

Investigating the effectiveness of integral crystalline waterproofing and microstructural analysis: A case study of national convention building basement

Warun Na Songkhla¹⁾, Kwanchanok Oonta-on^{*2)} and Gritsada Sua-iam²⁾

¹⁾Department of Civil Engineering, Faculty of Engineering and Architecture, Rajamangala University of Technology Suvarnabhumi, Nonthaburi, Thailand

²⁾Department of Civil Engineering, Faculty of Engineering, Rajamangala University of Technology Phra Nakhon, Bangkok, Thailand

Received 3 November 2023

Revised 14 March 2024

Accepted 22 March 2024

Abstract

Water permeability poses a significant challenge in underground concrete structures. This study investigates the effectiveness of integral crystalline waterproofing (ICW) in improving water impermeability and its impact on microstructural properties. The research focuses on a case study of a national convention building basement with a thickness of 100 cm. Concrete cylinders were drilled and divided into ICW-coated samples and control samples subjected to various curing conditions. Water absorption, mechanical properties, and microstructure characteristics were analyzed. The results indicate that ICW reduces water absorption compared to non-coated specimens. The compressive strength and ultrasonic pulse velocity (UPV) tests demonstrate that the specimens coated with ICW exhibit higher values, suggesting enhanced concrete quality and decreased porosity. SEM-EDS analysis detected titanium compounds, introduced by ICW, which successfully penetrated the concrete matrix. Interestingly, titanium, not typically found in cement compositions, was present due to the ICW material. Titanium compounds were detected at shallower depths under dry curing conditions, suggesting localized penetration. However, with water curing, titanium compounds were found throughout the depth profiles. These findings highlight the importance of proper application and curing conditions for optimal ICW performance.

Keywords: Impermeability, Mechanical properties, Integral crystalline waterproofing, Underground construction, Microstructure characteristics

1. Introduction

Concrete is widely recognized as a durable and resilient construction material, but its long-term service life can be compromised by degradation processes, resulting in significant negative impacts on society, the environment, and the economy. The deterioration of concrete is closely linked to various climate variables present in the surrounding environment, including humidity, temperature, rainfall, storms, waves, as well as tidal. These factors contribute to the degradation of concrete structures and require substantial financial resources for repair and maintenance, imposing significant costs on the economy [1-5]. To address this issue, extensive research has been conducted in the past two decades to produce superior and long-lasting concrete structures. Numerous testing techniques and methods have been devised to evaluate the remaining lifespan of current structures and assess the resistance to corrosion of concrete mixtures, elements, and newly built structures [1-2]. These assessment methods enable informed decision-making regarding the repair, maintenance, and future durability of concrete structures. Considering the interaction between the environment and concrete elements is essential for ensuring the long-term performance and durability of concrete structures. The water-resistant properties and porosity of concrete can be altered by modifying the composition of the concrete mixture, despite its natural resistance to water. Concrete is composed of an intricate arrangement of interconnected pores and capillaries, which can function as conduits for the entry of potentially deleterious substances, ultimately resulting in degradation. The water ingress permeability of concrete presents notable difficulties, such as the potential corrosion of reinforcing steel, structural deterioration, and a diminished lifespan of concrete structures. Therefore, it is of utmost importance to carefully consider the interaction between the environment and concrete elements in order to ensure long-term performance and durability [6-9].

One significant challenge in concrete structures is water permeability, especially in underground constructions where water ingress risk is high. The ability to enhance the water impermeability of concrete is crucial for ensuring the durability and longevity of such structures [10]. Water infiltration into concrete can lead to a range of issues, including reinforcement deterioration, steel corrosion, concrete strength degradation, and the growth of mold and bacteria [11-13]. Effective strategies to mitigate water permeability are of utmost importance in the construction industry. Various techniques and materials have been developed to address the issue of water permeability in concrete, such as the use of additives [14], surface coatings [15], and membranes [6]. However, these approaches often have limitations in terms of durability, susceptibility to damage during construction, and limited effectiveness in thick concrete sections. A potentially effective strategy for improving the water impermeability of concrete involves the application of crystalline waterproofing

*Corresponding author.

Email address: kwanchanok.o@rmutp.ac.th

doi: 10.14456/easr.2024.34

cementitious materials. These materials offer a unique solution for mitigating water permeability by creating crystalline structures within the concrete matrix [16, 17]. Upon application to the concrete surface, these materials penetrate the pores and capillary network, initiating a reaction with the existing minerals in the concrete, thereby forming insoluble crystals. These crystals effectively obstruct the pathways through which water can penetrate, resulting in reduced water absorption and improved overall water impermeability of the concrete [7, 18-21].

Crystalline waterproofing cementitious materials have demonstrated significant potential in enhancing the water resistance of concrete structures. By establishing a network of crystals, these materials possess a self-healing mechanism that can seal microcracks and small voids in the concrete over time. This self-healing property contributes to the durability of the concrete, reducing the need for frequent maintenance and repair [3, 9, 16, 22-24], as well as in seawater environment [25]. Consequently, these enhancements can substantially decrease the long-term costs associated with maintenance and repair of concrete structures [26]. Nevertheless, it is crucial to acknowledge that numerous essential waterproofing materials rely on hydrophilic pore-blocking compounds composed of silicate-based minerals that exclusively engage with water in its liquid state. On the contrary, a waterproofing system that undergoes crystallization and has the ability to interact with both water vapor and liquid can yield notably enhanced performance [13]. Recent publications have focused on the investigation of crystalline waterproofing admixtures and their effectiveness in enhancing the waterproofing properties, thermal effects during cement hydration and Mitigation of alkali silica reactions of concrete [27-30]. In their experimental program, Reiterman and Pazderka [31] substantiated the efficacy of crystalline coatings in mitigating water ingress, particularly in the context of construction joints. Prior research has also investigated the influence of water-binder ratio and the dosage of crystalline waterproofing admixture on the efficacy of these admixtures in concrete compositions [21]. The primary objective of the experimental phase of this study was to evaluate the impact of crystalline waterproofing admixtures on water penetration under pressure and the subsequent reduction of crack widths in concrete. Furthermore, the study sought to elucidate the underlying mechanism of the effects of crystalline waterproofing admixtures [21]. In the realm of topical treatment for concrete, a dual-crystallization waterproofing technology has been developed. The technology in question employs a dual-component reactive solution that is applied onto fully cured or aged concrete, thereby initiating a range of chemical reactions [32].

Furthermore, recent studies by Hu et al. [9] and Wang et al. [33] have demonstrated that the application of cementitious capillary crystalline waterproofing materials can effectively improve the sulfate resistance of cement-based materials. The utilization of crystalline waterproofing can be implemented in two ways: as a coating or as an admixture, with the purpose of diminishing capillary porosity in concrete [18]. In addition, crystalline waterproofing admixture can be used together with superabsorbent polymer for crack healing of cement-based material [34]. Nevertheless, there exists a dearth of scholarly investigations pertaining to the efficacy of crystalline waterproofing additives from the standpoint of the industrial sector. A literature review indicated that while there is some research on the efficacy of crystalline waterproofing additives in enhancing the waterproofing properties of basement concrete, more application-oriented research is needed to comprehensively understand their effectiveness in basement concrete [35]. The primary objective of this study is to examine the efficacy of integral crystalline cementitious waterproofing materials in augmenting the water impermeability of concrete. The study focuses on a challenging environment, specifically the underground floor of a large-scale national convention center. This floor is characterized by a substantial thickness of 100 centimeters, which poses significant challenges for achieving effective water resistance. The study involves the extraction of test samples from the concrete floor and subjecting them to various conditions and analyses. By examining the mechanisms underlying water absorption, as well as the resulting mechanical and microstructural changes induced by these materials, this research aims to provide valuable insights for construction projects that require exceptional water impermeability, especially in thick concrete sections. The findings from this study have the potential to advance concrete technology and contribute to the development of more efficient and sustainable construction practices. In conclusion, these observations will contribute to the overall sustainability and efficacy of concrete constructions in challenging conditions.

