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Influence of Integral Crystalline Waterproofing on Concrete Properties: Dosage Impact and Microstructural Analysis

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Abstract

The present research study aims to investigate the properties and performance of concrete containing an integral crystalline waterproofing (ICW) admixture, added at an optimal dosage to resist water without compromising structural integrity. To achieve this, an experimental program was conducted on specimens with ICW dosage variations ranging from 0 to 4.8 kg/m^2. The impact on water absorption, pulse velocity, and microstructural characteristics was tested and analyzed using XRF and EDS techniques. The findings reveal that increasing the dosage decreases water absorption by approximately 43% at maximum dosage compared with the control. A 41% increase in pulse velocity indicates a denser concrete matrix. The principle of optimization is highlighted, as an overdose of ICW generates a non-structural crystalline gel at the bottom of the specimens. The optimum dosage range for ICW to improve water resistance without adverse effects on structural performance was determined to be 3.2 to 4.0 kg/m^2. This research introduces a novel approach by evaluating the comprehensive performance of concrete in relation to ICW dosage, providing valuable insights into the practical application of ICW admixture to enhance concrete quality and durability.

Keywords: Integral Crystalline Waterproofing; Water Absorption; Pulse Velocity; Microstructure Characteristics; Crystal Gel.

1. Introduction

Concrete is set apart as the fundamental material needed for contemporary constructions; it is versatile and can be utilized in virtually all structures—from skyscrapers and bridges to roads and dams. However, the natural deterioration of concrete, especially under exceptional environmental conditions, is a major challenge [1-3]. The high costs of repairing, maintaining, or replacing the civil engineering infrastructure indicate a need to minimize whole life costs by reducing interventions to minimize economic, environmental, and also social impacts [4]. Some of the factors affecting durability and life span are the quality of its components, the environment, and construction practice [5]. Concrete damage is caused, on the one hand, by temperature-induced factors and, on the other, by carbonation, chloride ion penetration, sulfate and acid attack, and alkali-aggregate reactions, which all reduce the structural and functional properties of the material [6-9]. The moisture contained within the concrete has a significant implication for concrete materials' failure because it becomes a catalyst that accelerates the material's destruction and decreases its long-term service life [10-12].

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Water penetration is one of the primary dangers to concrete structures, primarily due to the corrosion of steel reinforcements [13, 14]. Although the alkaline concrete environment remains intact, providing a durable passive coating over the embedded steel, penetrating water transports chlorides and other aggressive ions into the material that compromise this protective shell [15]. This results in corrosion, which is a process that causes an increased volume of rust and subsequently increased internal stresses, ultimately leading to cracking, spalling, and a loss of structural integrity [16]. Corrosion is the most critical factor governing the deterioration of the concrete structure, particularly in coastal areas and in cases where deicing salts are used, leading to chloride-induced damage [17-19]. Water penetration can also trigger a cycle of freezing and thawing in cooler climates [20]. Concrete is a porous material, and when water enters its pores and then freezes, the volume increases by about 9% [21]. This expansion creates internal pressures that easily exceed the tensile strength of the material, initiating microcracking, scaling, and surface deterioration repeatedly [22-25]. Moreover, leaching may occur due to the interaction of water percolating through concrete with calcium hydroxide, a by-product of cement hydration, and calcium-silicate-hydrate gel dissolving, creating voids and reducing density, thus strength [26-28].

To that effect, proper waterproofing solutions are a necessity in the realm of concrete construction. Commonly used traditional measures include waterproofing admixtures and surface-applied membranes and coatings; however, they have several limitations, such as being prone to mechanical damage, weathering, and requiring constant repair and reapplication [29-32]. On the other hand, integral waterproofing solutions have attracted immense interest due to their ability to provide concrete with greater durability through the direct incorporation of waterproofing materials within the concrete mix. ICW admixtures work from within the concrete matrix rather than on the surface, thus providing long-lasting protection against water infiltration [33-35]. ICW technology is based on the concept that, immediately after the crystalline admixture is added to the concrete mix, it reacts with water and unhydrated cement particles to form insoluble, needle-like crystals, which fill capillary pores and micro-cracks—effectively blocking pathways of water effectively [36-38]. This crystalline growth becomes reactivated in the presence of moisture, allowing the concrete to self-seal new cracks—further enhancing durability over time by self-sealing, meaning no further maintenance or repair work will be required [39, 40].

Various studies have been done regarding crystalline waterproofing technologies, focusing on the influences they have on cement-based materials in terms of microstructure and durability. For example, Lim & Kawashima [41] compared two forms of crystalline waterproofing agents: surface treatment and admixture. According to Jahandari et al. [33], surface treatments provide a dense coating with very minimal material penetration, while admixtures are effective in minimizing porosity within the matrix and hence provide more homogeneous protection. These technologies have the advantages of ease of application, reduced maintenance, and less long-term deterioration compared to surface coatings. However, the exact mechanism of the ICW and the associated complexation-precipitation reactions are still being debated between two schools of thought; a combined mechanism may perhaps be advocated for better long-term performances [42, 43]. Further research has demonstrated the efficiency of crystalline agents of new generations. Al-Rashed & Al-Jabari [44-47], and Al-Kheetan et al. [48] reported that MCE and DCE provide a substantial decrease in porosity and improvements in moisture transport and mechanical behavior. Wu et al. [49] found the optimum composition of cementitious capillary crystalline waterproofing agents (CCCW) that provided maximum impermeability and durability improvement. However, there is a trade-off: while these agents decrease water absorption by up to 80%, they also decrease compressive strength by roughly 10% [33]. This clearly requires a balanced approach to optimize waterproofing and mechanical performance.

