ORIGINAL ARTICLE



# Enhancing microstructural properties and chloride resistance of seawater-mixed steel fiber-reinforced mortars through glass powder modification

Xiaowen Zhang · Jian-Xin Lu · Chi Sun Poon

Received: 2 March 2025 / Accepted: 14 April 2025 / Published online: 3 May 2025 © The Author(s) 2025

Abstract This study investigates how glass powder modification enhances steel fiber-reinforced mortar (SFRM) performance in marine environments, with focus on microstructural properties and chloride resistance. We developed a customized image segmentation technique that combines K-means clustering with concentric strip analysis to characterize the fiber-matrix interface. Results showed that replacing 25% of cement with glass powder reduced the interfacial transition zone thickness from 45 to 35 µm and decreased surface porosity from 95 to 85% after 28-d curing. The pozzolanic reactions generated additional C-S-H phases and modified phase assemblages, enhancing chloride resistance and increasing interfacial microhardness by 183.3 MPa. During wet-dry testing, glass powder-modified specimens showed 15-25% lower chloride concentrations and delayed corrosion initiation by 3-6 cycles in seawater-mixed specimens. This work provides both a testing methodology for fiber-matrix interface analysis and practical guidelines for improving SFRM durability in marine construction through waste glass utilization.

X. Zhang  $\cdot$  J.-X. Lu  $\cdot$  C. S. Poon ( $\boxtimes$ )

Department of Civil and Environmental Engineering, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong

e-mail: cecspoon@polyu.edu.hk

**Keywords** Steel fiber-reinforced mortar · Glass powder · Interfacial transition zone · Quantitative image segmentation · Chloride resistance · Microstructural characterization

#### 1 Introduction

Concrete and mortar remain fundamental materials in modern construction, serving critical roles in everything from structural elements to finishing applications. The scale of their global usage is staggering-in 2021 alone, global cement production was estimated to have reached 4.1 billion tons, with annual concrete consumption seven times higher [1]. This massive consumption pattern could raise significant sustainability concerns, particularly regarding freshwater usage and cement production in coastal regions and remote islands [2]. These challenges have driven researchers to seek sustainable alternatives and innovative solutions for construction materials like waste material utilization and alternative mixing water sources.

Waste glass recycling presents a critical environmental challenge, with global recycling rates stagnating at 21% of the 27 million tonnes produced annually [3]. This issue is particularly acute in densely populated urban centers like Hong Kong, where daily waste glass generation exceeds 300 tonnes with recycling rates below 20% [4]. While traditional recycling faces logistical and economic barriers,



incorporating glass powder as a supplementary cementitious material offers a sustainable alternative that potentially enhances concrete performance [5-10]. Unlike traditional supplementary cementitious materials, glass powder offers unique advantages including superior acid resistance with 45% glass powder showing 15% lower mass loss than control specimens [11], distinctive microstructural improvements through fibrillar hydration products, and enhanced flexural strength compared to fly ash [12]. Glass powder provides sustainability benefits by utilizing post-consumer waste with limited recycling options (< 20%), whereas the future availability of fly ash and blast furnace slag may be constrained by reduced coal power generation and changing steel manufacturing processes. The formation of Si/Al-rich residue in glass powder mortars creates a protective barrier against acid ions, enhancing durability in aggressive environments [11]. The effectiveness of glass powder in cementitious systems depends critically on particle size distribution and replacement ratios. Recent studies indicate that particles below 25 µm exhibit high pozzolanic reactivity [13-15] and replacement levels between 20 and 30% yield optimal results for strength development and durability enhancement [11, 16–18]. These characteristics become particularly relevant when considering aggressive exposure environments, such as marine construction where seawater mixing is increasingly considered.

The use of seawater in concrete mixing, driven by freshwater scarcity in coastal regions, introduces both opportunities and challenges. The presence of dissolved salts affects not only the hydration kinetics but also the microstructural development of the cementitious matrix [19-21]. For example, chloride ions in seawater accelerate cement hydration, leading to increased early-age strength of the materials [12, 19, 22]. While these effects can be beneficial for certain applications, the higher chloride content raises durability concerns, particularly regarding reinforcement corrosion. These durability considerations have led researchers to explore various material combinations that might mitigate potential negative effects while harnessing the benefits of seawater. One promising direction involves the incorporation of steel fibers. These fibers, typically ranging from 0.2 to 1.0 mm in diameter and 10-60 mm in length, provide three-dimensional reinforcement throughout



the matrix [23–26]. Research has demonstrated their ability to improve flexural strength, impact resistance, and fatigue performance [27-33]. The dispersed nature of fiber reinforcement potentially offers better corrosion resistance compared to traditional steel bars, as individual fibers are less likely to form continuous electrical paths [34, 35]. Individual fibers typically remain electrically isolated, preventing the formation of macrocells that accelerate corrosion in traditional reinforcement [36]. Additionally, seawater-mixed concrete with appropriate supplementary cementitious materials can be strategically applied in specific marine components with limited structural requirements or as protective layers for conventionally reinforced elements. This approach leverages the improved mechanical properties while minimizing corrosion risk through targeted application strategies.

Studies have shown that fiber-matrix interactions are influenced by the ionic composition of mixing water [37], affecting both mechanical properties and durability performance. However, the combined effects of glass powder incorporation and seawater mixing on steel fiber-reinforced cementitious systems remain largely unexplored, presenting an important research gap in sustainable construction materials.

Previous research [38] has demonstrated that appropriate pozzolanic materials can effectively bind chloride ions in stable phases, significantly reducing their mobility. Glass powder, as such a pozzolanic material, may enhance this binding capacity by forming phases with high chloride adsorption potential, thus minimizing the risk of salt redistribution. This research addresses these knowledge gaps through a systematic investigation of glass powder modification effects in seawatermixed steel fiber-reinforced mortars (SFRM). The primary objectives are to: (1) develop and validate a novel quantitative methodology for characterizing fiber-matrix interfaces; (2) elucidate the mechanisms by which glass powder modification affects interfacial microstructure and properties; (3) quantify the combined effects of glass powder and seawater mixing on chloride transport and binding mechanisms; and (4) develop recommendations for optimizing glass powder usage in steel fiberreinforced mortars based on laboratory findings while acknowledging the limitations of scale and the need for field validation. Through advanced microstructural analysis techniques and systematic property evaluation, this study provides fundamental insights into interface engineering of cementitious composites while addressing practical challenges in sustainable marine construction. The findings contribute to both theoretical understanding of complex cementitious systems and practical solutions for enhancing concrete durability in aggressive environments.