2. Materials and methods

2.1 Materials

The primary material employed in this study is Integral Crystalline Waterproofing (ICW), which is a specialized type of cementitious waterproofing material designed to enhance the water impermeability of concrete structures. The ICW is a powder-based substance that is combined with water to create a slurry, which is subsequently administered onto the concrete surface. The ICW utilized in this study was 1.60 kg/m². The chemical composition of ICW was examined through the utilization of X-Ray Fluorescence (XRF) and is displayed in Table 1. The visual representation of ICW can be observed in Figure 1.



Figure 1 The visual representation of ICW

Table 1 Physicochemical characteristics of ICW

Characterization	ICW
Chemical compound (%)	
Calcium oxide (CaO)	52
Silicon dioxide (SiO ₂)	20
Ferric oxide (Fe ₂ O ₃)	1.6
Aluminium oxide (Al ₂ O ₃)	1.3
Sulphur trioxide (SO ₃)	1.5
Magnesium oxide (MgO)	1.0
Potassium oxide (K ₂ O)	0.3
Sodium oxide (Na ₂ O)	1.3
Titanium dioxide (TiO ₂)	2.17
Other	15.73
Carbon	3.1
Physical Characteristics	
Color	Grey
Physical state	Solid powder
Density (kg/m ³)	1,800
Blaine surface area (cm ² /g)	3,800

2.2 Experiment program

2.2.1 Project studied information

The study was conducted in the reinforced concrete basement structure of a 30,000-square-meter national convention building, addressing concerns about water ingress. The 1-meter-thick basement floor provided a substantial section for evaluating ICW effectiveness. Sampling focused on the underground parking area, spanning 6,000 square meters and located 10–15 meters below the ground surface. Sampling points were strategically chosen for representation across the entire thickness. During the study, while the construction site was ongoing, precautions were taken to prevent groundwater interference. The research serves as an effect model for future conditions, considering potential impacts from groundwater levels at the surface area of the 1-meter-thick concrete basement structure. Notably, in Bangkok, the study's location is situated on the tidal zone and tidal flat of marine clays. The water in the topmost aquifer (within the 50-meter zone) is not potable due to its high salinity since its deposition [36].

2.2.2 Application of ICW

The ICW was applied to the M35 class of concrete basement floor 24 hours after pouring, at a rate of 1.6 kg/m². The application process involved thoroughly mixing the ICW powder with water to achieve a uniform slurry, which was then evenly distributed on the basement floor surface. Precision was employed during the application to ensure consistent coverage and adherence to the manufacturer's guidelines. Addressing the concern regarding the perceived difficulty in understanding how the surface-applied ICW agent could impact the concrete structure at a depth of 100 cm, it is acknowledged that direct penetration to such depths is unlikely. However, the study considers a scenario where the concrete surface interacts with groundwater. In such cases, the upward movement of groundwater facilitates the downward transport of the ICW, reaching lower layers of the concrete. This phenomenon results in denser and more waterproof concrete near the surface, consequently reducing susceptibility to environmental degradation, such as corrosion from groundwater ingress.

2.2.3 Coring of the basement

After applying the ICW to the basement floor, the concrete underwent a 28-day curing process. To evaluate the effectiveness of the ICW treatment, samples were collected from the treated basement floor for analysis. Specialized coring equipment was utilized to extract cylindrical specimens with a diameter of 10 cm and a depth of 100 cm, as illustrated in Figure 2. The cylindrical core samples were divided into two groups: a control group that did not undergo any ICW treatment, and an experimental group that received the ICW application. The purpose of conducting the 1-meter coring test for non-coating specimens was to establish a baseline reference for comparison with ICW-coated specimens at different depths within the same 1-meter thickness. It was assumed that the properties such as water absorption and compressive strength of the non-coating (control) specimens would be relatively consistent throughout the 1-meter thickness, given the homogeneous nature of the concrete floor.

2.2.4 Curing conditions

(a) 48-hour air curing: A subset of the core samples was subjected to a 48-hour air curing process. The specimens were subjected to the surrounding atmosphere at an average temperature and were carefully kept at a controlled level of moisture, not exceeding 15%, without the utilization of any supplementary curing techniques as depicted in Figure 3. This allowed for evaluating the performance of ICW under typical air exposure conditions.

(b) Water curing with intermittent water release: Another subset of the core samples underwent water curing. These samples were fully submerged in a water tank to ensure continuous contact with water. To simulate realistic conditions, water was periodically released from the tank every 48 hours. The water level was gradually reduced by approximately 10 cm with each release until the tank was completely emptied. This water curing method represented a more severe and dynamic condition to test the effectiveness of waterproofing as depicted in Figure 4.



Figure 2 Cylindrical specimens with a 100 cm depth and a 10 cm diameter



Figure 3 48-Hour air curing

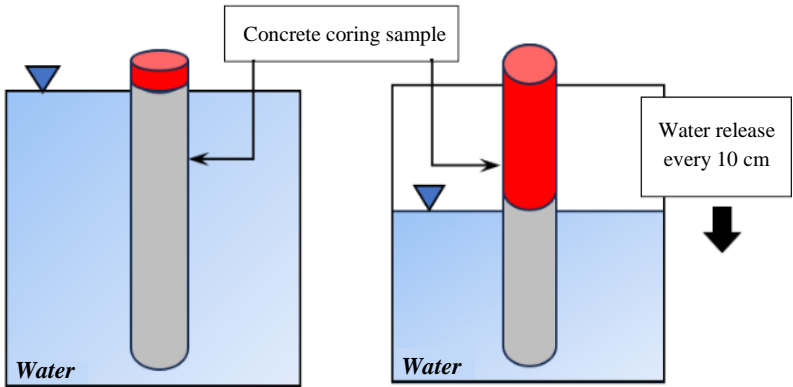


Figure 4 Water curing with intermittent water release

2.2.5 Leaching consideration

The importance of preventing leaching of ICW materials, which could lead to inaccurate assessment of its performance. The selected water curing method involves a controlled intermittent release of water, preventing excessive leaching while maintaining a moist environment for proper curing. This approach was adopted to ensure that the ICW material's properties are adequately retained, and its penetration and crystallization within the concrete matrix are not compromised.

2.2.6 Water absorption testing

After the specified curing processes, the core samples were precisely sawed into smaller sections at 20 cm intervals. These sections were then subjected to water absorption testing to quantify the impact of ICW on water absorption. The water absorption testing involved immersing the sawed sections in water and monitoring the weight increase over a defined period. This allowed for the determination of the water absorption rate and a comparison between the treated and control samples. In order to assess the efficacy of ICW in mitigating water absorption in cured concrete, experiments were carried out in accordance with the guidelines outlined in ASTM 642 [37]. A total of thirty concrete coring specimens, in the form of cylinders measuring 10 x 20 cm, were utilized for testing purposes, as depicted in Figure 5.



