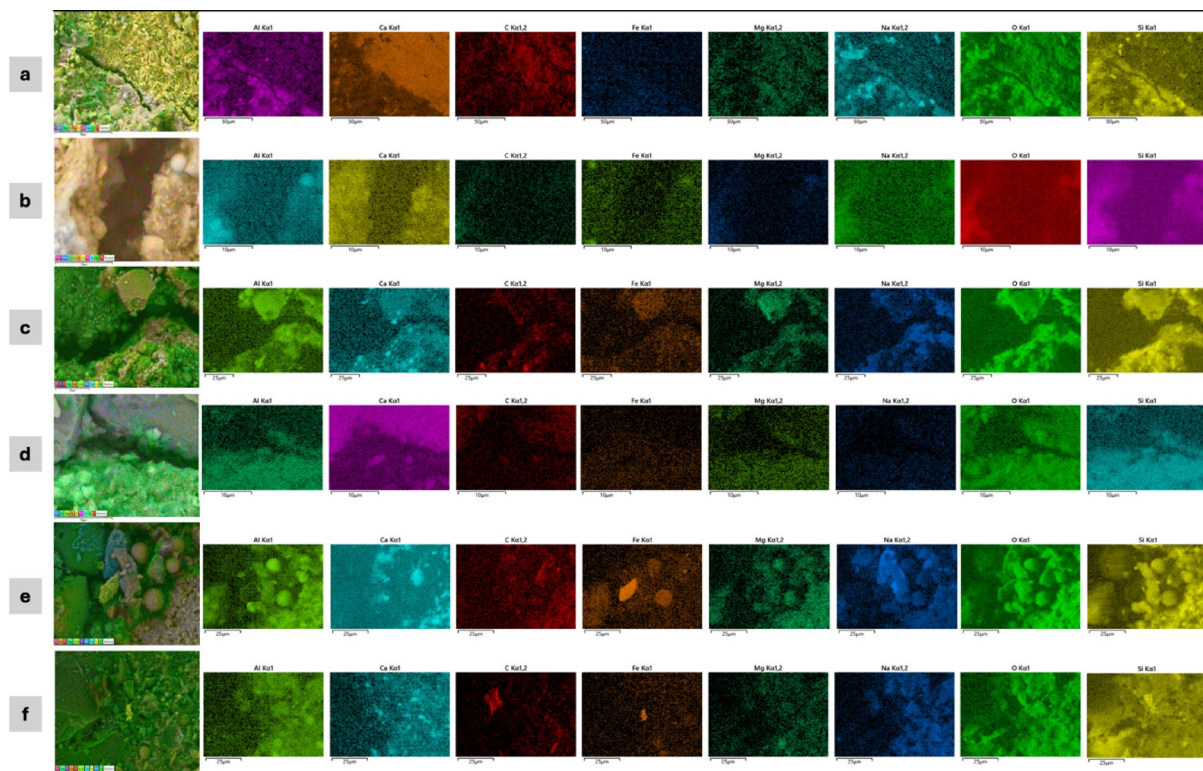


Fig. 12 Water permeability coefficient for GPC samples at 60 days (with/without acid exposure) for different acid concentrations.

matrix, with CaO trends closely mirroring the observed degradation and healing processes throughout the study. Bio-samples consistently exhibit higher CaO content at 0%, 3%, and particularly at 5% acid exposure, where CaO reaches a peak of 14.5 wt%, suggesting enhanced calcium-rich precipitation and stabilisation of calcium-bearing phases facilitated by biomodification. This is corroborated by their higher internal pH values (Fig. 6), reduced chloride migration (Fig. 11), decreased permeability (Fig. 12), and denser gel microstructures observed via SEM/EDS analysis.