### 2 Materials and methods

### 2.1 Materials

#### 2.1.1 Cement and glass powder

The cement used was CEM I ordinary Portland cement (strength class 52.5 N, Green Island Hong Kong Co. Ltd.) complying with BS EN 12350. The chemical composition and physical properties of the cement are provided in Table 1. Following the established protocol [12], glass powder was prepared from waste glass beverage containers (mixed colors). After removing contaminants through tap water washing and air-drying, the glass was crushed and ground in a laboratory ball mill (D 206 mm, L 330 mm, Speed 60 rpm, Ball-to-material ratio 6:1). The resultant glass powder appeared white in color with a measured average particle size of 22.3 µm.

 Table 1
 Chemical compositions and physical properties of the cement (wt.%)

Compositions	wt.%
SiO <sub>2</sub>	21.2
Al <sub>2</sub> O <sub>3</sub>	5.10
CaO	67.9
ИgO	1.21
K <sub>2</sub> O	0.12
$P_2O_5$	0.12
Fe <sub>2</sub> O <sub>3</sub>	0.21
50 <sub>3</sub>	4.12
Physical properties	
Density (g/cm <sup>3</sup> )	3.03
Average particle size (µm)	17.7
Average particle size (µm)	

#### 2.1.2 Steel fibers

Commercially sourced smooth steel fibers with a diameter of 0.20 mm and a length of 13 mm were adopted. The fibers had a nominal tensile strength of 1100 MPa and elastic modulus of 200 GPa.

## 2.1.3 Seawater

Artificial seawater was prepared according to ASTM D1141-98 (2013) to eliminate variables from organic matter and dissolved gases. The major salt compositions are shown in Table 2.

#### 2.2 Mix proportions and specimen preparation

Eight mortar mixtures were designed using a factorial experimental approach, incorporating two waterto-binder ratios (w/b: 0.45 and 0.30, designated as medium (M) and high (H) quality respectively), two water types (freshwater and seawater), and two binder compositions (with/without glass powder). The selected w/b ratios 0.45 and 0.30 represent typical values used in medium-quality and high-quality marine concrete applications, respectively. These values allow for assessment of glass powder modification effectiveness across different concrete quality levels commonly encountered in practical marine construction.

Glass powder replaced 25% of cement by mass in selected mixtures. The sand-to-binder ratio was fixed at 3:1 by volume. Steel fibers were incorporated at 2% by volume of binder. A polycarboxylate-based superplasticizer (ADVA-109, W.R. Grace) was used in H-series mixtures to maintain workability. The detailed mix proportions are shown in Table 3.

Tab	ole 2	Chemical	compositions	of	artificial	seawater
-----	-------	----------	--------------	----	------------	----------

Compound	Concentration (g/L)			
	(8,2)			
NaCl	24.53			
MgCl <sub>2</sub>	5.20			
Na <sub>2</sub> SO <sub>4</sub>	4.09			
CaCl <sub>2</sub>	1.16			
KCl	0.695			
NaHCO <sub>3</sub>	0.201			
KBr	0.101			

Table 3Mix proportionsof mortar (kg/m³)

	Freshwater	Seawater	Cement	Glass power	Sand	Steel fiber	Superplasticizer
F-M	235	0	523	0	1569	85	0
F–H	170	0	567	0	1702	85	17
FG-M	232	0	387	129	1550	85	0
FG-H	168	0	420	140	1680	85	16.8
S-M	0	235	523	0	1569	85	0
S–H	0	170	567	0	1702	85	17
SG-M	0	232	387	129	1550	85	0
SG-H	0	168	420	140	1680	85	16.8

Mix designations use a two-part system: the first part indicates water type (F=freshwater, S=seawater) and glass powder inclusion (FG/SG=with glass powder), while the second part denotes quality level (M=medium quality, H=high quality). The mixing procedure involved combining the binder components with steel fibers and sand in a mechanical mixer, followed by water addition to achieve uniform consistency. Fresh mortar was cast into  $40 \times 40 \times 160$  mm steel molds and vibrated for 15 s on a laboratory shaker. Plastic sheeting covered the molds to prevent moisture loss. After 24 h, specimens were demolded and water-cured at 23 °C for 28 days.

# 2.3 Exposure conditions

After 28-day curing, specimens underwent cyclic wetting–drying exposure following modified ASTM G109 procedures. Each cycle comprised 2 days of immersion in 5% NaCl solution at  $40\pm2$  °C followed by 4 days of drying at  $23\pm2$  °C and  $50\pm5\%$  RH. Solution pH and chloride concentration were monitored weekly and adjusted as needed. Three specimens per mixture were tested at 0, 5, and 15 cycles for comprehensive analysis.

# 2.4 Analytical methods

# 2.4.1 Sample preparation for microstructural analysis

Specimens were sectioned using a precision diamond saw under water cooling to obtain  $20 \times 20 \times 8$  mm samples. Cutting locations were standardized as shown in Fig. 1 to minimize variation. For scanning electron microscopy (SEM) analysis, samples underwent sequential grinding (SiC papers: 320, 500, 800,



Materials and Structures (2025) 58:143

1000 grit), ethanol immersion (48 h), and vacuum desiccation. This preparation protocol was selected to preserve the interfacial microstructure while creating suitable surfaces for SEM analysis. Sequential grinding rather than aggressive polishing minimized artificial disruption of the interface. Ethanol immersion halted hydration reactions without the microcracking associated with acetone replacement, while vacuum desiccation prevented the formation of carbonation products that could interfere with interface analysis. Validation of microstructural preservation was performed by analyzing multiple samples from each specimen with consistent results across different preparation batches.