Figure 5 Laboratory prepared of concrete coring cylinder specimens for water absorption testing

The determination of the water absorption percentage was conducted by utilizing the concrete mass values obtained through the application of the procedures outlined in ASTM C642 [37]. This estimation was accomplished by employing the subsequent Eq. (1)

$$\text{Water Absorption (\%)} = (W2 - W1) / W1 \quad (1)$$

The variable W1 represents the weight of the specimen when it is completely dry in an oven, while the variable W2 represents the weight of the specimen when it is saturated and at the point of being surface dry.

2.2.7 Compressive strength testing

The evaluation of the mechanical properties of concrete was conducted by performing compressive strength testing in accordance with ASTM C39 [38] with the aim of assessing the influence of ICW. This test is a standard procedure used to assess the concrete's ability to withstand compressive forces. Cylindrical specimens with a diameter of 10 cm and a height of 20 cm were fabricated from core samples obtained from the treated basement floor. These specimens were carefully cured under specified conditions, involving maintaining them in a controlled environment with adequate moisture and temperature for a designated period. Following the designated curing period, the specimens underwent compressive loading utilizing a hydraulic testing machine. The applied load was gradually increased until the concrete failed, and the maximum compressive strength was recorded.

2.2.8 Ultrasonic pulse velocity testing

The utilization of UPV testing is a prevalent non-destructive method employed for the assessment of concrete characteristics, providing valuable insights into the quality, homogeneity, and integrity of concrete structures. In this study, UPV testing was employed

to assess the soundness and homogeneity of the concrete specimens. The velocity of ultrasonic waves is influenced by the density, elasticity, and uniformity of the concrete. For this case study, concrete core samples obtained from the convention building basement were carefully prepared for UPV testing. The concrete core samples, sized 10 x 20 cm, were used for the UPV test. The mean UPV values were derived from three cylindrical specimens extracted from each coring sample utilizing a PUNDIT device. The ultrasonic transducers were securely attached to the surface of the concrete specimens at designated locations. Two measurements were taken at the middle of each cylinder, perpendicular to the casting direction. The experiment was carried out following the guidelines outlined in ASTM C 597 [39], using a direct transmission method.

2.2.9 Microstructural analysis

The microstructure analysis of the core samples collected from the treated basement floor was conducted using Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Spectroscopy (EDS). These techniques provide valuable insights into the composition and morphology of the concrete at a microscopic level. To facilitate the analysis, the core samples were accurately sawed into smaller sections at 20 cm intervals, as shown in Figure 6. This allowed for a systematic evaluation of the microstructural characteristics across the treated concrete. SEM was employed to observe and analyze microstructural features, including crystal formation, pore structure, and any notable alterations in the concrete matrix. In addition, EDS analysis was performed on selected areas of interest within the microstructure to provide elemental composition information by detecting the characteristic X-ray emissions generated when the specimen is bombarded with electrons.

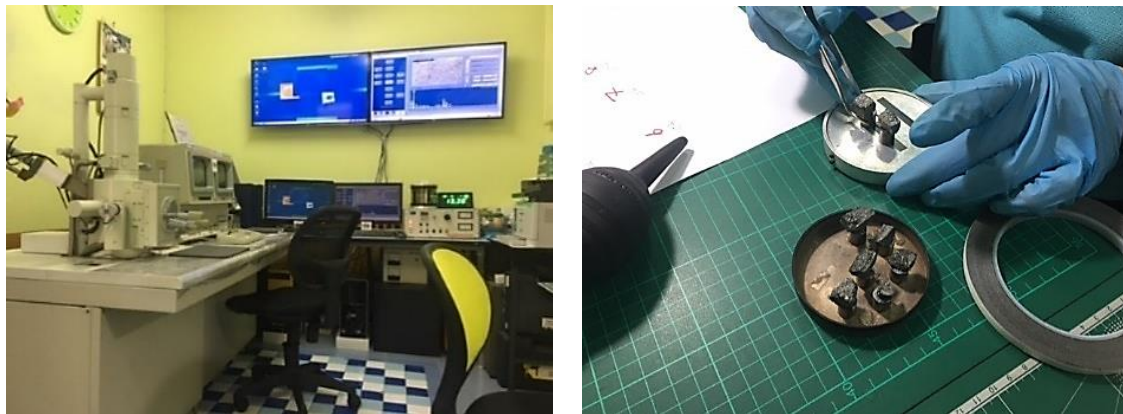


Figure 6 Sample preparation and microstructural analysis.

3. Results and discussion

3.1 Water absorption

Table 2 displays the water absorption outcomes acquired from various curing conditions. The averages were calculated based on testing three-cylinder specimens at an age of 28 days. The findings of this study offer valuable insights into the efficacy of ICW treatment in mitigating water absorption and enhancing the water impermeability of the concrete specimens. For the non-coated specimens, the 48-hour air curing condition resulted in a water absorption of 5.23%, which serves as the baseline or control value for comparison with the surface coated specimens. In the case of surface coated specimens, the water absorption values for the cored specimens at different depths demonstrated a significant reduction compared to the non-coated specimens. The water absorption percentage increased as the coring depth of the specimens increased. The water absorption rate values observed for the surface coated specimens varied from 4.32% to 4.78%. The water absorption at coring depths of 20, 40, 60, 80, and 100 cm exhibited enhancements of 17.4%, 15.9%, 13.4%, 11.3%, and 8.60%, correspondingly, in comparison to the specimens without coating.

Table 2 Water absorption of core specimen with different curing condition

Type of specimen	Curing condition	Water absorption (%)	Compared with control (%)
Non coating	48-hour air curing	5.23	100
Coating			
- 20 cm depth		4.32	82.6
- 40 cm depth		4.40	84.1
- 60 cm depth		4.53	86.6
- 80 cm depth		4.64	88.7
- 100 cm depth		4.78	91.4
Non coating	Water curing with intermittent water release	5.44	100
Coating			
- 20 cm depth		3.41	62.7
- 40 cm depth		3.44	63.2
- 60 cm depth		3.52	64.7
- 80 cm depth		3.65	67.1
- 100 cm depth		3.78	69.5

Furthermore, for the non-coated specimens, the specimens subjected to water curing with intermittent water release exhibited higher water absorption (5.44%) compared to the non-coated specimens under the 48-hour air curing condition. However, the water absorption values for the surface coated specimens at different depths demonstrated a notable reduction compared to the non-coated specimens. This suggests that the ICW coating was more effective in preventing water ingress under the water curing condition compared to the 48-hour air curing condition. The water absorption rate values for the surface coated specimens ranged between 3.41% and 3.78%. At coring depths of 20, 40, 60, 80, and 100 cm, the water absorption indicated an improvement of 37.3%, 36.8%, 35.3%, 32.9%, and 30.5%, respectively, compared to the non-coated specimens.