CCCW has been studied by several researchers for its effect on different properties of concrete. Wang et al. [50] reported that CCCW consumes Ca^{2+}, which in turn increases sulfate resistance and crack-healing capacity, even when there is erosion by sulfate. Zhang et al. [51] tested four mixtures containing 0% to 4.5% of CCCW in different environments and demonstrated better mechanical properties and water impermeability. The CCCW at 4.5% healed cracks that were 83.33% smaller than those of the control, while the crack area closure rates increased by 167.98% in water-saturated calcium hydroxide and 132.44% in water. Zhang et al. [52] also developed a new formulation of CCCW, which enhanced substrate impermeability and achieved impermeability strength of 1.5 MPa, reducing carbonation by 12.37% over 14 days. According to Ndoj et al. [34], the compressive and flexural strengths of the concrete increased by up to 23% and 25%, respectively, at higher dosages of crystalline admixture, though with corresponding increases in cost and setting times. Songkha et al. [53] illustrated the importance of uniform application; they demonstrated that a well-applied ICW slurry yields a denser and more durable concrete matrix. Moreover, curing environments and material composition also influence the efficiency of ICW admixtures [42, 54]. For example, the inclusion of 15% FA, 2% CCCW, and polypropylene fibers enhanced compressive strength by 12.5%, increased tensile strength by 48.4%, and reduced permeability height by 63.6% [55]. ICW also showed promise in applications such as tunnel engineering for water leakage control [56].

Despite these progresses, crystalline waterproofing still faces challenges, most notably related to dosage optimization, cost, and long-term performance under aggressive conditions [33, 35, 39, 42, 43, 48, 57]. The main concern of Jahandari et al. [33] is the irregular performance of the commercial waterproofing admixtures, emphasizing that more research must be conducted to develop affordable alternatives and establish standard test methods. These gaps highlight the need for systematic research on how different levels of ICW dosage influence microstructural characteristics, mechanical properties, and the performance of concrete. Such a study is necessary to assess the effects of varying ICW dosages on a standard M28 grade concrete mix. The present study aims to investigate an optimum dosage by systematically varying the dosage and analyzing its effects on concrete properties to offer optimum waterproofing while improving or maintaining mechanical and durability characteristics. This will provide insight to guide the effective use of ICW admixtures in concrete construction, leading to more durable, resilient, and cost-effective structures.

2. Materials and Methods

2.1. Materials

2.1.1. Concrete Mix

The concrete used for this mix design targets the M28 grade—28 MPa, typically adopted for general construction purposes. The mix design aims to achieve a good balance of workability and strength suitable for the application of ICW admixtures. The mix used Ordinary Portland Cement (OPC) as the primary binder in compliance with ASTM C150 [58]. This choice was due to its wide use and extensive international documentation of hydration properties, which are essential for developing crystalline structures when used with ICW. Aggregates used in the mix included both fine and coarse components. The fine aggregates were natural river sand, with a fineness modulus of 2.6, and met ASTM C33 specifications [59]. The coarse aggregates were crushed limestone, with a maximum size of 20 mm, conforming to ASTM C33 [59]. Both types of aggregate were washed and dried to remove impurities that could affect hydration. Potable water free from deleterious impurities was used for both mixing and curing the concrete, with a water-to-cement ratio of 0.45 to ensure adequate workability until the specified strength levels were reached.

2.1.2. Integral Crystalline Waterproofing

The ICW admixture used for this study was a proprietary dry shake product specially designed to enhance water resistance in concrete. It is dark grey, in solid-powdered form, with a density of 1700 kg/m³ and a Blaine surface area of 3600 cm²/g. Its chemical composition includes 43% calcium oxide (CaO), 27% silicon dioxide (SiO_2), 1.4% ferric oxide (Fe_2O_3), and 1.2% aluminum oxide (Al_2O_3). Table 1 presents the physiochemical properties of the ICW admixture. Figure 1 shows the particle size distribution of the cement and ICW, comparing their characteristic particle sizes. This type of admixture was selected because it reacts with water and unhydrated cement particles, forming insoluble crystalline structures within the concrete matrix. By effectively blocking water pathways with these crystals, they are quite applicable in this present study, focused on improvement of concrete water resistance and longevity of the concrete because of the improvement in concrete durability.

Table 1. Physiochemical characteristics of the ICW admixture

Physical characteristics	
Appearance	Powder
Color	Dark grey
Physical state	Solid
pH solution	12-13
Density (kg/m^3)	1,700
Blaine surface area (cm^2/g)	3,600
Chemical compounds (%)	
CaO	43.0
SiO_2	27.0
Fe_2O_3	1.4
Al_2O_3	1.2
SO_3	1.8
MgO	1.2
K_2O	0.4
Na_2O	2.2
TiO_2	2.4

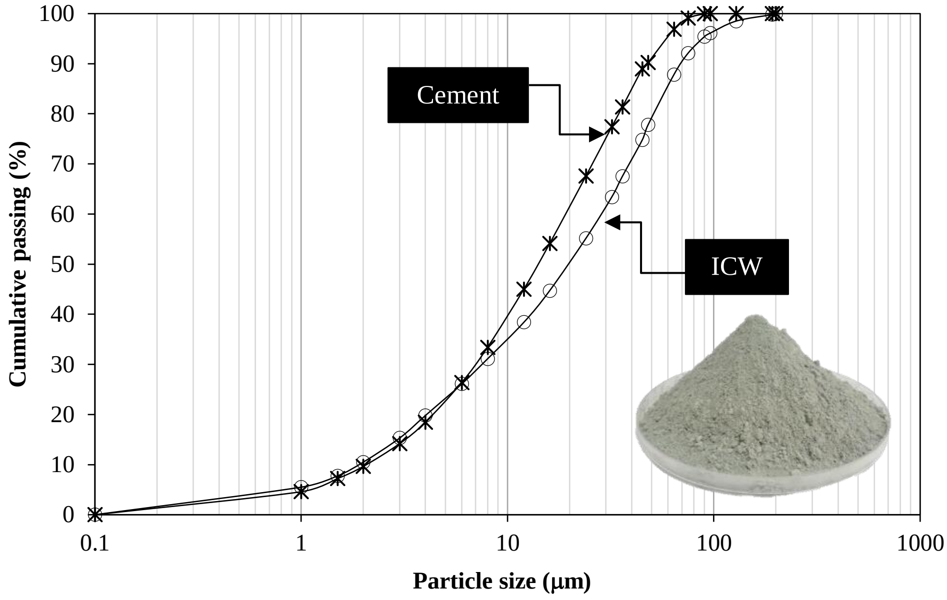

Figure 1. Particle size distribution of the cement and ICW admixture

2.2. Concrete Specimen Preparation

2.2.1. Mixing and Casting

Specimens were prepared at the laboratory using a concrete pan mixer. Mixing was conducted according to ASTM C192 [60]. The materials were batched by weight and introduced into the mixer in the following sequence: coarse aggregates, fine aggregates, cement, and water. The concrete mix was blended at a moderate speed for 3 min to ensure uniform distribution of all ingredients. The fresh concrete was then cast into molds measuring 100 cm × 100 cm × 10 cm.