# 2.4.2 Microstructural characterization

Microstructural analysis was conducted using a Tescan VEGA3 scanning electron microscope equipped with an Oxford Instruments X-Max 50 energy dispersive X-ray spectroscopy (EDS) detector. Operating parameters were standardized: accelerating voltage 20 kV, working distance 15 mm. At least 20 images were captured per specimen with 3 cubes analyzed per mix proportion to ensure statistical reliability. EDS analysis utilized standardized collection parameters (acquisition time: 100 s, process time: 5, dead time: 30–40%).

# 2.4.3 Image analysis methodology

Traditional image analysis methods for cementitious materials often rely on threshold-based segmentation or manual delineation, which present significant challenges when analyzing complex fiber–matrix interfaces. These methods typically struggle with distinguishing subtle grayscale





(b)

variations in the interfacial transition zone and often lack reproducibility in quantifying phase distributions around steel fibers. Moreover, existing techniques rarely account for the radial distribution of properties from the fiber surface, limiting their ability to characterize gradual microstructural transitions.

To address these limitations, a customized image segmentation method was developed to quantitatively analyze the SEM images of steel fiber-reinforced mortar microstructure. The analysis procedure integrated preprocessing, phase segmentation, and interface characterization techniques to evaluate the distribution and properties of different phases within the composite system. For each mix proportion, 3 cubes were analyzed with at least 20 SEM images captured per specimen. Sampling locations were selected using a systematic grid approach to ensure unbiased representation of the interfacial zones.

The analysis began with image preprocessing, where SEM images were converted to grayscale and optimized using a  $5 \times 5$  Gaussian blur filter to reduce noise while preserving structural boundaries. Phase segmentation was achieved through K-means clustering, enabling the identification of distinct material phases including steel fibers, cement matrix, voids, and sand particles. This unsupervised learning algorithm grouped image pixels based on intensity values, providing objective classification of microstructural features. The segmented images underwent morphological operations using a  $5 \times 5$ rectangular kernel for boundary enhancement and noise suppression. The absolute difference between dilated and eroded images was calculated





Fig. 2 Sequential image processing methodology. a Original image. b Preprocessed image. c Segmented image. d Boundary image. e Region of interest. f Filled area on original image

to generate precise boundary delineation of the distinct regions. The methodology comprises the following sequential steps, as illustrated in Fig. 2:

- Image preprocessing (Fig. 2a→b): SEM images were converted to grayscale and optimized using a 5×5 Gaussian blur filter to reduce noise while preserving structural boundaries.
- 2. Phase segmentation (Fig.  $2b \rightarrow c$ ): K-means clustering identified distinct material phases including steel fibers, cement matrix, voids, and sand particles. This unsupervised learning algorithm grouped image pixels based on intensity values, providing objective classification of microstructural features.
- 3. Boundary enhancement (Fig.  $2c \rightarrow d$ ): Segmented images underwent morphological operations using a 5×5 rectangular kernel for boundary enhancement and noise suppression. The absolute difference between dilated and eroded images was calculated to generate precise boundary delineation.
- 4. Interface analysis (Fig. 2d→e, f): Steel fiber boundaries were determined through contour extraction with a threshold value of 50, selecting the largest contour as the primary region of interest. The surrounding area was analyzed using concentric strips of 5 µm width extending from the fiber surface. Within each strip, porosity was calculated as the ratio of void pixels to total pixels.



Interface analysis focused on regions surrounding steel fibers. The steel fiber boundaries were determined through contour extraction with a threshold value of 50, selecting the largest contour as the primary region of interest. The surrounding area was analyzed using concentric strips of 5 µm width extending from the fiber surface. Following the established protocol by Scrivener et al. [39], the interface transition zone was divided into several parallel strips for detailed analysis. Within each strip, porosity was calculated as the ratio of void pixels to total pixels, enabling quantitative assessment of interfacial transition zone properties. For porosity profiling, the method analyzed successive 5 µm strips from the steel fiber surface outward, generating detailed distribution patterns of pore structures. This approach allowed precise determination of the interface transition zone thickness by identifying the point where microstructural properties stabilized. The method's reliability was enhanced through multiple image analysis per specimen, with results validated across different sampling locations. This systematic approach provided consistent quantification of microstructural features, particularly the porosity distribution patterns in the fiber-matrix interface. The practical application of this methodology is illustrated in Fig. 3, where successive 5  $\mu$ m strips clearly show the gradual transition in microstructural properties from the fiber surface outward.

#### 2.4.4 Free chloride analysis

Free chloride content was determined following the AFREM procedure [40]. Specimens were sliced at 10 mm intervals using precision saw, powder was collected through controlled drilling with depth 2 mm and ground to <315  $\mu$ m, and 5 g powder was extracted with 200 mL deionized water. The filtered solution was stabilized with 2 mL nitric acid (53%) before potentiometric titration against silver nitrate.

#### 2.4.5 Microhardness testing

Microhardness measurements were performed using a Vickers HMV-G21 microhardness tester with a load of 0.98 N (HV0.1) and dwell time of 15 s. Indentations were made at 5  $\mu$ m intervals from the fiber-matrix interface up to 50  $\mu$ m into the matrix. The selected load of 0.98 N was determined through preliminary testing to be optimal for the ITZ analysis, creating indentations with diagonal lengths of approximately 20–25  $\mu$ m in the matrix. This resolution allows effective differentiation



Fig. 3 Image analysis methodology for the interfacial transition zone (ITZ) characterization: **a** schematic illustration of concentric 5  $\mu$ m strips for porosity analysis; **b** application of segmentation method on SEM image showing phase identification



of mechanical property gradients across the interfacial transition zone while maintaining measurement precision. Higher loads caused excessive material deformation while lower loads produced inconsistent measurements. At least 10 measurements were taken for each distance to ensure statistical reliability.

### 3 Results and discussion

The steel fiber-reinforced cementitious system comprises three distinct yet interconnected phases: steel fibers, bulk matrix, and the interfacial transition zone (ITZ). The ITZ, while physically continuous with the bulk matrix, exhibits distinct microstructural characteristics including preferential formation of C-S-H clusters, ettringite crystals, and oriented calcium hydroxide (CH) deposits. This unique composition significantly influences the composite's mechanical behavior and durability performance. The transition from fiber surface to bulk matrix occurs gradually through the ITZ, characterized by the 'wall effect' phenomenon. This effect manifests as disrupted packing of cement particles near the fiber surface, leading to altered hydration product formation and microstructural development. The resulting gradient in physical and chemical properties across the ITZ directly impacts the fiber-matrix bond strength and overall composite performance.