The decline in CaO at 3% signals moderate decalcification, consistent with increased capillary absorption (Fig. 10b). At 10%, the control shows anomalously high CaO of 26.9 wt%, which is explained by four converging lines of evidence rather than analytical error: (i) SiO₂ collapses from 48.2 to 24.5 wt% and Al₂O₃ from 11.0 to 6.77 wt% in the same specimen (Table 4), confirming preferential Si-Al network dissolution that concentrates residual Ca phases in the surviving solid; (ii) XRD shows dominant gypsum peaks at 10% control (Fig. 2), meaning the reported CaO reflects gypsum accumulation rather than retained geopolymer calcium; (iii) EDS Ca at 10% control is only 1.63 wt% locally (Table 5), confirming Ca is concentrated in discrete sulphate deposits rather than uniformly distributed in the gel, the bulk XRF captures these deposits while EDS spot analysis does not; (iv) bio-samples at 10% retain SiO₂ of 46.3 wt% versus 24.5 wt% in controls, preserving the Si-Al framework and preventing the concentration effect, which explains their lower CaO of 10.7 wt% despite higher SO₃ of 9.19 wt%. It should be noted that, consistent with the XRF protocol applied across all conditions, the 26.9 wt% CaO value represents the mean of three independent XRF measurements on replicate specimens from this condition rather than a single reading, confirming its reproducibility within the experimental framework.

Together, these confirm that the elevated CaO represents a residual Ca-sulphate-enriched solid following selective Si-Al leaching, consistent with Ariyadasa et al. (2024)³⁰ and Yang et al. (2021)²⁸. This interpretation is further corroborated by the FTIR spectrum of the 10% control (Table 6), which shows near-absence of carbonate bands at ~1418 cm⁻¹ and very weak Si-O-T bands, consistent with advanced gel dissolution and gypsum-dominated residue rather than retained geopolymer calcium. Quantitative Rietveld refinement to determine precise gypsum and calcite phase fractions is recommended for future work.

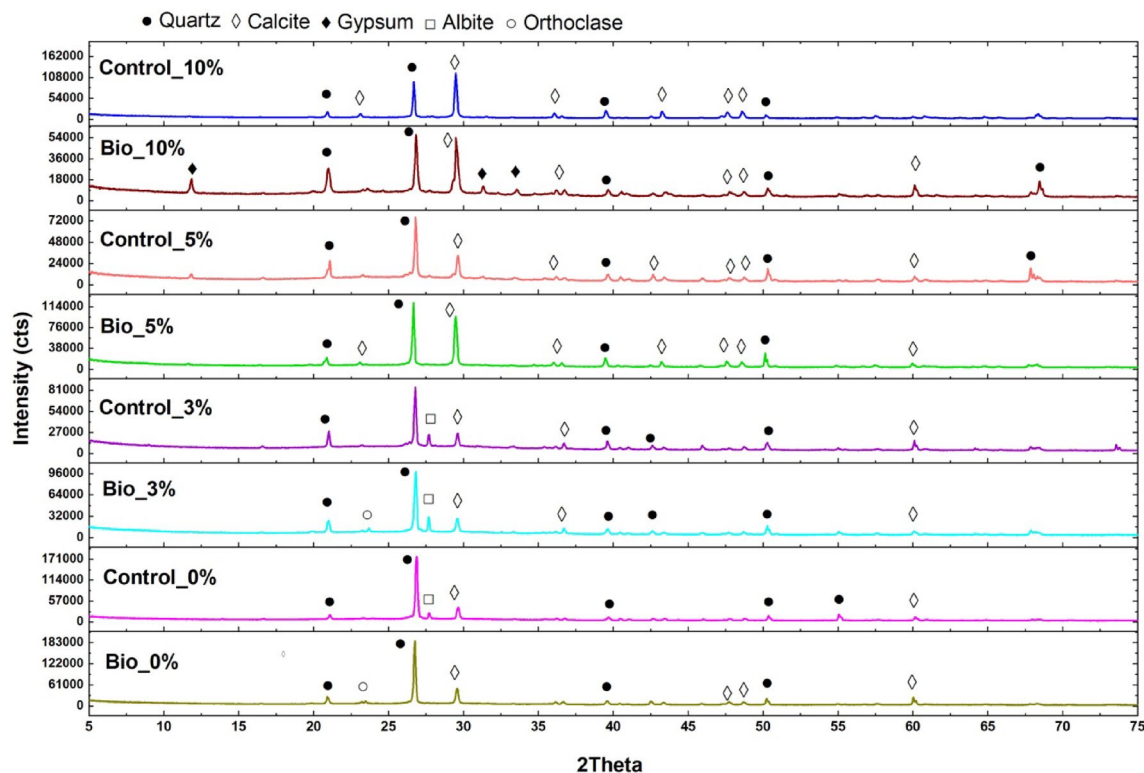
Figure 13 illustrates that, prior to acid exposure, both mixtures exhibit the typical FA-based geopolymer matrix with partially reacted particles embedded within the gel. However, the control specimens (a-b) demonstrate a higher degree of interconnected voids and visible microcracks, while the bio-modified specimens (c-d) display a more compact, dense matrix with fewer open pores. This initial pore refinement in bio-GPC accounts for its lower capillary absorption, reduced chloride ion migration, and decreased permeability observed in subsequent