2.2.2. Application of ICW Admixture

After placing, consolidating, and leveling fresh concrete, the ICW admixture was applied to the top surface using dry-shake method. The application was made during the initial set of the concrete, after the disappearance of bleed water. The ICW admixture was spread uniformly across the concrete surface at seven dosages: 0, 0.8, 1.6, 2.4, 3.2, 4.0, and 4.8 kg/m², ensuring that the admixture was evenly distributed over the entire area, as represented in Figure 2. In the study, the dosage range of the ICW admixture was extended beyond the manufacturer's recommended range from 1.6 to 3.2 kg/m², increasing the range from 0.8 to 4.8 kg/m². This expanded range was chosen to better understand the admixture's effects on concrete performance at both sub- and higher-than-recommended dosages. Once the ICW admixture was fully dispersed, the surface was trowelled with a steel trowel to press the material into the top layer of concrete to achieve the desired finish. Proper bonding between the concrete and the ICW crystals was established.

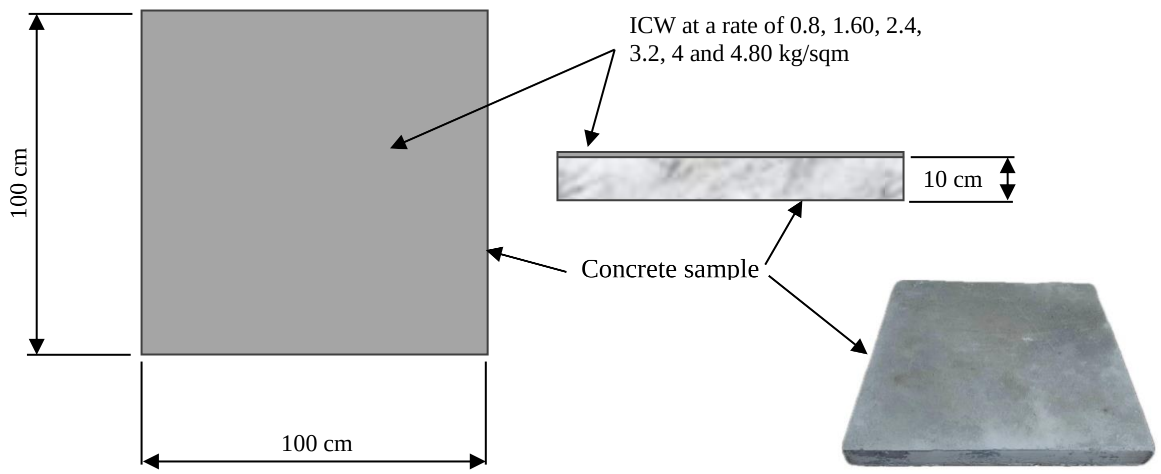

Figure 2. Application of ICW admixture on the surface of concrete specimens

2.2.3. Curing

The importance of proper curing cannot be overestimated in order to attain complete performance benefits from the ICW admixture. The concrete specimens were water-cured, with the top surface of the specimen kept continuously moist by the application of water to maintain a saturated condition, as shown in Figure 3. Curing conditions significantly affect the mechanical properties and crystal formation. Moist curing improves these properties more than atmospheric curing, emphasizing the importance of maintaining water availability [38]. This curing method was chosen to ensure uniform hydration of the cement and optimal crystalline growth. The curing period lasted 28 days to allow the specimens to develop the desired mechanical strength and durability.

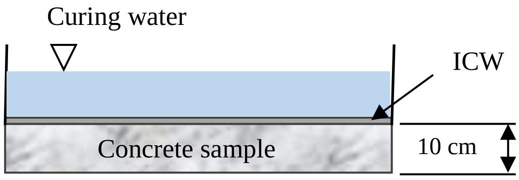

Figure 3. Curing conditions to create ICW

2.3. Testing Methods

To study the impact of ICW dosage on concrete performance, the following tests were conducted on the cured specimens: water absorption test, pulse velocity test, and micro-structural analysis such as X-ray fluorescence (XRF) and Energy Dispersive X-ray Spectroscopy (EDS). These tests provide comprehensive insights into the internal structure and durability of the treated concrete.

2.3.1. Water Absorption Test

Water absorption tests were conducted to explore the permeability of concrete specimens incorporated with various dosages of ICW. The testing followed procedures outlined in ASTM C642 [61]. Concrete samples were oven- dried at 105°C until a constant weight was reached, then submerged in water for 24 hours. The water absorption rate was determined by measuring the difference in weight before and after immersion, indicating the effectiveness of ICW in reducing porosity and water ingress. Concrete core samples, cubical in shape (10x10x10 cm), were used for the test, as shown in Figure 4.

Figure 4. Water absorption testing

2.3.2. Pulse Velocity Test

Ultrasonic pulse velocity (UPV) tests were performed using a portable ultrasonic NDT device, following the guidelines of ASTM C597 [62]. This test measures the velocity at which an ultrasonic pulse travels through concrete. Velocity in the concrete depends on the density and elasticity. Higher pulse velocities generally indicate a denser, more uniform internal structure with fewer voids. This test aimed to assess the integrity of the concrete matrix after ICW treatment at various dosages to determine the optimal dosage that maintains or enhances structural integrity. UPV wave velocity was measured on the plane surface of the concrete samples at distances of 20, 40, and 80 cm from the transmitter and receiver, respectively (see Figure 5).

Figure 5. Pulse velocity testing

2.3.3. Microstructural Analysis

XRF and EDS tests were performed to assess the impact of ICW dosage on internal structure and composition. In this study, the XRF technique was used to determine the elemental composition of concrete samples, focusing on the distribution and concentration of key components such as CaO, SiO_2, Al_2O_3, and Fe_2O_3. This involved grinding the concrete specimens into fine powder form and placing them inside an XRF spectrometer, enabling precise identification and quantification of elements essential to crystalline formation through the concrete matrix. Complementing the XRF technique, EDS was carried out to investigate in detail the properties of microstructure and the distribution of elements at a more localized level, as will be shown in Figure 6 below. EDS coupled with SEM was employed in the study of microstructural aspects of concrete specimens treated with different dosages of ICW, enabling a wider understanding of crystalline growth and the influence of the admixture on the concrete's internal structure. Therefore, the complementary uses of XRF and EDS were good enough to provide an analytical framework necessary to assess how different dosages of ICW might affect the chemical composition and properties in concrete at the microstructural level and, therefore, how to arrive at an optimal dosage regarding durability and waterproofing performance.