This section presents a systematic investigation of the ITZ characteristics under different mixing

conditions and the influence of glass powder incorporation. The analysis encompasses microstructural features, elemental distributions, porosity patterns, and mechanical properties at the interface.

#### 3.1 Effects of seawater on ITZ properties

The incorporation of seawater as mixing water significantly alters the ITZ characteristics through modified hydration processes and ionic interactions. These modifications manifest in both microstructural features and elemental distributions across the fiber–matrix interface.

#### 3.1.1 Chemical composition and ion distribution

The elemental composition and distribution across the F-M interface were analyzed using EDS line scans, with the steel fiber boundary clearly defined by the Fe content profile as shown in Fig. 4. F–M specimens mixed with freshwater exhibited substantial Ca content enrichment within approximately 40  $\mu$ m from the fiber surface, with Ca/Si ratios reaching 4–5. This elevated Ca/Si ratio significantly exceeds typical bulk paste values of 1.5–2.0, indicating the preferential formation of calcium hydroxide (CH) crystals in the interfacial zone. The Fe gradient extending from the fiber surface into the matrix suggests partial dissolution of iron, creating a distinct chemical environment in the immediate vicinity of the fiber.

The pronounced Ca enrichment stems from two primary mechanisms: the wall effect causing



Fig. 4 EDS elemental mapping and line scan profiles across the fiber-matrix interface of F-M

localized water accumulation near the fiber surface, and the preferential nucleation of CH crystals on the steel fiber surface due to their crystallographic compatibility.

When seawater was introduced as mixing water (S-M specimens), the interfacial zone exhibited markedly different elemental distributions, as shown in Fig. 5. The Ca/Si ratio decreased substantially to 1-2, approaching values typical of C-S-H gel. This significant reduction in Ca/Si ratio results from multiple concurrent mechanisms. The presence of chloride ions from seawater alters the dissolution-precipitation kinetics of cement phases, affecting CH crystal formation and orientation. Additionally, the reaction between tricalcium aluminate, CH, and chloride ions leads to the formation of Friedel's salt (3CaO·Al<sub>2</sub>O <sub>3</sub>·CaCl<sub>2</sub>·10H<sub>2</sub>O). Magnesium ions present in seawater react with CH to form magnesium hydroxide (brucite), further reducing available calcium for CH formation. The increased ionic strength in seawater mixing environments modifies the local pH gradients near the fiber surface, influencing hydration product morphology and distribution.

EDS analysis of S-M specimens also revealed characteristic changes in ionic distribution across the ITZ. Na concentrations increased from 0.27 to 0.86 atomic ratio due to seawater mixing, while Cl concentrations showed relatively minor variations across the interface. This pattern of chloride distribution can be attributed to the binding of chloride ions during Friedel's salt formation, effectively immobilizing them within the hydration products. The Fe gradient in seawater specimens showed a more gradual decline compared to freshwater specimens, indicating enhanced iron dissolution from the fiber surface in the presence of seawater. This increased iron dissolution can be attributed to the aggressive nature of the chloriderich environment, which promotes metal dissolution. The combination of increased iron dissolution and altered calcium distribution creates a unique chemical environment that significantly influences the development of the interfacial transition zone, potentially contributing to the reduced durability observed in seawater-mixed specimens.

The combined effects of these chemical modifications manifest in distinct differences in fiber-matrix bonding. S-M specimens showed visible gaps at the fiber-matrix interface, with an ITZ thickness of approximately 45 µm compared to 40 µm in F-M specimens. This increased thickness and reduced bonding quality result from the modified hydration product formation in the presence of seawater ions, altered crystal growth patterns near the fiber surface, and changes in local ionic concentrations affecting product precipitation. The presence of multiple competing ions creates a complex chemical environment that fundamentally alters the development of the fiber-matrix interface, with significant implications for mechanical properties and durability performance.



Fig. 5 EDS elemental mapping and line scan profiles across the fiber-matrix interface of S-M

**Fig. 6** Average porosity distribution across the steel fiber–matrix interface transition zone for specimens with different w/c ratios



#### 3.1.2 Physical structure and properties

Based on the image segmentation method described in Sect. 2.4.3, the porosity of the interface transition zone was quantitatively analyzed. Figure 6 compares the average porosity of the steel fiber–matrix interface transition zone between specimens with different w/c ratios cast with seawater and fresh water, where each data point represents the average porosity calculated from multiple SEM images across different sampling locations.

The microstructural development at the fiber-matrix interface exhibited distinct characteristics between freshwater and seawater specimens. Through quantitative image analysis, a clear pattern emerged in porosity distribution across the interfacial transition zone. In freshwater specimens (F-H and F-M), peak porosity occurred at approximately 20 µm from the steel fiber interface. This distinctive distribution pattern results from the wall effect and subsequent hydration mechanisms. During initial mixing, the wall effect causes water migration toward the steel fiber surface, forming a water film layer. The thickness of this layer influences the microstructure development of the interfacial transition zone through ionic concentration gradients. The first hydration product formed in this water film is typically calcium

. Silen hydroxide, which tends to orient along the fiber surface.

However, unlike conventional aggregate interfaces, the steel fiber-matrix interface exhibits a unique feature—the presence of dissolved Fe ions, as evidenced by the Fe element gradients in the EDS line scans (Figs. 4 and 5). The concentration profile near the fiber results from the superposition of dissolved ions from both the steel fiber and cement hydration, as illustrated in Fig. 7. This increased ionic concentration closest to the fiber surface promotes the formation of C–S–H clusters near the fibers, enhancing the microstructural compactness of the immediate interface region. Consequently, the most porous area develops not at the interface but approximately 20 µm from the surface.