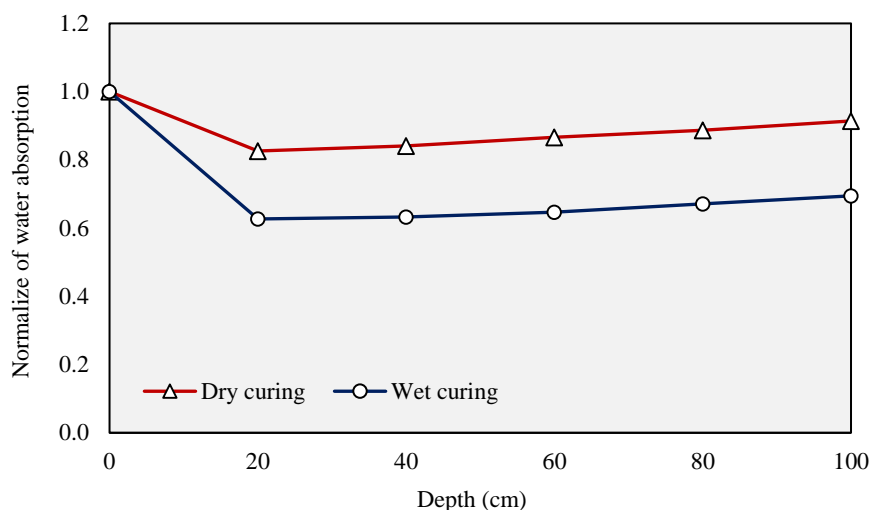


Figure 7 Water absorption of core specimen compared with control

The findings depicted in Figure 7 illustrate the progression of secondary crystallization, as evidenced by the declining absorption values observed in the coring cylinder specimens under both curing conditions. The initial measured segment, encompassing a range of 0 to 20 cm within the coring cylinder, exhibited the greatest degree of impact. Following a period of 28 days, the absorption value exhibited a decrease to below 17% and 37% of the control specimen for the dry curing method and the water curing method with intermittent water release, respectively. The second depth (20–40 cm) was less influenced by crystallization compared to the first section, with a reduction of approximately 15% and 36%. The remaining sections that were impacted by the application of the crystallizing coating exhibited marginally diminished reductions, while the absorption values experienced a slight increase. According to the findings of Reiterman and Pazderka [31], the utilization of this measurement technique resulted in an enhanced ability to accurately determine the extent to which the crystalline coating affected the surface layer in terms of depth. The efficacy of the applied coating in terms of sealing is observable after a period of 28 days, specifically for the concrete surface section measuring 0–20 cm. This is indicated by the absorption coefficient values reaching 50%. The rate of decrease in the inward progression of the crystalline effect exhibits a gradual pattern. The findings also suggest that the efficacy of the ICW treatment is enhanced as the depth of the coating increases during the process of underwater curing with intermittent water release. The efficiency of secondary hydration induced by the crystalline coating is contingent upon the quantity of water present within the concrete, with a greater degree of saturation leading to increased range and speed [31, 32]. As the coring depth decreases, more crystals are formed within the concrete matrix, resulting in a denser and more impermeable structure. The aforementioned discovery aligns with prior research investigations that have established an inverse relationship between depth profile and water impermeability. The significant reduction in water absorption observed in the coated specimens can be attributed to the unique mechanism of integral crystalline waterproofing. This mechanism involves the formation and growth of crystals within the concrete matrix in the presence of moisture, leading to a significant decrease in the infiltration of water across all phases [32, 40]. When applied to the concrete surface, the coating initiates a crystallization process within the concrete matrix, forming insoluble crystals that penetrate the capillary network and microcracks. These crystals effectively block the pathways for water penetration, thereby enhancing the water impermeability of the concrete [8, 18, 23, 25, 32]. Furthermore, Cappellesso et al. [18], conducted an observation which revealed that the utilization of ICW coating demonstrates superior efficiency when compared to the use of ICW admixture. The efficiency of ICW coatings can be attributed to their composition, which includes cement mixed with resin and applied in paste form. This mixture has the ability to penetrate the pores of the surface, undergo crystallization, and ultimately provide a buffering effect.

3.2 Compressive strength

The compressive strength data obtained under various curing conditions are displayed in Table 3. The findings of this study offer valuable insights regarding the influence of ICW on the compressive strength of concrete coring specimens. The averages were derived by conducting tests on three-cylinder specimens with an age of 28 days.

For the non-coated specimens, the 48-hour air curing condition resulted in a compressive strength of 36 MPa, which serves as the baseline or control value for comparison with the surface coated specimens. The compressive strength values for the surface coated specimens at different depth profiles exhibited variations compared to the non-coated specimens. The compressive strength values decreased as the coring depth of the specimens increased. The compressive strength rate values for the surface coated specimens ranged between 36 and 42 MPa. At coring depths of 20, 40, 60, 80, and 100 cm, the compressive strength indicated an improvement of 13.9%, 18.3%, 6.7%, 5.6%, and 0%, respectively, compared to the non-coated specimens. Interestingly, the compressive strength of the specimens subjected to water curing with intermittent water release was higher than that of the non-coated specimens under the same curing condition. This suggests that the ICW coating may have contributed to the improvement in compressive strength, even in the

presence of water curing. The surface coated specimens at all depth profiles exhibited higher compressive strength values compared to the non-coated specimens.

However, the compressive strength values for the surface coated specimens at different depths demonstrated a notable decrease with increased depth profile. The compressive strength values for the surface coated specimens ranged between 42.2 and 46.6 MPa. At coring depths of 20, 40, 60, 80, and 100 cm, the compressive strength indicated an improvement of 17.4%, 15.8%, 14.5%, 13.2%, and 11.1%, respectively, compared to the non-coated specimens

Table 3 Compressive strength of core specimen with different curing condition

Type of specimen	Curing condition	Compressive strength (MPa)	Compared with control (%)
Non coating	48-hour air curing	36.0	100.0
Coating			
- 20 cm depth		41.0	113.9
- 40 cm depth		39.0	108.3
- 60 cm depth		38.4	106.7
- 80 cm depth		38.0	105.6
- 100 cm depth		36.0	100.0
Non coating	Water curing with intermittent water release	38.0	100.0
Coating			
- 20 cm depth		44.6	117.4
- 40 cm depth		44.0	115.8
- 60 cm depth		43.5	114.5
- 80 cm depth		43.0	113.2
- 100 cm depth		42.2	111.1

The observed enhancement in compressive strength can be ascribed to the distinctive characteristics of the ICW material. The formation of crystals within the concrete matrix enhances the interfacial bonding and overall cohesion of the concrete structure. This improved bond strength between the cementitious matrix and aggregates contributes to the enhanced compressive strength of the coated specimens [9, 40]. Furthermore, the incorporation of ICW can result in a homogeneous distribution within the structure, effectively occupying the pores via capillary crystallization reactions. This phenomenon contributes to an increased density of the overall structure [5, 18, 20]. Furthermore, the effect of depth profile on compressive strength was not as pronounced as on water absorption. The variations in compressive strength values among the surface coated specimens at different depths were relatively minor. This suggests that the impact of depth profile on compressive strength may be less significant compared to its effect on water absorption. The findings suggest that the implementation of the ICW treatment has a beneficial impact on the compressive strength of the concrete specimens obtained through coring. Although the concrete depth profile is not a major factor in compressive strength improvement [13, 21, 41], it still contributes to enhancing the overall strength of the concrete [9, 25]. These findings align with previous studies that have reported the beneficial effects of ICW on compressive strength, confirming the results reported by Azarsa et al. [4] and Jalali and Afgan [19].

Upon analyzing the data presented in Figure 8, it is evident that the specimens containing ICW consistently demonstrate superior compressive strength. Additionally, the incorporation of ICW leads to a reduction in water absorption for all concrete coring specimens.

This observation demonstrates the efficacy of the waterproofing treatment in enhancing both the structural integrity and hydrophobic properties of the concrete. The correlation between compressive strength and water absorption is intricate, as it is impacted by multiple factors including density, pore structure, and interfacial bonding within the concrete material.