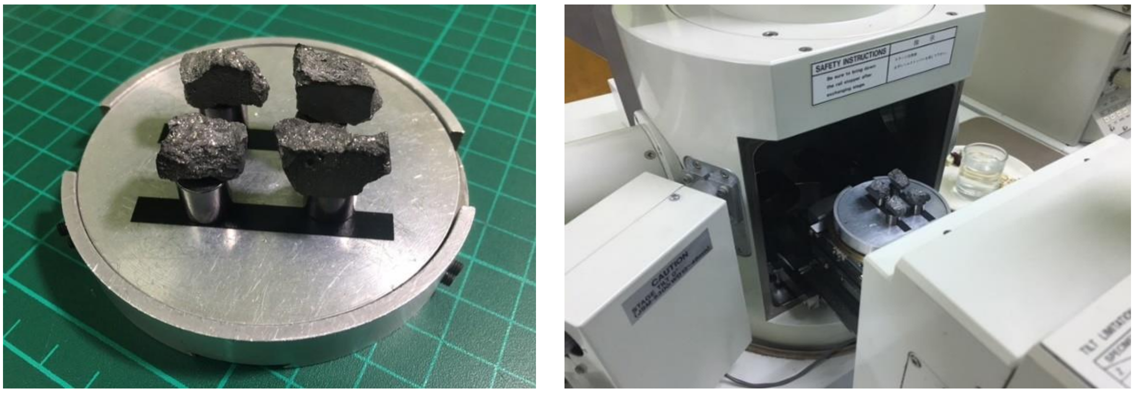

Figure 6. EDS analysis with scanning electron microscope

As a result, a better explanation and understanding of this studied process were illustrated in Figure 7, which is a visual representation of the research methodology flowchart that describes the main procedures followed in this study.

3. Results

3.1. Water Absorption

The water absorption tests revealed a notable reduction in water absorption as the dosages of ICW admixture increased, as shown in Figure 8. The control specimen, which did not contain any ICW admixture (0 kg/m² dosage), exhibited a water absorption rate of 6.33%, representing 100% of the baseline water absorption. With the addition of 0.8 kg/m² of ICW admixture, the water absorption dropped to 5.90%, which is 93% of the control value. When the dosage increased to 1.6 kg/m², the water absorption further decreased to 5.54%, equivalent to 88% of the control specimen. A more significant reduction occurred at a dosage of 2.4 kg/m², where the absorption rate really fell to 5.00%, representing 79% of the control specimen. At 3.2 kg/m², the water absorption rate was reduced to 4.35%, which corresponds to 69% of the control value. Further reduction was observed with an ICW dosage of 4.0 kg/m², resulting in an absorption rate of 4.05%, corresponds to 64% of the control. The lowest absorption rate was observed with an ICW dosage of 4.8 kg/m², where the absorption rate was 3.60%, which is only 57% of the control specimen's absorption rate.