In contrast, seawater specimens (S–H and S–M) showed maximum porosity at the steel fiber surface. This pattern was consistently observed across multiple fiber-reinforced specimens mixed with seawater, where SEM images revealed poor bonding between matrixes and fibers as shown in Fig. 8a–c. The presence of seawater ions fundamentally alters the local chemical environment during hydration. The competitive effects of various ions (Na<sup>+</sup>, Mg<sup>2+</sup>, Cl<sup>-</sup>) modify both the dissolution–precipitation kinetics and the morphology of hydration products. Furthermore, the higher dry shrinkage typically observed in seawater

Fig. 7 Schematic representation of superimposed

interface

ion concentration gradients at the steel fiber-matrix







Fig. 8 Microstructural comparison of steel fiber-matrix interfaces: **a**-**c** Seawater mixed specimens (S-M/H); **d**-**f** Seawater-glass powder modified specimens (SG-M/H)

cement mortars compared to freshwater mortars contributes to this reduced fiber-matrix bonding quality. As a result, the steel fiber-matrix interfacial transition zone in seawater specimens does not benefit from the same enhancement in compactness seen in freshwater specimens, leading to elevated porosity immediately adjacent to the fiber surface.

The distinct differences in interfacial structure between freshwater and seawater specimens have significant implications for composite performance. The increased porosity and modified hydration product distribution in seawater specimens affect not only the mechanical bond strength but also the transport properties of the interfacial zone. These modifications influence both the stress transfer characteristics between fiber and matrix and the resistance to aggressive agents such as chloride ions. The understanding of these seawater-induced modifications guides the development of potential solutions, particularly through the incorporation of supplementary materials like glass powder, which will be examined in the following section.

#### 3.2 Glass powder modification effects

The incorporation of glass powder fundamentally alters the steel fiber-matrix interface through multiple mechanisms: pozzolanic reactions, particle packing optimization, and microstructural modifications. This section examines these mechanisms and their effects on interface properties through detailed microstructural analysis, elemental mapping, and mechanical characterization.

# 3.2.1 Microstructural evolution at the fiber–matrix interface

Materials and Structures (2025) 58:143

The morphological characteristics of the steel fiber-matrix interface were systematically analyzed using scanning electron microscopybackscattered electron (SEM-BSE) imaging at multiple magnifications. Figure 8 demonstrates distinct differences between modified and unmodified specimens. Control specimens mixed with seawater (S-M/H) exhibited notable interfacial gaps between the matrix and steel fibers, indicating poor bond development. In contrast, specimens containing 25% glass powder replacement (SG-M/H) showed significantly enhanced interface continuity, with reduced void formation and improved matrix densification.

This enhancement stems from dual modification mechanisms. The physical mechanism involves fine glass powder particles (average size 22.3  $\mu$ m) filling interfacial voids and optimizing particle packing near the fiber surface. Simultaneously, pozzolanic reactions between glass powder and calcium hydroxide generate additional C–S–H gel, creating a more intricate and interconnected microstructure. The combined presence of seawater ions and glass powder creates synergistic effects, where initial hydration modifications by seawater ions are complemented by ongoing pozzolanic reactions that progressively strengthen the interface.



Fig. 9 EDS elemental mapping and line scan profiles across the fiber-matrix interface of SG-M

# 3.2.2 Chemical composition and phase distribution at the fiber–matrix interface

The EDS line scan analysis of specimens incorporating 25% glass powder revealed distinct compositional modifications across the fiber-matrix interface as shown in Fig. 9. Glass powder integration dramatically increased Si content within the interfacial zone, with the EDS line scan showing prominent Si peaks 5–15  $\mu$ m from the fiber surface. These peaks correspond to both partially reacted glass powder particles and newly formed C–S–H gel from pozzolanic reactions. The amorphous SiO<sub>2</sub> in glass powder actively reacts with calcium hydroxide produced during cement hydration, generating secondary C–S–H gel with distinctive compositional characteristics.

The Ca/Si ratio exhibited а marked transformation pattern through glass powder incorporation. While F-M specimens showed Ca/ Si ratios of 4-5 and S-M specimens demonstrated ratios of 1–2, SG-M specimens achieved significantly lower ratios of 0.8-1.5 at the critical 20–35  $\mu$ m region from the steel fiber surface. This transformation reflects the glass powder's dual modification mechanisms: consumption of calcium hydroxide through pozzolanic reactions and formation of additional C-S-H phases. The pozzolanic reactions not only modified the chemical composition but also generated additional hydration products that filled the interfacial pores, creating a more densified and refined microstructure. The alumina content from glass powder contributed to the formation of additional phases, including aluminate-containing hydration products, which further modified the interfacial zone properties.

The measured interfacial transition zone thickness, determined through Fe and Ca content variations in EDS line scans as shown in Table 4,

 Table 4
 Thickness of the interfacial transition zone (determined by EDS line scan)

Number	Starting point (µm)	Endpoint (µm)	Thickness (µm)		
F-M	85	45	40		
S-M	100	55	45		
SG-M	85	50	35		

decreased from 45  $\mu$ m in S-M specimens to 35  $\mu$ m in SG-M specimens. The refined structure created by pozzolanic reaction products directly enhances the fiber-matrix bond characteristics, as evidenced by the reduced interfacial transition zone thickness.

This modified chemical composition has significant implications for chloride binding capacity and overall durability performance. The secondary C–S–H gel formed through pozzolanic reactions exhibits different morphological and chemical characteristics compared to primary C–S–H from cement hydration, creating a more intricate and interconnected microstructure that better resists chloride penetration and enhances long-term durability.

## 3.2.3 Porosity distribution analysis

Based on the image segmentation methodology described in Sect. 2.4.3, porosity distributions were quantitatively analyzed for all seawater-mixed specimens. Figure 10 presents the comparative porosity profiles, where each data point represents the average porosity calculated from multiple SEM images across different sampling locations.

Glass powder-modified specimens demonstrated superior pore structure refinement, with surface porosity reducing from over 95% in S-M specimens to approximately 85% at the immediate fiber-matrix interface (0–5  $\mu$ m) in SG-M specimens. Most notably, SG-M specimens exhibited a sharp porosity decrease in the 10–20  $\mu$ m region from the fiber surface. The interfacial transition zone thickness decreased from 45  $\mu$ m to approximately 35  $\mu$ m, accompanied by decreased porosity throughout this region.