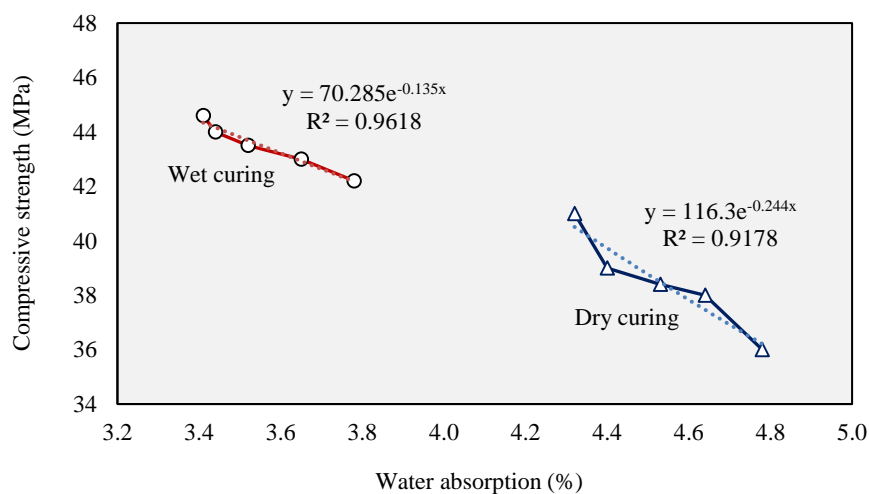


Figure 8 Relationship between water absorption and compressive strength

3.3 Ultrasonic pulse velocity

The test results for the various curing conditions of the UPV are displayed in Table 4. The UPV values provide insights into the integrity and quality of the concrete specimens, particularly in terms of their soundness and homogeneity.

Under the 48-hour air curing condition, the non-coated specimens exhibited an average pulse velocity of 3353 m/s. When a coating of ICW was applied to the concrete surface, the UPV values showed variations compared to the non-coated specimens. The UPV values for the surface coated specimens at different coring depths ranged between 3331 and 3982 m/s. The specimens with a surface coating at a coring depth profile of 0-20 cm demonstrated the highest pulse velocity of 3982 m/s, representing an increase of 18.8% compared to the non-coated specimens. As the coring depth increased to 40, 60, and 80 cm, the pulse velocity values gradually decreased but still remained higher than the non-coated specimens, except at the coring depth of 100 cm. Similarly, for the water curing with intermittent water release condition, the non-coated specimens exhibited an average pulse velocity of 3755 m/s. The UPV values for the surface coated specimens at different coring depths ranged between 3766 and 4288 m/s. At coring depths of 20, 40, 60, 80, and 100 cm, the pulse velocity indicated an improvement of 14.2%, 10.5%, 7.0%, 3.2%, and 0.3%, respectively, compared to the non-coated specimens.

Table 4 UPV of core specimen with different curing condition

Type of specimen	Curing condition	UPV (m/s)	Compared with control (%)
Non coating	48-hour air curing	3353	100
Coating			
- 20 cm depth		3982	118.8
- 40 cm depth		3824	114.1
- 60 cm depth		3654	109.0
- 80 cm depth		3516	104.9
- 100 cm depth		3331	99.3
Non coating	Water curing with intermittent water release	3755	100
Coating			
- 20 cm depth		4288	114.2
- 40 cm depth		4150	110.5
- 60 cm depth		4018	107.0
- 80 cm depth		3875	103.2
- 100 cm depth		3766	100.3

The findings suggest that the implementation of ICW has a beneficial impact on the UPV of the core concrete specimens, irrespective of the curing condition. The efficacy of the ICW coating is presumably influenced by the attributes of the porous structure of the concrete. The treatment is anticipated to be advantageous for concretes that exhibit higher density and possess a closely porous system [31]. The coring depth profile appears to have a slight influence on the pulse velocity, with specimens coring at shallower depths exhibiting higher values. The enhanced uniformity and structural soundness of the concrete matrix can be ascribed to the crystalline growth stimulated by the waterproofing material. The higher pulse velocity values observed in the surface coated specimens indicate better soundness and improved structural quality. However, even with deeper coring depths, the pulse velocity values remain higher than the non-coated specimens, indicating the beneficial effects of ICW on the overall concrete quality. Moreover, ICW enhances bond strength and reduces the presence of internal defects within the concrete [9], leading to faster transmission of ultrasonic waves. This demonstrates the effectiveness of the waterproofing treatment in enhancing the overall integrity and durability of the concrete [7].

It is important to note that the UPV test provides valuable insights into the quality of the concrete specimens, but it should be used in conjunction with other test methods to obtain a comprehensive evaluation. The combination of compressive strength, water absorption, and The UPV tests offer a comprehensive assessment of the correlation between the waterproofing treatment and the properties of concrete. The relationship between UPV and water absorption is significant, as illustrated in Figure 9. The specimens with lower water absorption, such as those with a coring depth of 20 cm, exhibit higher pulse velocity values. This indicates a stronger and more compact concrete structure with reduced porosity and internal defects. The improved homogeneity and soundness achieved through the waterproofing treatment contribute to faster transmission of ultrasonic waves, resulting in higher pulse velocity values. Furthermore, the results demonstrate the influence of coring depth on water absorption and pulse velocity. As the coring depth increases, the water absorption values and pulse velocity tend to show slight variations. However, even at greater coring depths, the coring specimens consistently exhibit lower water absorption and higher pulse velocity compared to the non-coated specimens. This highlights the long-lasting effects of ICW in mitigating water ingress and enhancing the overall performance of the concrete.

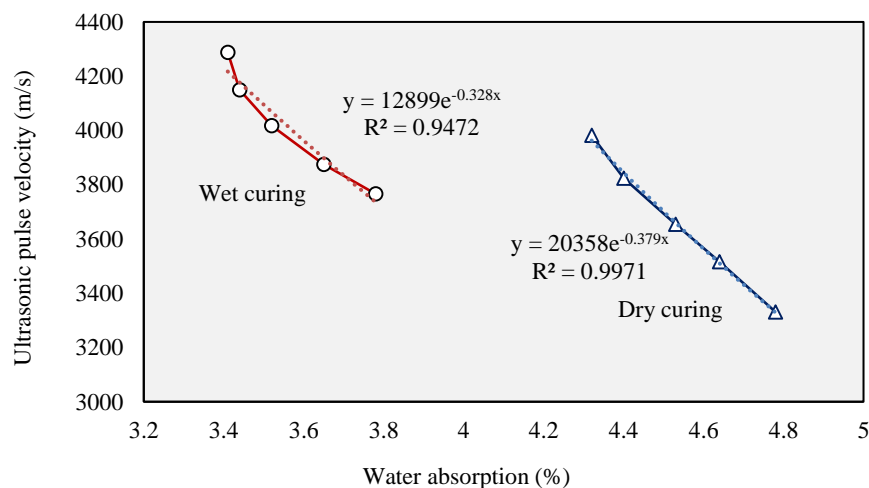


Figure 9 Relationship between water absorption and ultrasonic pulse velocity

3.4 Microstructural analysis

SEM-EDS was utilized in order to visually observe the emergence of novel phases within the empty regions resulting from ICW, and subsequently determine their chemical composition. The microstructural analysis conducted using SEM-EDS on freshly fractured surfaces of the coring concrete specimens is shown in Figure 10 and Figure 11. The SEM-EDS test yielded interesting findings regarding the presence of titanium compounds in the surface coated specimens treated with ICW.

In the specimens that were not coated and subjected to a 48-hour air curing process, no titanium compounds were observed in the coring specimens, as depicted in Figure 10(a). Nevertheless, it was observed that titanium compounds were present in the surface coated specimens at depths of 20 cm and 40 cm, indicating that the ICW material facilitated the incorporation of titanium into the concrete matrix, as depicted in Figure 10(b)-Figure 10(c). In contrast, it was observed that titanium compounds were not detected at the deeper coring depths of 60 cm, 80 cm, and 100 cm (Figure 10(d)), suggesting that the distribution of titanium was restricted to shallower depths.