Table 5 EDS elemental analysis after acid immersion for bio and control samples at different concentrations (wt%).

	Weight (%)							
	O	Mg	Al	Si	K	Ca	Ti	Fe
0% - Control inside	44.68	0.9	8.8	29.98	1.34	2.56	0.4	2.65
0% - Control outside	44.34	1.01	9.45	29.53	2.59	3.46	0.49	3.98
0% - Bio inside	47.13	1.4	8.92	23.75	1.39	8.12	0.33	2.5
0% - Bio outside	41.37	1.6	8.96	29.26	2.38	8.54	0.51	4.11
3% - Control inside	43.81	0.26	6.18	38.96	1.33	1.07	0.56	1.75
3% - Control outside	43.5	0.29	8.48	29.8	1.86	1.04	0.19	1.47
3% - Bio inside	48.49	0.79	6.91	23.05	1.27	8.17	0.19	1.59
3% - Bio outside	51.34	0.21	3.8	38.9	1.07	1.84	0.44	1.64
5% - Control inside	37.88	1.81	11.81	35.43	2.47	4.04	0.54	4.31
5% - Control outside	34.33	0.23	1.96	42.73	0.91	1.09	3.07	9.62
5% - Bio inside	42.92	0.95	9.64	27.5	1.84	8.22	0.63	3.55
5% - Bio outside	53.5	0.24	8.65	26.46	0.68	4.54	0.14	1.34
10% - Control inside	47.17	0.89	12.22	30.75	1.6	1.63	0.94	3.35
10% - Control outside	46.22	0.28	4.27	39.62	1.25	0.7	0.25	2.18
10% - Bio inside	48.51	0.91	6.64	24.25	1.24	7.79	0.32	1.93
10% - Bio outside	41.65	0.24	5.82	24.68	1.56	6.01	1	4.27

Table 6 FTIR spectral characteristics of control and bio GPC specimens across all acid exposure conditions, highlighting variations in aluminosilicate framework vibrations (Si-O-T, Al-O-Si), carbonates formed through Ca retention, and hydroxyl-related bands indicative of gel disruption.