The water-to-cement ratio significantly influenced overall porosity levels, with higher w/c ratios resulting in increased porosity values. However, glass powder incorporation showed more pronounced improvement effects in specimens with higher w/c ratios (S-M & SG-M) compared to those with lower w/c ratios. In specimens with lower watercement ratios, the inherently denser matrix structure limited the observable benefits of glass powder addition, suggesting that glass powder modification is particularly effective in refining pore structure in more porous systems. Fig. 10 Porosity distribution profiles across the steel fiber-matrix interface for seawater-mixed specimens with varying w/c ratios and glass powder content



#### Fig. 11 Microhardness distribution profiles across the steel fiber-matrix interface for seawater-mixed specimens with varying w/c ratios and glass powder content

# 3.2.4 Mechanical properties: interface microhardness

Microhardness profiles across the fiber-matrix interface provided comprehensive quantitative evidence of glass powder's strengthening effects. Using Vickers microhardness measurements at 10  $\mu$ m intervals from the fiber surface to the bulk matrix, distinct patterns emerged between modified and unmodified specimens. As shown in Fig. 11, glass powder-modified specimens (SG-M; SG-H) exhibited enhanced microhardness values throughout the interfacial transition zone. The weak zone thickness, characterized by reduced microhardness values, decreased



Fig. 12 Free chloride ion concentration profiles: a initial  $\blacktriangleright$  distribution before exposure; b distribution after 5 wet–dry cycles; c distribution after 15 wet–dry cycles

substantially from approximately 50  $\mu m$  in control specimens to 30–40  $\mu m$  in glass powder-modified specimens.

In the interfacial region (0-50 µm from fiber powder-modified surface). glass specimens showed an average microhardness increase of 183.3 MPa compared to unmodified specimens. At the immediate vicinity of the fiber surface (0-10 µm), local microhardness values were higher by 147.5 MPa in average. Multiple measurements at each distance point indicated that glass powdermodified specimens exhibited more consistent properties compared mechanical to control specimens.

Specimens prepared without glass powder (S-M; S–H) demonstrated consistently lower microhardness values extending to 50  $\mu$ m from the fiber surface, indicating a broader zone of mechanical weakness. The transition from interfacial zone to bulk matrix properties was more gradual in these specimens, with higher measurement fluctuations throughout the measured profile.

The water-to-cement ratio influenced the magnitude of microhardness improvements, with specimens having lower w/c ratios (SG-H) showing higher absolute values but relatively smaller percentage increases compared to their unmodified counterparts. This trend aligns with the porosity measurements and suggests that glass powder modification is particularly effective in enhancing interface properties in systems with higher initial porosity.

3.3 Chloride ion distribution and corrosion development

The chloride ion transport behavior in seawatermixed mortars revealed distinct patterns influenced by glass powder modification. Figure 12 demonstrates the characteristic chloride concentration gradients, showing decreasing concentrations from specimen surfaces toward interior regions. Following the AFREM procedure detailed in Sect. 2.4.4, free chloride content was determined at 10 mm depth intervals from the exposed surface to 40 mm depth. Glass



powder-modified specimens consistently exhibited lower free chloride ion concentrations compared to their unmodified ones; a behavior that can be directly attributed to the microstructural refinements documented in Sect. 3.2.

The enhanced chloride resistance of glass powder-modified specimens stems from multiple complementary mechanisms operating simultaneously at different scales. The physical modifications manifested in significant microstructural refinements, with interface porosity reducing from 95 to 85% in SG-M specimens and interfacial transition zone thickness decreasing from 45 to 35  $\mu$ m. These structural improvements create substantially more tortuous pathways for chloride ion transport through the matrix. The refined pore network significantly affects both the primary transport mechanisms: the diffusion process during continuous exposure and capillary absorption during wetting cycles.

The chemical modifications introduced through glass powder incorporation demonstrate equally important contributions to chloride resistance. When glass powder particles undergo pozzolanic reactions, they consume calcium hydroxide, generating additional calcium silicate hydrate phases with modified compositions. EDS line scan analyses shown in Figs. 4, 5 and 9 revealed a transformation in Ca/Si ratios from 4-5 in control specimens to 0.8-1.5 in glass powdermodified specimens within the critical 20-35 µm region from the steel fiber surface. This microstructural transformation enhances chloride resistance through several distinct mechanisms. The additional C-S-H gel generated through pozzolanic reactions fills existing capillary pores, significantly reducing their connectivity and limiting transport paths. Furthermore, the alumina content provided by the glass powder promotes the formation of Friedel's salt, a phase known for its chloride-binding capabilities. The resulting modified phase assemblage creates a more complex and tortuous microstructure that impedes chloride ion movement.

Analysis of free chloride ion concentrations provided quantitative evidence of these improvements. Seawater-mixed specimens initially showed baseline chloride levels of 0.1-0.2% by mass of binder, reflecting the mixing water composition. Glass powder modification demonstrated remarkable effectiveness in reducing these concentrations by 15–25%. This reduction aligns with studies demonstrating that pozzolanic materials can enhance chloride binding through both physical and chemical mechanisms [38, 41, 42]. Notably, this improvement maintained its effectiveness throughout the extended testing period, with glass pow-

der-modified specimens consistently showing lower free chloride concentrations across all measured depths. This sustained performance indicates the long-term stability of the modifications introduced by glass powder incorporation, suggesting durable enhancement of chloride resistance in these systems.

The chloride concentration profiles after 15 wet–dry cycles (Fig. 12c) revealed the longer-term effectiveness of glass powder modification. While surface concentrations continued to increase in all specimens, glass powder-modified specimens maintained consistently lower chloride concentrations throughout their depth profiles. The difference in surface chloride concentrations between modified and unmodified specimens reached approximately 25–30%, highlighting the sustained effectiveness of glass powder modification in restricting chloride accumulation. This enhanced resistance can be attributed to the continued pozzolanic reactions of glass powder, which progressively refine the pore structure and modify the chemical environment affecting chloride transport.

The combined presence of seawater ions and glass powder creates synergistic effects in the interfacial zone. While seawater ions modify the local chemical environment and affect initial hydration product formation, glass powder's pozzolanic reactions continue to refine and strengthen the interface over time. This ongoing microstructural modification process results in a more chemically stable and physically robust fiber-matrix interface, directly contributing to enhanced chloride resistance of the composite system.