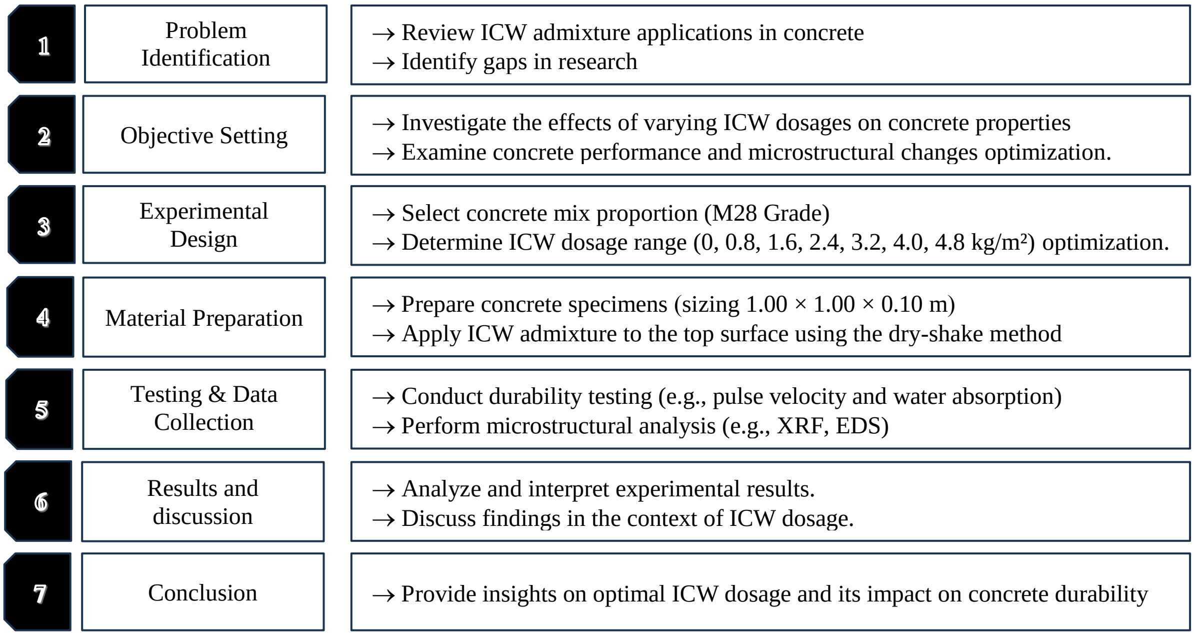

Figure 7. The flowchart of research methodology used in the study

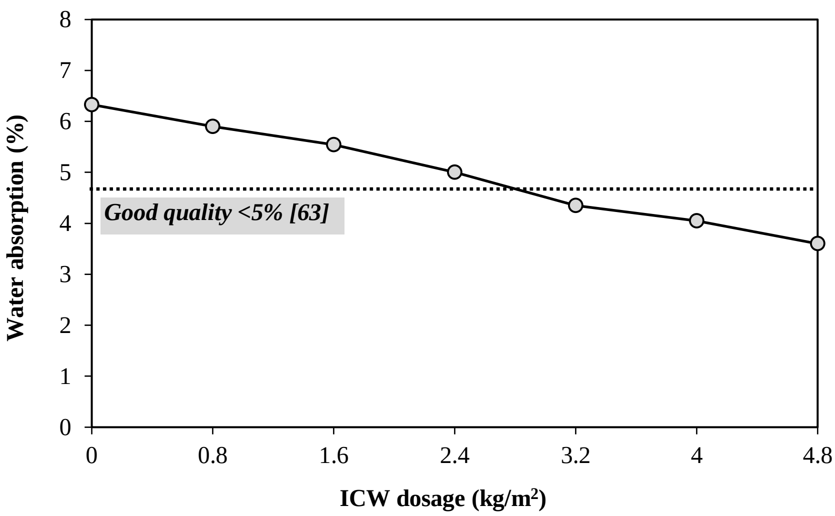

Figure 8. Water absorption with different ICW dosage at 28 days

From a theoretical perspective, water absorption is influenced by the capillary pore structure of concrete. According to Goloski [63], for good-quality concrete, water absorption should fall within the range of 4–6%, with values below 5% indicating a well-sealed matrix. In our study, a dosage of 2.4–4.8 kg/m² achieved a water absorption rate of between 3.60%–5.00%, bringing it below the critical 5% threshold. This is consistent with findings by previous researchers who reported that crystalline admixtures can reduce water absorption by 63.6% [55] up to 80% [33], and our highest dosage reduced it by 43%. The significant reduction in water absorption observed in this study can be explained by the formation of crystalline structures within the concrete matrix. These crystals block capillary pores, reducing permeability and preventing water ingress, as highlighted by previous researchers [41, 48-49, 52]. Moreover, the improvement in impermeability at higher dosages is in line with Al-Rashed et al.'s [44] findings that crystalline admixtures reduce porosity by filling larger pores with crystalline products, resulting in a denser concrete matrix. The density increased with crystalline admixtures dosed at 2% over those dosed at 1%, by a percentage in the range of 3.3–4.2%.

It follows from the water absorption test graphs that the ICW admixture was quite effective in bringing down the permeability of concrete. Also, such a significant reduction in water absorption observed with increasing dosages of ICW should be due to the formation of insoluble crystalline structures within the concrete matrix. Crystalline admixtures are known to reduce the water permeability of concrete significantly. The addition of crystalline admixtures to concrete mixes has been demonstrated to reduce the water permeability coefficient by a factor of threefold, indicating a strong reduction in the absorption of water [33, 63]. The sealer action in crystalline admixtures, which reduces water permeability, is a result of crystalline formation within the concrete matrix, thus blocking capillary pores and inhibiting the access of water [36].

As can be seen from the trend, even with the minimum dosage of 0.8 kg/m², ICW admixture begins to show a quantifiable advantage of absorbing less water compared to the control specimen, with a reduction of 7%. As the dosage rate increases, the extent of the reduction in water absorption becomes more pronounced. The reduction at 2.4 kg/m² is already 21%, illustrating the admixture's ability to create a very significant improvement in concrete impermeability even at slightly moderate dosages. This result tends to indicate that at this dosage level, enough crystalline material is formed to effectively seal a substantial portion of the capillary network within the concrete [33-34, 36]. The further reduction in water absorption at higher dosages, with the maximum reduction at 4.8 kg/m² (a reduction of 43% compared to the control), suggests that the performance of the ICW admixture increases with dosage. This improvement in performance is due to the greater availability of reactive compounds within the ICW admixture, which react with unhydrated cement particles and moisture to form a denser crystalline matrix [37, 39, 42, 49].

The reduction in water absorption in this study is consistent with results reported by several previous investigations. For instance, Al-Rashed & Al-Jabari [47] demonstrated that DCE could reduce water absorption by up to 75%, while in this study, a maximum reduction of 43% was achieved at 4.8 kg/m². Additionally, Zhang et al. [51] observed that higher ICW dosages improved water impermeability and reduced porosity. This aligns with our findings, where increased dosages led to reductions in water absorption, albeit with diminishing returns at higher concentrations. Similar to our study, Al-Kheetan et al. [50] found that excessive dosages could create larger void spaces, potentially reducing the effectiveness of the admixture due to trapped air voids in the concrete, a factor that should be carefully managed. In terms of water absorption thresholds, concrete treated with crystalline admixtures in Songkhla et al.'s [53] study exhibited water absorption values ranging from 4.32% to 4.78%, similar to our findings at the 4.0 kg/m² dosage (4.05%). This suggests that the admixture dosages used in our research are well-aligned with prior work and produce comparable improvements in concrete impermeability.

However, while the results show greater reductions in water absorption with increased dosages of ICW, it is also crucial to take into consideration the economic and practical implications of using larger quantities of the admixture [4, 31, 33, 47]. The continued decrease in water absorption is realized at a diminishing rate beyond the 3.2 kg/m² dosage, suggesting that there could be an optimum dosage level beyond which further additions of ICW do not contribute significantly to the development of waterproofing capability. Such plateauing could arise from saturation within the concrete matrix of reactive sites for the crystalline product, with further additions of ICW developing minimal amounts of new crystalline structures [53, 64]. Hence, though the maximum dosage tested was indeed 4.8 kg/m², which imparted maximum water absorption reduction, optimum dosage regarding cost and material usage can be achieved with a slightly lesser dosage.

3.2. Pulse Velocity

It can be seen from Figure 9 that the test results of pulse velocity showed a clear trend of higher ultrasonic pulse velocity with increasing dosages of the ICW admixture. The pulse velocity of the test specimen without the addition of any ICW admixture, considered as 0 kg/m² dosage, recorded 3548 m/s as 100% of the base value. At 0.8 kg/m², the pulse velocity increased to 3758 m/s, 106% of the control value. With further dosage at 1.6 kg/m², the increase in pulse velocity was 3996 m/s, equivalent to 113% of control. A dosage of 2.4 kg/m² produced a pulse velocity of 4105 m/s, equivalent to 116% of control, while 3.2 kg/m² further pushed the pulse velocity up to 4285 m/s, or 121% of control. Significant increases in pulse velocity continued at higher dosages—up to 4.0 kg/m²—reaching 4810 m/s (136% of control), and then increasing to 5012 m/s (141% of control) at the highest dosage, 4.8 kg/m².