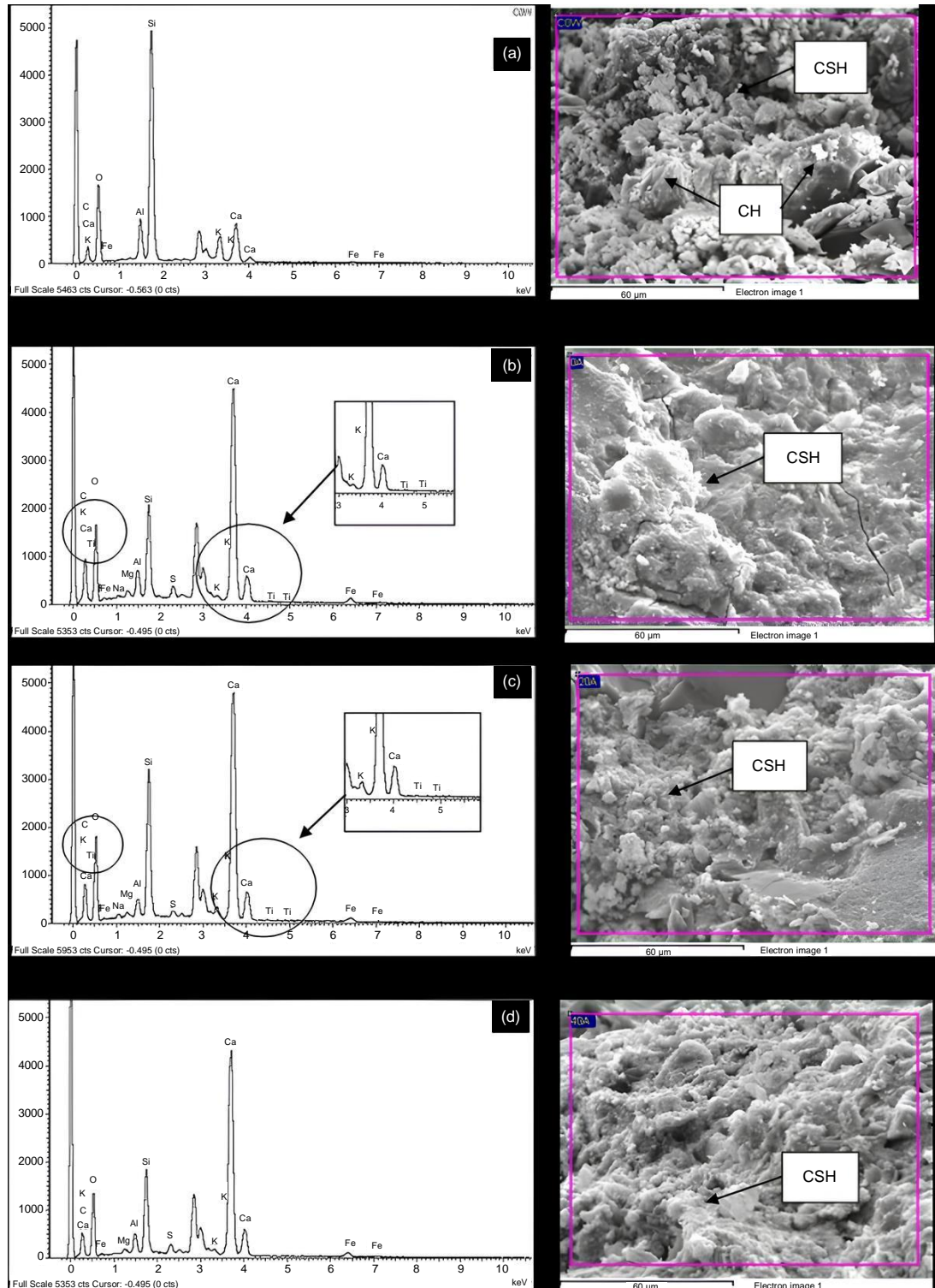


Figure 10 SEM-EDS analysis of the coring concrete specimens under 48-hour air curing

The observed improvements cannot be fully explained by the presence of titanium compounds alone, especially since they are not found beyond a depth of 40 cm. To offer a coherent explanation, it is crucial to consider the complex interaction of various factors. The self-healing crystalline network formation likely enhances properties by preventing water ingress and strengthening the concrete matrix. Furthermore, it is important to take into account the capillary structure of the concrete, the distribution of the ICW material within the matrix, and the interaction between the ICW crystals and the existing minerals in the concrete. The enhanced compressive strength can be attributed to several factors, such as improved hydration caused by the presence of ICW, decreased porosity resulting from crystalline growth, and a more interconnected microstructure due to crystal growth within capillaries. Moreover, the lack of titanium compounds beyond a distance of 40 cm could indicate a localized impact of the ICW substance. The efficiency of the crystallization process may be higher in shallower depths due to factors such as increased moisture availability, which promotes a more extensive reaction. The localized effect may account for the observed improvements at specific depths, highlighting the importance of accurate application methods and curing conditions.

In addition, it was observed that the non-coated specimens, when subjected to water curing with intermittent water release, did not exhibit the presence of titanium compounds in the coring samples, as depicted in Figure 11(a). Nevertheless, the specimens with surface coating exhibited the presence of titanium compounds across the entire depth profiles, encompassing depths of 20 cm (as depicted in Figure 11(b)), 40 cm (as depicted in Figure 11(c)), 60 cm, 80 cm, and 100 cm (as depicted in Figure 11(d)).