The progression of chloride-induced corrosion was systematically monitored throughout the wet–dry cycles, with results summarized in Table 5. Visual inspection was conducted after each cycle to identify the first appearance of rust spots on specimen surfaces.

**Table 5**Time for corrosioninitiation of specimens

Specimen	F-M	F–H	FG-M	FG-H	S-M	S–H	SG-M	SG-H
Cycles	13–16	15–16	15–16	15–16	5–6	5–6	8–10	8–12



When rust spots were detected, specimens were split to confirm internal corrosion initiation through microscopic examination. The cycle number at which corrosion was first confirmed was recorded as the corrosion initiation point. Glass powder-modified specimens demonstrated significantly delayed corrosion initiation compared to unmodified specimens. For freshwater specimens, glass powder incorporation delayed corrosion initiation by 0–3 cycles, while seawater-mixed specimens showed a delay of 3–6 cycles. This enhancement directly correlates with the reduced chloride penetration rates and modified binding mechanisms discussed above.

The incorporation of glass powder in seawatermixed mortars raises potential concerns regarding alkali-silica reaction (ASR), particularly given the elevated alkali content from seawater ions. However, our microstructural analysis revealed no evidence of ASR gel formation after 15 wet-dry cycles. This resistance can be attributed to the fine particle size of the glass powder (average 22.3 µm), which promotes pozzolanic reactions that consume available alkalis before deleterious expansion can occur. Additionally, the modified calcium-silicate-hydrate gel formed through pozzolanic reactions demonstrates enhanced alkali binding capacity, further mitigating ASR risk. These findings align with previous research demonstrating that glass particles below 25 µm significantly reduce expansion potential compared to coarser fractions.

# 4 Conclusion

This research investigated how glass powder modification enhances steel fiber-reinforced mortars in marine environments, revealing several significant findings:

1. The novel image segmentation methodology developed in this study provides quantitative insights into interfacial transition zone characteristics. By combining preprocessing techniques, K-means clustering, and analysis of concentric 5  $\mu$ m strips from the fiber surface, we achieved precise characterization of porosity gradients and phase distributions around steel fibers.

- 2. Glass powder incorporation transformed the fiber–matrix interface, reducing transition zone thickness from 45 to 35  $\mu$ m and decreasing surface porosity from 95 to 85%. This refinement, resulting from both physical filling and pozzolanic reactions, increased interfacial microhardness by 183.3 MPa compared to unmodified specimens.
- Chemical analysis revealed modified C–S–H composition with reduced Ca/Si ratios (from 4–5 to 0.8–1.5) near the fiber surface. This modification enhanced chloride resistance, with glass powder specimens showing 15–25% lower chloride concentrations and delayed corrosion initiation by 3–6 cycles in seawater-mixed specimens.
- 4. Based on these findings, 25% glass powder replacement appears optimal for enhancing chloride resistance in steel fiber-reinforced mortars for marine applications, particularly in non-structural elements or protective layers.

Several limitations should be acknowledged: testing was conducted on laboratory-scale specimens rather than full-scale elements; exposure duration may not fully represent long-term performance in actual marine environments; and corrosion assessment techniques could be expanded in future studies.

Future research directions should include longterm performance evaluation, optimization of glass powder properties for specific applications, investigation of potential synergistic effects with other supplementary materials, and field validation in actual marine structures to confirm laboratory findings.

Acknowledgements This research was supported by the National Natural Science Foundation of China (52308275) and the Hong Kong Green Technology Fund (K-ZB5B). The authors would like to express their gratitude to Dr. Li Zhanzhao for his valuable suggestions during the manuscript preparation.

Author contribution XZ: Conceptualization, Methodology, Investigation, Data analysis, Writing—original draft. J-XL: Methodology, Writing—review and editing. CSP: Conceptualization, Supervision, Funding acquisition, Writing—review and editing.

**Funding** Open access funding provided by The Hong Kong Polytechnic University.



**Data availability** The authors declare that the data supporting the findings of this study are available within the paper. Should any raw data files be needed in another format they are available from the corresponding author upon reasonable request. Source data are provided with this paper.

#### Declarations

**Conflict of interest** The authors declare that they have no conflict of interest.

**Open Access** This article is licensed under a Creative Commons Attribution 4.0 International License, which permits use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons licence, and indicate if changes were made. The images or other third party material in this article are included in the article's Creative Commons licence, unless indicated otherwise in a credit line to the material. If material is not included in the article's Creative Commons licence and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission directly from the copyright holder. To view a copy of this licence, visit http://creativecommons.org/licenses/by/4.0/.

#### References

- Siddiqui AR, Khan R, Akhtar MN (2025) Sustainable concrete solutions for green infrastructure development: a review. JSCMT 10:108–141
- Jiang Y, Ling T-C, Mo KH, Shi C (2019) A critical review of waste glass powder—multiple roles of utilization in cement-based materials and construction products. J Environ Manag 242:440–449
- Lebullenger R, Mear FO (2019) Glass recycling. In: Musgraves JD, Hu J, Calvez L (eds) Springer handbook of glass. Springer, Cham, pp 1355–1377. https://doi.org/10. 1007/978-3-319-93728-1\_39
- Kua HW, Teoh WS, Xu X, Huang B, Geng Y (2024) A review of glass recycling policies in Stockholm, Hong Kong SAR and Shanghai from a circular economy perspective. J Clean Prod 434:140068
- Lu J-X, Poon CS (2019) 6. Recycling of waste glass in construction materials. In: de Brito J, Agrela F (eds) New trends in eco-efficient and recycled concrete. Woodhead Publishing, Sawston, pp 153–167. https://doi.org/10.1016/ B978-0-08-102480-5.00006-3
- ChandraPaul S, Šavija B, Babafemi AJ (2018) A comprehensive review on mechanical and durability properties of cement-based materials containing waste recycled glass. J Clean Prod 198:891–906
- Khan MdNN, Saha AK, Sarker PK (2020) Reuse of waste glass as a supplementary binder and aggregate for sustainable cement-based construction materials: a review. J Build Eng 28:101052
- 8. Patel D, Tiwari RP, Shrivastava R, Yadav RK (2019) Effective utilization of waste glass powder as the

substitution of cement in making paste and mortar. Constr Build Mater 199:406–415