Sample	Si-O/Al-O bending (~690–800)	Al-O-Si symmetric (~870–890)	Main Si-O-T band (~970–1030)	Carbonate band (~1415)	H-O-H bending (~1630–1650)	O-H stretching (3200–3600)	Notable observations
0% Control	691, 712, 777	874	991 (weak)	1418	1636	3226–3629	Strong polymerised network; minimal carbonate presence.
0% Bio	691, 712, 773	874	988 (weak)	1418	1634	3213–3606	Similar to control but with a slightly higher carbonate concentration, leading to early CaCO ₃ formation.
3% Control	688, 712, 777	874	992 (weak)	1418	1558 & 1646	3211–3625	Broader H-O-H band indicates increased gel disruption and moderate carbonate levels.
3% Bio	693, 712, 777	874	993 (weak)	1418	1634	3140–3615	Higher carbonate levels with a slight shift in O-H bonds lead to the precipitation of CaCO ₃ and a denser matrix.
5% Control	712, 773	874	995 (weak)	1418	1634–1698	3095–3604	Strong carbonate combined with expanded H-O-H leads to significant acid attack and decalcification.
5% Bio	693, 712, 777	874	990 (weak)	1418	1644	3149–3591	The carbonate levels increased with less gel distortion compared to the control, consistent with the ‘5% critical condition’ observed in other tests.
10% Control	(No 700–800 peaks recorded)	-	~1027 (very weak)	1405 (very low intensity)	weak 1600s	3211–3632	Severe network dissolution accompanied by carbonate consumption or absence leads to extensive leaching.
10% Bio	669, 712, 792	874	1027 (weak)	1418	1619–1683	3174–3606	The gel structure and carbonate retention are improved compared to the control; however, significant degradation remains.

**Fig. 13** Morphology of GPC samples without acid immersion at 60 days: (a) Control inside fragment, (b) Control outside fragment, (c) Bio inside fragment, (d) Bio outside fragment.

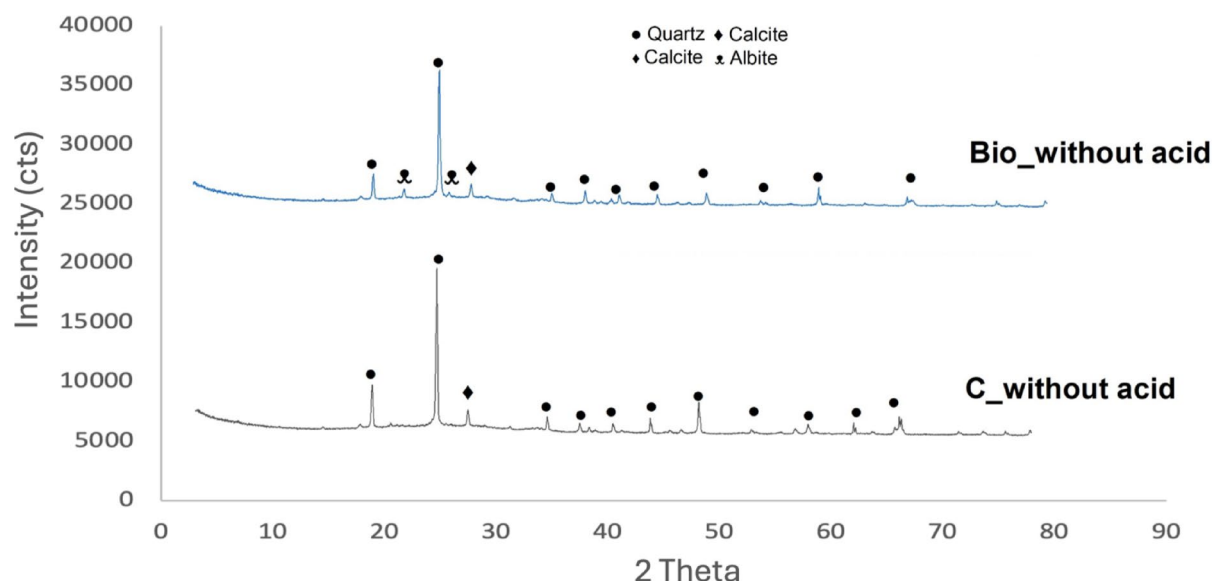


Fig. 14 Morphology of GPC samples after acid immersion: (a) 0% Control inside fragment, (b) 0% Control outside fragment, (c) 0% Bio inside fragment, (d) 0% Bio outside fragment, (e) 3% Control inside fragment, (f) 3% Control outside fragment, (g) 3% Bio inside fragment, (h) 3% Bio outside fragment, (i) 5% Control inside fragment, (j) 5% Control outside fragment, (k) 5% Bio inside fragment, (l) 5% Bio outside fragment, (m) 10% Control inside fragment, (n) 10% Control outside fragment, (o) 10% Bio inside fragment, (p) 10% Bio outside fragment.