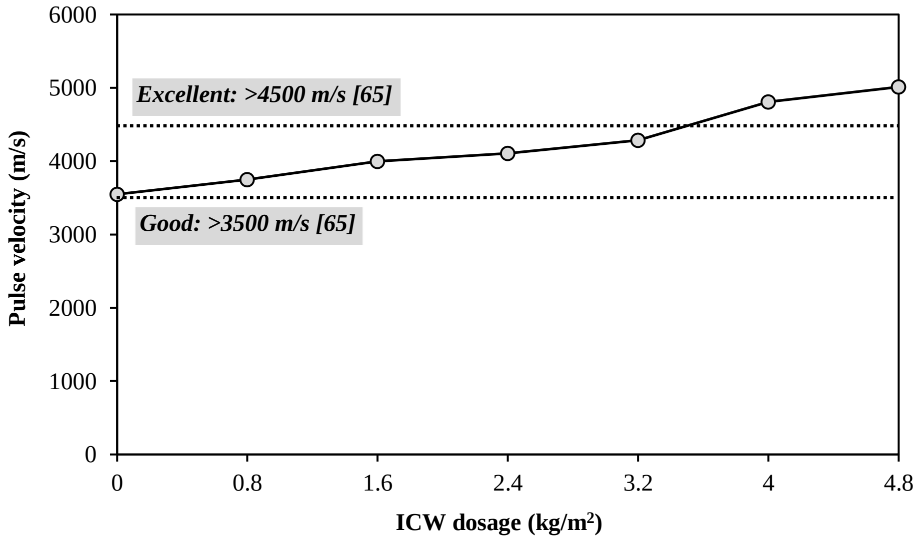

Figure 9. Pulse velocity with different ICW dosage at 28 days

Theoretically, pulse velocity is related to concrete density and integrity as an indirect measure of the propagation speed of ultrasonic waves through a material [53]. UPV depends on the internal structure, that is, the volume of voids and cracks inside, and generally on the homogeneity of the matrix. High pulse velocity values indicate a denser, less porous concrete with fewer internal defects [9, 14, 17]. According to established guidelines, concrete with UPV values in the range of 3500–4500 m/s is considered to be of "good" quality, while velocities above 4500 m/s fall into the "excellent" category [65]. Therefore, the test results showed that all the dosages treated by ICW were above the category "good", and the highest dosage of ICW admixture reached the "excellent" category. These test results reveal that the admixture based on ICW has an important contribution to enhancing compactness and quality. The pulse velocity test results provide useful information about the internal structure and integrity of the concretes treated with different dosages of the ICW admixture. Increasing the dosage of ICW increases the pulse velocity; this means that the ICW admixture was effective in enhancing the density and homogeneity of the concrete matrix. Generally, the higher the pulse velocity, the denser and more homogeneous the material is, with fewer internal voids or defects [2, 9, 24, 53]. This improvement in the concrete quality can be explained as a development of insoluble crystalline structures within the capillary pores, since the reaction of the ICW admixture with free water and unhydrated cement particles happened [33, 37, 64].

Even with the minimum dosage of 0.8 kg/m², the gain in pulse velocity is about 6%, which indicates that from this minimum value, the addition of ICW starts enhancing compactness and reducing porosity of concrete. It is further emphasized by the increase in dosage, so that at 1.6 kg/m², an incremental gain of 13% in pulse velocity is obtained. A trend now towards such dosages indicates that much more substantial crystalline growth might occur within the concrete matrix at such dosages, effectively filling capillary pores and microcracks that could otherwise weaken the structure [40, 42-43]. This corresponded to 2.4 kg/m² when the pulse velocity had increased to 116% of the control value, representing a gain of 16%. This increased improvement suggests a sharp decrease in the number of voids, with a rise in overall concrete density, likely because of more crystalline structure formation that effectively blocks the pathways for water and air ingress [43-44]. The further increase in pulse velocity at 3.2 kg/m² (121% of the control) and 4.0 kg/m² (136% of the control) indicates that the continued improvement of the internal quality of the concrete by means of the ICW admixture tends to make the concrete denser and more homogeneous [19, 39, 43].

These results confirm previous works highlighting crystalline admixtures' role in enhancing concrete density and reducing permeability. For instance, Lin et al. [39] mention that crystalline admixtures have the potential to enhance pulse velocity by promoting the closure of cracks and matrix densification. Uygunoğlu et al. [57] equally recorded pulse velocities greater than 4 km/s for concretes with ICW dosages within the range 1-5%, indicating good quality concretes. The observations are also confirmed by the present study, as dosage higher than 2.4 kg/m² of ICW-treated samples surpassed 4105 m/s, and they manifested as good-quality concrete. Another related study by Zhang et al. [52] illustrated that resultant crystalline admixtures reduce pore sizes and porosity and result directly in a rise in pulse velocity along with increased impermeability. It agrees with the present observations, where increased dosage of ICW results in denser matrices with reduced porosity and is reflected by the significant pulse velocity increases. Songkhla et al. [53] reports that UPV improvement up to 14.2% was found in surface-coated concrete. While this is impressive, the current study has even larger increases, such as a 41% increase in pulse velocity for the highest dosage of ICW, further indicating how effective concrete property improvement can be with incorporated ICW admixtures.

Of importance is the fact that the maximum pulse velocity reached was 5012 m/s at the highest dosage tested, 4.8 kg/m², which is 141% of the control. This sharp increase therefore signifies that at this dosage rate, the admixture has adequately interacted with the available cementitious material in providing a highly dense and compact concrete matrix. The result shows that this trend of reduced voids and increased density would contribute toward improved mechanical properties and durability observed in the concrete treated with ICW at this dosage level. The increasing trend in pulse velocity with an increase in higher dosages of ICW supports the hypothesis that ICW admixture improves the internal structure of concrete by promoting crystalline structure growth within the matrix [53, 64]. These structures not only block the capillary pores but also improve the mechanical integrity and durability of the concrete by means of reducing its permeability and possible ways of degradation [39-40]. The highest dosage of 4.8 kg/m², which yielded the greatest gain in pulse velocity, indicates a denser concrete matrix, which might imply better quality concrete. However, the implications of this regarding long-term structural strength require further investigation. A higher pulse velocity generally indicates lower porosity and resistance to water penetration, which is of course essential for long-term durability in aggressive environments.