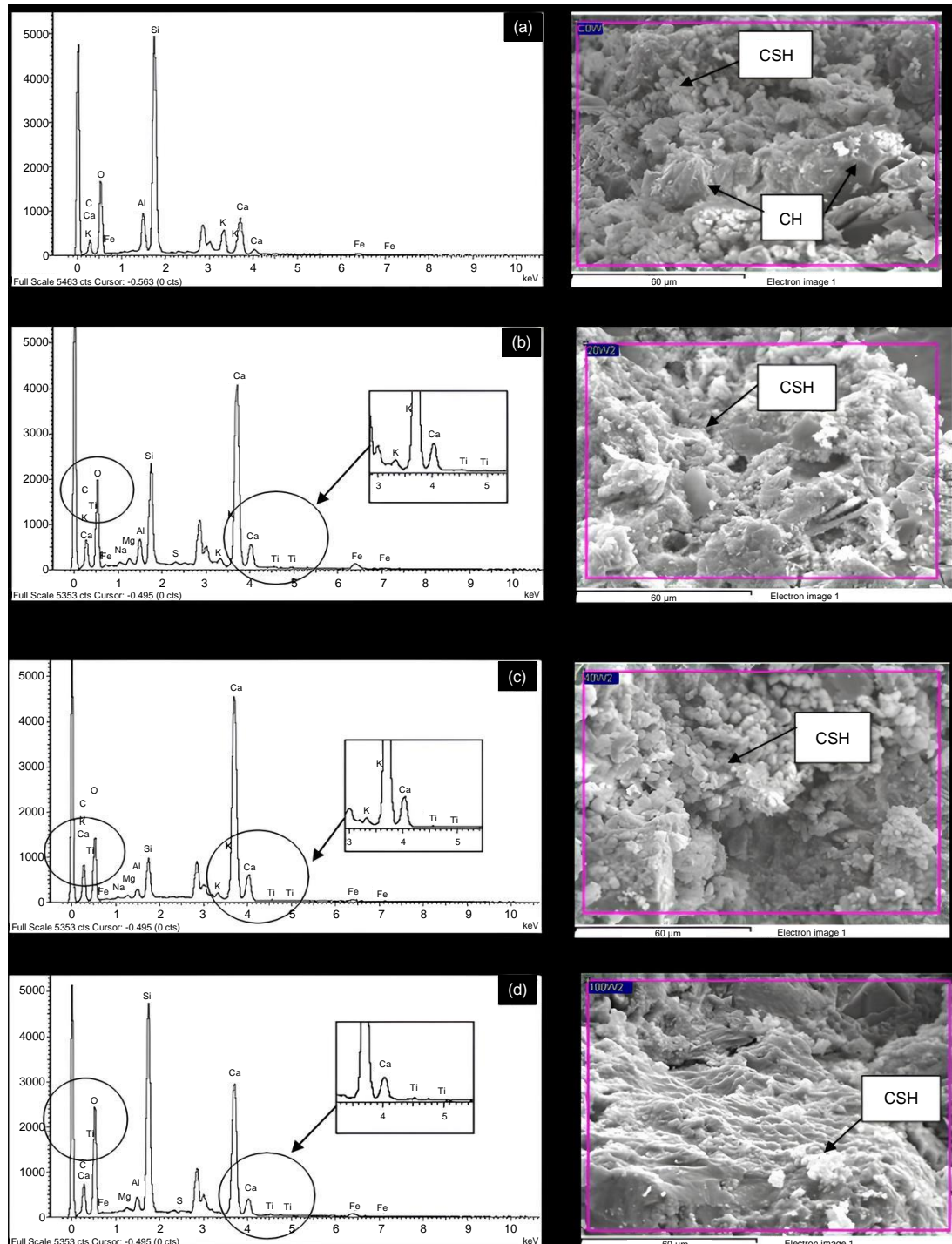


Figure 11 SEM-EDS analysis of the coring concrete specimens under water curing with intermittent water release condition

The identification of titanium compounds within the coated specimens indicates that the ICW material played a significant role in facilitating the incorporation of titanium into the concrete matrix. The presence of titanium in the ICW material was confirmed through XRF analysis, which provided insight into its chemical composition. It is noteworthy that titanium is not a customary constituent of cement compositions. The EDS analysis conducted in cement studies revealed the presence of the predominant elements found in hydration products of cement, such as calcium, oxygen, and silicon. Additionally, smaller quantities of magnesium, aluminium, and potassium were observed on the surface of ICW-modified specimens [23]. The identification of titanium compounds within the coated specimens at various depths underscores the efficacy of the ICW material in infiltrating the concrete matrix. The occurrence of titanium compounds in the processed concrete can be ascribed to the chemical interaction between the ICW substance and the particles existing within the concrete matrix during the process of water reduction. This chemical reaction resulted in the formation of titanium compounds, which improved the density of the concrete and increased its resistance to water permeation. Additionally, the reaction increased the amount of crystalline calcium hydroxide, particularly during the early stages of hydration. This accelerated the formation of calcium silicate hydrate gel and ultimately increased the strength of the concrete specimens [42-45]. Furthermore, the microstructural analysis indicated that the incorporation of titanium compounds in the concrete samples treated with the waterproofing substance can be attributed to the interaction between external water and the calcium hydroxide compounds. The dissolution of these compounds occurs, leading to their reaction with the particles of the waterproofing material, ultimately resulting in the formation of compounds containing titanium. The categorization of these compounds as polymers implies their potential role in augmenting the water impermeability and overall durability of concrete.

4. Conclusions

In summary, the primary objective of this research was to examine the efficacy of ICW and its influence on the microstructural characteristics of concrete. Based on thorough examination and rigorous analysis, the subsequent deductions can be made:

- ICW demonstrated its effectiveness in enhancing the water impermeability of concrete. The specimens that were coated on the surface demonstrated a decrease in water absorption in comparison to the specimens that were not coated, indicating improved resistance against water ingress.
- Compressive strength testing showed that the surface coated specimens exhibited higher compressive strength values compared to the non-coated samples. This improvement in compressive strength suggests that ICW not only enhances water impermeability but also contributes to the overall strength and durability of the concrete.
- UPV testing provided additional evidence of the effectiveness of ICW. The surface coated samples demonstrated higher UPV values compared to the non-coated samples, indicating a denser and more structurally sound concrete matrix. This finding further supports the enhanced performance of the concrete in relation to its durability and ability to resist water infiltration.
- SEM-EDS revealed the presence of titanium compounds in the concrete samples treated with ICW. These compounds, identified as polymers, played a crucial role in improving the water impermeability of the concrete. The interaction between external water and calcium hydroxide compounds in the concrete resulted in the formation of titanium-containing compounds, contributing to the enhanced performance of the waterproofing system.
- The results highlight the importance of proper application and curing conditions for ICW to achieve optimal effectiveness. The curing condition, whether air curing or water curing with intermittent water release, influenced the presence of titanium compounds and ultimately affected the water absorption properties of the concrete.
- Further research can focus on long-term performance evaluation, including the durability and sustainability aspects of ICW, to assess its effectiveness in various environmental conditions and ensure its reliable application in real-world construction projects.

5. Acknowledgements

The authors express their gratitude to the Institute of Science and Technology Research at King Mongkut's University of Technology North Bangkok and Rajamangala University of Technology Suvarnabhumi for facilitating laboratory testing in this study. Additionally, the authors would like to acknowledge the support received from Rajamangala University of Technology Phra Nakhon, Ultimatum Technology Co., Ltd., and Thai Obayashi Corporation Limited.

6. References

- [1] Basheer L, Kropp J, Cleland DJ. Assessment of the durability of concrete from its permeation properties: a review. *Constr Build Mater.* 2001;15(2-3):93-103.
- [2] Medeiros-Junior RA. 3 - Impact of climate change on the service life of concrete structures. In: Pacheco-Torgal F, Melchers RE, Shi X, Belie ND, Tittelboom KV, Sáez A, editors. *Eco-Efficient Repair and Rehabilitation of Concrete Infrastructures*. Cambridge: Woodhead Publishing; 2018. P. 43-68.
- [3] Li G, Huang X, Lin J, Jiang X, Zhang X. Activated chemicals of cementitious capillary crystalline waterproofing materials and their self-healing behaviour. *Constr Build Mater.* 2019;200:36-45.
- [4] Azarsa P, Gupta R, Azarsa P, Biparva A. Durability and self-sealing examination of concretes modified with crystalline waterproofing admixtures. *Materials (Basel).* 2021;14(21):6508.
- [5] Tan Y, Zhao B, Yu J, Xiao H, Long X, Meng J. Effect of cementitious capillary crystalline waterproofing materials on the mechanical and impermeability properties of engineered cementitious composites with microscopic analysis. *Polymers.* 2023;15(4):1013.
- [6] Muhammad NZ, Keyvanfar A, Majid MZA, Shafaghat A, Mirza J. Waterproof performance of concrete: a critical review on implemented approaches. *Constr Build Mater.* 2015;101:80-90.
- [7] Lim S, Kawashima S. Mechanisms underlying crystalline waterproofing through microstructural and phase characterization. *J Mater Civ Eng.* 2019;31(9):04019175.