analyses. The improved gel density and ITZ morphology provide a mechanistic explanation for the increased resistance of bio-modified mixes under 3–5% acid exposure, confirming that bacterial integration enhances matrix stability prior to aggressive attack.

Figure 14 documents progressive microstructural deterioration across all acid concentrations. At 0% and 3% (panels a–h), control specimens show discontinuous gel-FA interfaces, developing microcracks, and increasing inter-particle porosity. Bio-GPC counterparts display a denser, more continuous matrix with localised Ca-rich deposits at pore throats consistent with secondary mineral precipitation, explaining the lower capillary absorption and permeability in bio-modified specimens at mild concentrations. At 5% (panels i–l), control specimens show the most pronounced etching, widened cracks, and gypsum-driven pore coarsening, while bio-GPC maintains comparatively refined pore structures with dense secondary phase zones, the microstructural contrast that directly corresponds to the maximum performance differential between the two mixes. At 10% (panels m–p), both systems show severe gel dissolution and extensive open porosity; bio-GPC retains slightly fewer interconnected voids and lower crack density than controls, consistent with its marginally superior transport properties despite advanced degradation. It should be noted that individual bacterial cells cannot be unambiguously identified at the magnifications employed; the localised Ca-rich deposits in bio-GPC panels are identified as secondary mineral phases on the basis of EDS elemental analysis rather than direct cell imaging.

Table 5 shows that acid exposure primarily alters Ca and O contents. Bio samples recorded consistently higher Ca concentrations of approximately 8–9 wt% at 0–5% exposure compared to approximately 2–4 wt% in control samples. This difference is consistent with enhanced calcium retention and secondary precipitation processes, though it does not in itself confirm the mechanism driving this retention. The observed Ca enrichment is independently corroborated by SEM evidence of a denser gel matrix, refined pore structure, and improved ITZ morphology, providing a coherent structural basis for the reduced capillary absorption and permeability in bio-GPC. The maximum Ca enrichment is observed at 5% acid exposure, suggesting this as the optimal regime for densification with the lowest transport coefficients. At 10% exposure, although Ca levels remain elevated in bio-treated samples, microstructural deterioration becomes predominant, implying that dissolution and sulfate formation

surpass the protective effects. Overall, EDS confirms that biomodification stabilises Ca-rich phases under mild to moderate acidity, thereby supporting enhanced transport properties.

Figure 8 presents EDS elemental maps recorded at 180 days, 120 days beyond the immersion endpoint, confirming that elemental differentiation between bio-modified and control specimens persists beyond the study window. The unexposed control exhibits more Ca-rich regions, while the unexposed bio sample shows a higher Si-Al distribution and lower Ca, indicating the formation of a more polymerised aluminosilicate gel and a more uniform matrix. Post-exposure, bio samples at 0% retain a stable Si-Al framework with limited Ca redistribution, confirming their structural stability. At 3% and particularly 5%, Ca becomes locally concentrated, while Si decreases, evidencing controlled decalcification followed by secondary Ca-rich precipitation that partially occludes pores. The appearance of sulphur at 5% indicates initial sulphate formation without causing complete gel disintegration. This accounts for the observed reduction in permeability and transport coefficients under moderate acidity. At 10%, the redistribution of sulphur and alkali elements intensifies, and elemental heterogeneity becomes more evident, reflecting predominant gel dissolution and sulphate-induced degradation. Thus, EDS mapping confirms that bio-modified specimens retain distinct elemental distributions under mild to moderate acidity (0–5%), consistent with the proposed pore refinement and Ca-stabilisation mechanism, whereas severe acid exposure surpasses this effect regardless of mix type.