3.3. X-Ray Fluorescence (XRF)

XRF tests gave comprehensive data on the elemental composition variation of different dosage ICW admixture-treated concrete specimens. This test was undertaken for the identification and quantification of main elements such as carbon (C), sodium (Na), magnesium (Mg), aluminum (Al), silicon (Si), sulfur (S), potassium (K), calcium (Ca), iron (Fe), titanium (Ti), rubidium (Rb), chlorine (Cl), zinc (Zn), strontium (Sr), and zirconium (Zr). Results obtained showed that there were significant trends in the concentrations of these elements as the dosage of the ICW increased from 0.0 kg/m² (control) to 4.8 kg/m² as shown in Table 2.

Table 2. The XRF analysis in the different dosage of ICW

Element (%)	ICW dosage (kg/m^2)						
	0.0	**0.8**	**1.6**	**2.4**	**3.2**	**4.0**	**4.8**
C	10.15	10.90	10.44	10.04	10.00	10.20	10.15
Na	0.34	0.32	0.66	0.50	0.22	0.11	0.26
Mg	0.65	0.55	0.62	0.32	0.60	0.70	0.58
Al	4.33	4.10	3.33	3.00	2.80	2.60	2.45
Si	33.47	33.49	34.74	35.33	36.79	36.80	37.00
S	0.44	0.46	0.28	0.53	0.41	0.51	0.28
K	14.00	11.64	11.24	10.54	10.20	9.62	9.20
Ca	34.15	34.60	35.78	36.50	37.51	37.71	38.09
Fe	2.44	2.80	1.50	1.80	0.00	0.10	0.00
Ti	0.00	1.10	1.33	1.36	1.40	1.60	1.90
Rb	0.01	0.01	0.05	0.05	0.04	0.04	0.05
Cl	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Zn	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Sr	0.01	0.02	0.01	0.01	0.02	0.00	0.02
Zr	0.01	0.01	0.02	0.02	0.01	0.01	0.02
Ca/Si	1.02	1.03	1.03	1.03	1.02	1.02	1.03

For the control specimen (0.0 kg/m² ICW dosage), the major elements included carbon (C) at 10.15%, silicon (Si) at 33.47%, potassium (K) at 14.00%, calcium (Ca) at 34.15%, and iron (Fe) at 2.44%. As the ICW dosage increased, notable changes were observed in these and other elemental concentrations, as shown in Figure 10. Silicon content, which is vital for the formations of silica-based compounds, increased gradually from 33.47% in the control to 37.00% at the highest dosage of ICW, which was 4.8 kg/m². The increased Si concentration shows an increased incorporation of silica-based materials within the concrete matrix and could be due to the reason that more silicate phases are formed in the presence of ICW admixture [33]. The Ca content also increased steadily from 34.15% for the control to 38.09% at 4.8 kg/m². The gradual increase in the Ca content in an increasing order indicates the development of C-S-H gel and other crystalline phases of Ca, making the concrete matrix more compact and impermeable [40, 45].

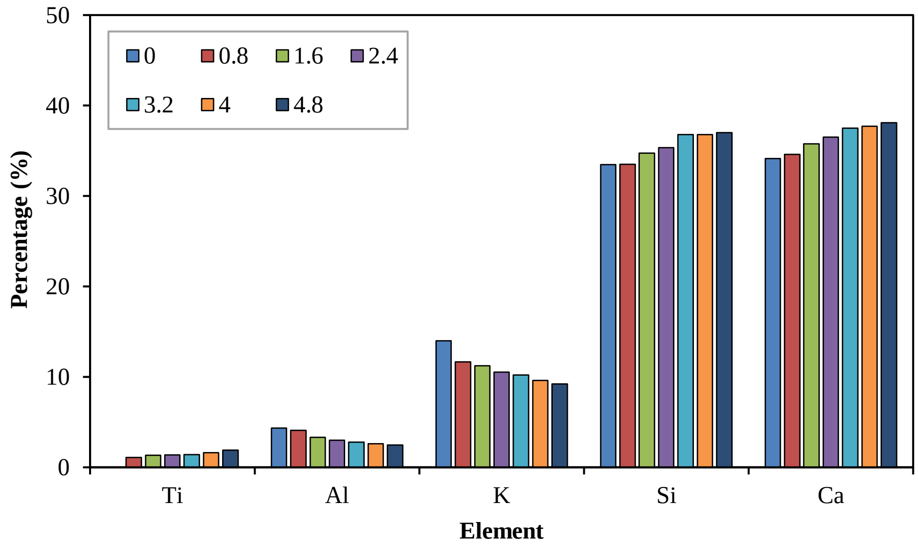

Figure 10. The XRF analysis of main element

The introduction of Ti in this work has been found to take place only in the ICW-treated samples, ranging from 1.10% at 0.8 kg/m² to 1.90% at 4.8 kg/m². The presence of Ti most likely shows the contribution of the ICW admixture to forming new crystalline structures or phases that increase the durability and resistance of concrete to water penetration [9, 53]. Other elements such as aluminum (Al) exhibited a downward trend from 4.33% in the control to 2.45% at the highest dosage. This reduction may be indicative that either the aluminum compounds are either being consumed in some complex crystalline structure formations or encapsulated within the denser matrix, hence reducing their availability [1, 50]. Iron (Fe) reduced significantly from the control value of 2.44% to 0.00% at the highest dosage, which may indicate a reduction in iron oxides or their inclusion into non-reactive forms within the ICW-treated concrete. Potassium (K) decreased from the control value of 14.00% to 9.20% at 4.8 kg/m². This may indicate that potassium compounds have become less available through incorporation into more stable crystalline forms [3, 53].