- [8] Zhang Y, Zuo L, Yang J, Cai X, Zhao Y, Zeng X. Effect of cementitious capillary crystalline waterproofing coating on the gas permeability of mortar. *Struct Concr.* 2019;20(5):1763-70.
- [9] Hu X, Xiao J, Zhang Z, Wang C, Long C, Dai L. Effects of CCCW on properties of cement-based materials: a review. *J Build Eng.* 2022;50:104184.
- [10] Romer M, Holzer L, Pfiffner M. Swiss tunnel structures: concrete damage by formation of thaumasite. *Cem Concr Compos.* 2003;25(8):1111-7.
- [11] Bastidas-Arteaga E, Sánchez-Silva M, Chateaneuf A, Silva MR. Coupled reliability model of biodeterioration, chloride ingress and cracking for reinforced concrete structures. *Struct Saf.* 2008;30(2):110-29.
- [12] Bertolini L, Elsener B, Pedferri P, Redaelli E, Polder RB. *Corrosion of steel in concrete: prevention, diagnosis, repair.* 2nd ed. Weinheim: Wiley-VCH; 2013.
- [13] Al-Rashed R, Al-Jabari M. Concrete protection by combined hygroscopic and hydrophilic crystallization waterproofing applied to fresh concrete. *Case Stud Constr Mater.* 2021;15:e00635.
- [14] Skutnik Z, Sobolewski M, Koda E. An experimental assessment of the water permeability of concrete with a superplasticizer and admixtures. *Materials.* 2020;13(24):5624.
- [15] Almusallam AA, Khan FM, Dulaijan SU, Al-Amoudi OSB. Effectiveness of surface coatings in improving concrete durability. *Cem Concr Compos.* 2003;25(4-5):473-81.
- [16] Zhang C, Guan X, Li J, Li Y, Lu R. Coupling effect of cementitious capillary crystalline waterproof material and exposure environments on self-healing properties of engineered cementitious composites (ECC). *J Build Eng.* 2023;63:105471.
- [17] Zhang C, Guan X, Lu R, Li J, Li Y. Effect of cementitious capillary crystalline waterproof material on the various transport properties of cracked cementitious composites. *Constr Build Mater.* 2023;365:130138.
- [18] Cappellessio VG, dos Santos Petry N, Dal Molin DCC, Masuero AB. Use of crystalline waterproofing to reduce capillary porosity in concrete. *J Build Pathol Rehabil.* 2016;1:9.
- [19] Jalali UH, Afgan S. Analysis of integral crystalline waterproofing technology for concrete. *Int Res J Eng Technol.* 2018;5(10):1076-85.
- [20] Kheaw-on T, Khomwan N, Sujavanich S. The effect of crystalline waterproofing materials on accelerated corrosion of steel reinforcement in concrete. *Int J Civ Eng.* 2021;19:699-716.
- [21] Gojević A, Ducman V, Netinger Grubeša I, Baričević A, Banjad Pečur I. The effect of crystalline waterproofing admixtures on the self-healing and permeability of concrete. *Materials.* 2021;14(8):1860.
- [22] Roig-Flores M, Moscato S, Serna P, Ferrara L. Self-healing capability of concrete with crystalline admixtures in different environments. *Constr Build Mater.* 2015;86:1-11.
- [23] Azarsa P, Gupta R, Biparva A. Inventive microstructural and durability investigation of cementitious composites involving crystalline waterproofing admixtures and portland limestone cement. *Materials.* 2020;13(6):1425.
- [24] Tsampali E, Stefanidou M. The role of crystalline admixtures in the long-term healing process of fiber-reinforced cementitious composites (FRCC). *J Build Eng.* 2022;60:105164.
- [25] Wang C, Xiao J, Long C, Zhang Q, Shi J, Zhang Z. Influences of the joint action of sulfate erosion and cementitious capillary crystalline waterproofing materials on the hydration products and properties of cement-based materials: a review. *J Build Eng.* 2023;68:106061.
- [26] Biparva A. Integral crystalline waterproofing. *Struct Mag.* 2015:52-3.
- [27] Al-Rashed R, Al-Jabari M. Multi-crystallization enhancer for concrete waterproofing by pore blocking. *Constr Build Mater.* 2021;272:121668.
- [28] Al-Rashed R, Al-Jabari M. Managing thermal effects in waterproofed concrete with multi-crystallization enhancer. *Cement.* 2022;10:100050.
- [29] Al-Jabari M, Al-Rashed R, Ayers ME. Mitigation of alkali silica reactions in concrete using multi-crystalline intermixed waterproofing materials. *Cement.* 2023;12:100065.
- [30] Liu P, Liu M, Sha F, Chen Y, Zhi W, He S, et al. Preparation and performance investigation of a high efficiency cement permeation type waterproofing materials. *Constr Build Mater.* 2023;365:130140.
- [31] Reiterman P, Pazderka J. Crystalline coating and its influence on the water transport in concrete. *Adv Civ Eng.* 2016;2016:2513514.
- [32] Al-Rashed R, Jabari M. Dual-crystallization waterproofing technology for topical treatment of concrete. *Case Stud Constr Mater.* 2020;13:e00408.
- [33] Wang R, Ding Z, Zhang Y, Xu Y. Self-healing of high-performance engineered cementitious materials with crystalline admixture in the seawater environment. *J Build Eng.* 2023;63:105472.
- [34] Li D, Chen B, Chen X, Fu B, Wei H, Xiang X. Synergetic effect of superabsorbent polymer (SAP) and crystalline admixture (CA) on mortar macro-crack healing. *Constr Build Mater.* 2020;247:118521.
- [35] Hickman BSD, Macmillan S. Efficacy of crystalline waterproofing additives for basement concrete. *Proc Inst Civ Eng Constr Mater.* 2019;172(5):256-62.
- [36] Buapeng S, Wattayakorn G. Groundwater situation in Bangkok and its vicinity. *Proceedings of the HydroChange 2008 in Kyoto: hydrological changes and management from headwater to the ocean conference; 2008 Oct 1-3; Kyoto, Japan.* p. 1-7.
- [37] ASTM. ASTM C642: Standard test method for density, absorption, and voids in hardened concrete. West Conshohocken: ASTM International; 2021.
- [38] ASTM. ASTM C39: Standard test method for compressive strength of cylindrical concrete specimens. West Conshohocken: ASTM International; 2021.
- [39] ASTM. ASTM C597: Standard test method for ultrasonic pulse velocity through concrete. West Conshohocken: ASTM International; 2021.
- [40] Ravitheja A, Redd TCS, Sashidhar C. Self-healing concrete with crystalline admixture-a review. *J Wuhan Univ Technol Mater Sci Edit.* 2019;34:1143-54.

- [41] Pazderka J, Hájková E. Crystalline admixtures and their effect on selected properties of concrete. *Acta Polytech.* 2016;56(4):306-11.
- [42] Nazari A, Riahi S. The effect of TiO₂ nanoparticles on water permeability and thermal and mechanical properties of high strength self-compacting concrete. *Mater Sci Eng A.* 2010;528(2):756-63.
- [43] Diamantopoulos G, Katsiotis M, Fardis M, Karatasios I, Alhassan S, Karagianni M, et al. The role of titanium dioxide on the hydration of portland cement: a combined NMR and ultrasonic study. *Molecules.* 2020;25(22):5364.
- [44] Orakzai MA. Hybrid effect of nano-alumina and nano-titanium dioxide on Mechanical properties of concrete. *Case Stud Constr Mater.* 2021;14:e00483.
- [45] Jagadesh P, Nagarajan V, Karthik Prabhu T, Karthik Arunachalam K. Effect of nano titanium di oxide on mechanical properties of fly ash and ground granulated blast furnace slag based geopolymer concrete. *J Build Eng.* 2022;61:105235.