Critically, the persistence of distinct Ca-rich localised zones and maintained Si-Al framework in bio-GPC at 180 days, compared to the more homogeneous elemental distribution in control specimens, confirms that the elemental differences between bio-modified and control specimens are not confined to the 60-day immersion window. This does not indicate whether bacteria remained metabolically active during this period; it demonstrates only that the chemical legacy of early-age biomineralisation persists at 180 days. Whether this reflects ongoing bacterial activity or the durable imprint of precipitation that occurred during the curing and immersion phases cannot be determined from elemental mapping alone.

Chemical composition

X-ray diffractometer

Figure 2 illustrates distinct mineralogical responses as acid severity increases. Peak assignments follow standard reference patterns: the broad amorphous hump at 20–35° 2 θ is characteristic of the N-A-S-H geopolymer gel network; quartz reflections appear at 26.6° and 20.8° 2 θ ; feldspar (albite) at 27.9° 2 θ ; calcite (CaCO₃) at 29.4° 2 θ ; and gypsum (CaSO₄·2 H₂O) at 11.6° and 20.7° 2 θ . At 0% and 3%, both bio and control mixes display the characteristic amorphous hump alongside quartz and feldspar phases; bio samples exhibit more pronounced calcite reflections at 29.4° 2 θ , indicating enhanced Ca retention and secondary CaCO₃ formation. At 10%, both mixtures show dominant gypsum peaks and diminished amorphous content, indicative of severe sulphate attack and gel dissolution. Gypsum reflections are more substantial in control samples, while bio samples retain weaker yet detectable calcite, implying limited residual Ca stabilisation.

The mineralogical shift from more prominent calcite reflections at 5% to dominant gypsum peaks at 10% is consistent with the proposed performance threshold, though it does not in itself establish the underlying mechanism. At 5%, the stronger calcite reflections in bio-GPC relative to control samples support the interpretation that a carbonate precipitation pathway is active at this concentration. At 10%, dominant gypsum peaks in both systems are consistent with sulphate phase formation overwhelming calcite precipitation capacity, though the specific kinetic and thermodynamic conditions driving this transition cannot be confirmed from qualitative XRD data alone. The tenfold increase in SO₃ content in bio-samples from 1.38 wt% at 5% to 9.19 wt% at 10% (Table 4) is consistent with this proposed transition.

Figure 3 demonstrates that both control and bio mixtures retain the primary FA-derived phases, such as quartz and albite, along with the characteristic amorphous hump at 20–35° 2 θ , thereby confirming successful geopolymerisation. Notably, the bio sample displays more prominent calcite peaks, while calcite in the control is weak or

nearly absent. This suggests that bio-induced CaCO_3 precipitation and enhanced calcium stabilisation occur prior to acid exposure, leading to a more cohesive gel network. The increased amorphous content and stronger calcite reflections in bio-GPC provide a structural basis for its lower transport coefficients and superior resistance when subjected to subsequent 3–5% acid attack. Consequently, even prior to chemical attack, XRD analysis confirms that biomodification refines the matrix and enhances the initial chemical stability.

FTIR

FTIR analysis (Table 6) confirms progressive depolymerisation of the aluminosilicate network as acid severity increases. Peak assignments are as follows: Si-O/Al-O bending vibrations at $690\text{--}800\text{ cm}^{-1}$; Al-O-Si symmetric stretching at $870\text{--}890\text{ cm}^{-1}$; the main Si-O-T asymmetric stretching band at $970\text{--}1030\text{ cm}^{-1}$, which is the primary indicator of aluminosilicate network integrity and shifts to higher wavenumbers with progressive gel depolymerisation; carbonate stretching at $\sim 1418\text{ cm}^{-1}$, indicating CaCO_3 formation; H-O-H bending at $1630\text{--}1650\text{ cm}^{-1}$ reflecting adsorbed water; and broad O-H stretching at $3200\text{--}3600\text{ cm}^{-1}$ associated with hydroxyl groups and gel-bound water.