- Aliabdo AA, Abd Elmoaty AEM, Aboshama AY (2016) Utilization of waste glass powder in the production of cement and concrete. Constr Build Mater 124:866–877
- Mejias JA, Berry AJ, Refson K, Fraser DG (1999) The kinetics and mechanism of MgO dissolution. Chem Phys Lett 314:558–563
- Siad H, Lachemi M, Sahmaran M, Hossain KMA (2016) Effect of glass powder on sulfuric acid resistance of cementitious materials. Constr Build Mater 113:163–173
- 12. Lu J-X et al (2021) Early-age and microstructural properties of glass powder blended cement paste: improvement by seawater. Cem Concr Compos 122:104165
- Jin W, Meyer C, Baxter S (2000) "Glascrete"-concrete with glass aggregate. ACI Mater J 97(2):208–213
- Mirzahosseini M, Riding KA (2015) Influence of different particle sizes on reactivity of finely ground glass as supplementary cementitious material (SCM). Cem Concr Compos 56:95–105
- Lu J, Duan Z, Poon CS (2017) Combined use of waste glass powder and cullet in architectural mortar. Cem Concr Compos 82:34–44
- Du H, Tan KH (2017) Properties of high volume glass powder concrete. Cem Concr Compos 75:22–29
- Lu J, Duan Z, Poon CS (2017) Fresh properties of cement pastes or mortars incorporating waste glass powder and cullet. Constr Build Mater 131:793–799
- Nahi S, Leklou N, Khelidj A, Oudjit MN, Zenati A (2020) Properties of cement pastes and mortars containing recycled green glass powder. Constr Build Mater 262:120875
- Li P, Li W, Yu T, Qu F, Tam VWY (2020) Investigation on early-age hydration, mechanical properties and microstructure of seawater sea sand cement mortar. Constr Build Mater 249:118776
- Zhao Y, Hu X, Shi C, Zhang Z, Zhu D (2021) A review on seawater sea-sand concrete: mixture proportion, hydration, microstructure and properties. Constr Build Mater 295:123602
- Li P, Li W, Wang K, Zhao H, Shah SP (2023) Hydration and microstructure of cement paste mixed with seawater: AN advanced investigation by SEM-EDS method. Constr Build Mater 392:131925
- Kaushik SK, Islam S (1995) Suitability of sea water for mixing structural concrete exposed to a marine environment. Cem Concr Compos 17:177–185
- Kang S-T, Lee Y, Park Y-D, Kim J-K (2010) Tensile fracture properties of an ultra high performance fiber reinforced concrete (UHPFRC) with steel fiber. Compos Struct 92:61–71
- Martinola G, Meda A, Plizzari GA, Rinaldi Z (2010) Strengthening and repair of RC beams with fiber reinforced concrete. Cem Concr Compos 32:731–739
- Aruntaş HY, Cemalgil S, Şimşek O, Durmuş G, Erdal M (2008) Effects of super plasticizer and curing conditions on properties of concrete with and without fiber. Mater Lett 62:3441–3443
- Kim SB, Yi NH, Kim HY, Kim J-HJ, Song Y-C (2010) Material and structural performance evaluation of recycled PET fiber reinforced concrete. Cem Concr Compos 32:232–240



- 27. Bernal S, De Gutierrez R, Delvasto S, Rodriguez E (2010) Performance of an alkali-activated slag concrete reinforced with steel fibers. Constr Build Mater 24:208–214
- Nematzadeh M, Shahmansouri AA, Fakoor M (2020) Post-fire compressive strength of recycled PET aggregate concrete reinforced with steel fibers: optimization and prediction via RSM and GEP. Constr Build Mater 252:119057
- Grabois TM, Cordeiro GC, ToledoFilho RD (2016) Fresh and hardened-state properties of self-compacting lightweight concrete reinforced with steel fibers. Constr Build Mater 104:284–292
- Khaloo, A., Molaei Raisi, E., Hosseini, P. & Tahsiri, H. Mechanical performance of self-compacting concrete reinforced with steel fibers. *Construction and Building Materials* 51, 179–186 (2014).
- Marcos-Meson V, Fischer G, Edvardsen C, Skovhus TL, Michel A (2019) Durability of steel fibre reinforced concrete (sfrc) exposed to acid attack—a literature review. Constr Build Mater 200:490–501
- Bakhshi, M., & Nasri, V. (2014). Developments in design for fiber reinforced concrete tunnel segments. FRC 2014, 441.
- Singh H (2017) Steel fiber reinforced concrete: behavior, modelling and design. Springer, Singapore. https://doi. org/10.1007/978-981-10-2507-5
- Mason TO, Campo MA, Hixson AD, Woo LY (2002) Impedance spectroscopy of fiber-reinforced cement composites. Cem Concr Compos 24:457–465
- Woo LY, Wansom S, Ozyurt N, Mu B, Shah SP, Mason TO (2005) Characterizing fiber dispersion in cement composites using AC-Impedance Spectroscopy. Cement Concrete Comp 27(6):627–636

- Blunt J, Jen G, Ostertag CP (2015) Enhancing corrosion resistance of reinforced concrete structures with hybrid fiber reinforced concrete. Corros Sci 92:182–191
- 37. Wu R et al (2023) Degradation of fiber/matrix interface under various environmental and loading conditions: insights from molecular simulations. Constr Build Mater 390:131101
- Wang Y et al (2019) Understanding the chloride binding and diffusion behaviors of marine concrete based on Portland limestone cement-alumina enriched pozzolans. Constr Build Mater 198:207–217
- Scrivener KL, Crumbie AK, Laugesen P (2004) The interfacial transition zone (ITZ) between cement paste and aggregate in concrete. Interface Sci 12:411–421
- 40. Chaussadent T, Arliguie G (1999) AFREM test procedures concerning chlorides in concrete: extraction and titration methods. Mat Struct 32:230–234
- Garcia R et al (2021) Chloride binding capacity of metakaolin and nanosilica supplementary pozzolanic cementitious materials in aqueous phase. Constr Build Mater 298:123903
- 42. He H, Qiao H, Sun T, Yang H, He C (2024) Research progress in mechanisms, influence factors and improvement routes of chloride binding for cement composites. J Build Eng 86:108978

**Publisher's Note** Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.