In control samples, the Si-O-T band diminishes and broadens from 3% to 10% exposure, indicating gel break-down; at 10%, carbonate peaks are near-absent while O-H bands intensify, reflecting extensive decalcification. Conversely, bio-GPC exhibits more consistent Si-O-T peak positions and stronger carbonate bands ($\sim 1418\text{ cm}^{-1}$), demonstrating CaCO_3 precipitation and improved Ca retention. This stabilisation is particularly evident at 3% and 5% acid treatments, where aluminosilicate bands remain clearly defined and carbonate signals persist, suggesting delayed depolymerisation and better gel preservation. At 10%, both systems experience considerable degradation; however, bio samples maintain slightly stronger carbonate features, indicating some resistance to severe attack. These results confirm that biomodification improves network stability and carbonate retention under mild to moderate acidity (3–5%), whereas high acid exposure (10%) exceeds the protective capacity.

Conclusion

This study offers an assessment of the durability and microstructural development of fly ash-based geopolymer concrete (GPC), both with and without *Shewanella*-based biomodification, subjected to simulated sewer acid environments containing 0–10% H_2SO_4 . Results show that *Shewanella*-modified GPC exhibited consistently better durability performance than control specimens under mild to moderate sulphuric acid exposure (3–5%), conditions representative of biogenic sulphuric acid corrosion in sewer systems^{12,46}. Across durability indicators, bio-GPC consistently outperformed the control in permeability, capillary absorption, internal pH retention, mass stability, and microstructural cohesion. Chloride migration coefficients showed the same trend at all conditions except 5% acid, where transient gypsum pore-filling in control samples produced a marginally lower D_{nssm} at 60 days, a temporary effect inconsistent with the simultaneously higher permeability, lower pH, and greater micro-cracking observed in those specimens. These improvements are consistent with Ca-rich biomineral precipitation, partial pore refinement, and slower gel depolymerisation as proposed underlying mechanisms, though direct bacterial viability verification was not conducted in this study.

A clear concentration-dependent trend was identified across all durability indicators: bio-GPC performance improvement relative to control specimens was progressive from 0% to 5% acid exposure, reaching maximum stabilisation at 5%, before diminishing substantially at 10%. This trend reflects the competition between two mineralogical pathways: at 3–5% H_2SO_4 , sulphate activity remains insufficient to drive preferential gypsum formation, allowing the stabilising calcite precipitation pathway to dominate and producing the lowest transport coefficients, highest internal pH retention, and greatest mass stability observed in the study. At 10% H_2SO_4 , dissolved SO_4^{2-} concentration is sufficient to redirect Ca^{2+} toward expansive gypsum crystallisation rather than CaCO_3 formation, overwhelming the biomodification benefit and producing degradation patterns, dominant gypsum XRD peaks,

tenfold SO_3 increase, severe microcracking, comparable between bio and control specimens. The diminishing improvement at 10% therefore reflects a thermochemical threshold rather than a failure of the biomodification concept: within the pH range representative of active sewer crown corrosion (pH 0.5–4, approximately 1–5% H_2SO_4), bio-GPC consistently demonstrated superior performance. This interpretation is proposed on the basis of converging qualitative evidence and should be regarded as a plausible mechanistic explanation rather than a demonstrated mechanism, pending quantitative phase analysis. Overall, the bio-modified specimens consistently outperformed controls across durability indicators within realistic sewer acidity ranges. The convergence of XRD, XRF, EDS, FTIR, SEM, and transport evidence is consistent with a biomineralisation interpretation, though this remains a proposed mechanism pending direct bacterial viability and activity verification.

Under the controlled laboratory conditions investigated, microbial biomineralisation is associated with improved acid resistance within the H_2SO_4 concentration range tested (0–10%), with the most consistent improvements observed at 3–5%^{11,47}. These findings establish a laboratory-scale performance baseline for bio-modified fly ash geopolymers. Whether these improvements would translate to real sewer environments, where bacterial viability, biofilm competition, wet-dry cycling, and continuously generated biogenic acid were not investigated, cannot be determined from the present data and requires targeted field validation before any practical implementation conclusions can be drawn^{48–50}. Several limitations should be noted. Static mineral acid immersion, while the established first-tier methodology in geopolymer acid resistance research^{28–31,35}, differs from real sewer corrosion in four important respects: (i) biogenic H_2SO_4 is generated continuously by sulphur-oxidising bacteria, creating dynamic pH gradients more locally severe than bulk immersion; (ii) surface biofilm formation in real sewers creates diffusion barriers and competing microbial communities absent from our specimens; (iii) wet-dry cycling imposes salt crystallisation stresses not replicated under constant immersion; and (iv) the evolving acidity of real sewer environments means our fixed-concentration results may overestimate durability at mild acid concentrations where acidity would intensify progressively. Grengg et al. (2020)¹¹ demonstrated directly that dynamic MIC conditions produce more heterogeneous and accelerated degradation than mineral acid immersion predicts. Our results should therefore be interpreted as quantifying the chemical acid resistance of bio-modified GPC under controlled conditions, providing a necessary baseline for comparison, rather than as a direct prediction of field performance.

The 60-day study duration represents a further limitation. While consistent with the established range for geopolymer acid resistance studies^{28,31,32} and sufficient to characterise early-to-medium term degradation mechanisms and performance rankings, it cannot support direct extrapolation to decadal infrastructure service life. Three specific long-term uncertainties follow: (i) direct bacterial viability measurements beyond the curing period were not finalised yet (at the moment we are measuring the oxygen consumption inside the concrete as detecting localised changes in O_2 proves the bacteria are metabolically active. The technique will be published soon); EDS elemental maps at 180 days (Fig. 8) confirm that elemental differentiation between bio and control specimens persists at this timepoint, demonstrating the durability of the chemical legacy of early-age biomineralisation, but providing no information on whether bacteria remained metabolically active beyond the immersion period. Two opposing long-term scenarios are plausible: sustained precipitation leading to progressive pore occlusion and stabilisation, or declining bacterial activity leading to accelerating mass loss as acid ingress overcomes the depleted calcite protection; (ii) gypsum formation identified at 5–10% acid exposure is expansive over longer durations and would progressively increase permeability beyond the 60-day snapshot, potentially narrowing the bio-control performance gap; and (iii) the 5% threshold may shift as matrix buffering capacity decreases with continued acid exposure. Patrisia et al. (2024)³¹ demonstrated that geopolymer degradation mechanisms identified at early ages do not always persist linearly over 18 months, underscoring the need for extended testing. Direct in-matrix bacterial activity measurements remain an additional priority for future work. It should further be noted that a TSB-only control, containing the growth medium without bacteria, was not included in the experimental design. Although the equivalence of XRD and FTIR characteristics between bio and control specimens prior to acid exposure suggests TSB addition did not substantially alter primary geopolymerisation chemistry, the organic components of the growth medium cannot be formally excluded as a contributing factor to the observed

performance improvements. Future work should include a TSB-only condition to isolate bacterial metabolic contributions from medium effects. Future work should prioritise closing the gap between these laboratory findings and field conditions through bioreactor-simulated MIC exposure incorporating sulphur-oxidising bacterial communities, cyclic wet-dry acid exposure, quantitative Rietveld XRD analysis, and bacterial viability monitoring at multiple timepoints. Only following such validation would assessment of scalability, cost-effectiveness, and compatibility with rehabilitation techniques such as spray-lining and CIPP systems be meaningful.

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Data availability Data will be available upon request.

Declarations

Competing interest The authors declare no competing interests.

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