The observed trends in elemental composition align with existing theories on how crystalline admixtures affect concrete structure. Researchers have demonstrated that ICW promotes the formation of insoluble crystals, which seal capillary pores and enhance the impermeability of concrete. This process not only blocks water ingress but also refines the pore structure and densifies the concrete matrix, leading to improvements in both durability and mechanical properties [42, 49, 53, 55]. According to Hu et al. [42], calcium oxide (CaO) and silicon oxide (SiO_2) often make up a large proportion of the active chemicals in ICW materials. These chemicals permeate the structure, reacting with unhydrated cement particles to generate insoluble crystalline products, such as calcium silicate hydrate (C-S-H) phases, which reduce porosity and increase matrix density. This mechanism is consistent with the increased silicon and calcium content observed in the XRF results, especially at higher ICW dosages.

XRF results confirm that the incorporation of ICW modified the elemental composition, which helps improve the durability of the concrete matrix. The gradual increase in Si and Ca with increased doses of ICW agrees with the expected improvement in the formation of C-S-H phases and other crystalline silica phases within the concrete. These stages are of utmost importance in reducing concrete permeability and achieving an improvement in the resistance to water ingress, thereby enhancing durability and longevity of the concrete structure [38]. As evident, the appearance and gradual increase of the titanium (Ti) content in the ICW-treated samples confirm its role in the densification process of the concrete matrix. Other functional benefits of photocatalytic properties of titanium include self-cleaning and resistance to UV degradation, along with a general improvement in durability within treated concrete [5, 9]. General trends show the reduction of Al and Fe content with increased dosage of ICW indicate the likely transformation or encapsulation of such elements in more stable, less reactive phases. The decrease of iron might reveal a reduced risk of reinforcement corrosion, being one of the critical elements of concrete durability [13, 15, 18].

Several works confirm these results. Among them, in the work of Tampali et al. [40], crystalline admixtures interact with tricalcium silicate (C_3S) and water to form modified C-S-H and pore-blocking precipitates, significantly increasing the impermeability of concrete. Al-Rashid and Al-Jabari [45] also determined that hydrophilic crystalline admixtures based on silicates increase the rate of pore blocking, which is crucial in increasing the service life of concrete. A similar mechanism was observed in Hu et al. [42], where crystalline admixtures were identified to refine pores and promote the self-healing of cracks. The results of forming a densified, high-silicon healing product agree with our findings, in which the increase in silicon content with increasing ICW dosages points to better crystallization and enhancement within the matrix. Elsalamawy et al. [38] reiterated that Ca/Si ratios modify C-S-H morphologies. They found that increasing Ca/Si ratios provide denser C-S-H structures, supporting the stable Ca/Si ratios in our study. It was found that this Ca/Si ratio remained quite stable with respect to the increase in the content of calcium and silicon within the range of 1.02 to 1.03 for all dosages of ICW. Works like those by de Souza Oliveira et al. [36] and Elsalamawy et al. [38] underlined the importance of this ratio in the establishing the morphology and strength of the C-S-H phases. It implies stability in the course of our work, meaning balanced formation of dense crystalline structures without disruption of the fundamental chemical composition, which leads to optimal mechanical performance. Various synthesized C-S-H phases with Ca/Si ratios between 1.0 and 2.0 have indicated that denser C-S-H, corresponding to a higher Ca/Si ratio, is associated with lower permeability and higher durability of the concrete [26]. This trend of Ca/Si ratio with C-S-H morphology corroborates our observation that the increase in calcium and silicon content of the ICW-treated concretes, which was according to a constant ratio, resulted in less permeable and more durable concretes.

3.4. Energy Dispersive X-ray Spectroscopy (EDS)

EDS analysis provided an overall view of changes in elemental composition of the concrete specimens treated with different dosages of the ICW admixture. As shown in Table 3, for some elements, the increase in concentration varied linearly with the dosage of ICW. A consistent trend of increased silicon dioxide (SiO_2) and calcium oxide (CaO) was observed, indicating a significant shift in the composition of hydration products with higher ICW dosages. This correlates with the formation of more calcium silicate hydrate (C-S-H), a critical component in concrete's strength and water impermeability.

The key components for the control specimen, which was 0 kg/m² ICW dosage, were CO_2 at 16.35%, Na_2O at 0.35%, MgO at 0.77%, Al_2O_3 at 5.39%, SiO_2 at 34.9%, SO_3 at 1.04%, K_2O at 4%, CaO at 35.47%, and FeO at 1.73%. With the increased dosage of ICW, these elemental compositions changed significantly. The SiO_2 content increased progressively from 34.9% for the control to 39.96% at the highest dosage of ICW, which was 4.8 kg/m², indicating an increased amount of silicon compounds in the matrix. Similarly, the increase in the percentage of CaO surged from 35.47% in the control to 41.6% at 4.8 kg/m², which indicating a higher concentration of calcium compounds, crucial for crystalline formation in the concrete matrix. The presence of titanium dioxide (TiO_2) was noticed only in treated ICW samples, starting from 3.1% at 0.8 kg/m² and progressively increasing to 3.9% at 4.8 kg/m². This reflects the presence of compounds in ICW contributing to denser crystalline structures. Other elements also varied significantly. For example, there was a considerable decline in CO_2 content from 16.35% for the control to 10.15% at 4.8 kg/m². This indicates reduced carbonation potential in the specimens treated with ICW. A decrease in CO_2 suggests that the ICW admixture reduce the permeability of concrete to CO_2, thereby reducing carbonation risk. The Al_2O_3 content also showed a downward trend, from 5.39% for the control sample down to 0.65% for the highest dosage, which could mean that the aluminum compounds were reacting or integrating into new crystalline phases.

Table 3. The EDS analysis in the different dosage of ICW

Compound (%)	ICW dosage (kg/m²)						
	0.0	**0.8**	**1.6**	**2.4**	**3.2**	**4.0**	**4.8**
CO_2	16.35	13.81	12.46	10.78	12.00	10.67	10.15
Na_2O	0.35	0.43	0.78	0.24	0.17	0.22	0.27
MgO	0.77	0.76	0.73	0.83	0.68	0.74	0.88
Al_2O_3	5.39	1.56	1.37	1.33	0.88	1.05	0.65
SiO_2	34.90	37.02	37.54	38.2	39.59	39.66	39.96
SO_3	1.04	1.34	1.24	1.46	1.42	1.52	1.18
K_2O	4.00	1.18	1.25	1.25	1.26	1.34	1.41
CaO	35.47	38.00	38.86	39.4	40.60	41.00	41.6
FeO	1.73	2.80	2.57	1.88	0.00	0.20	0.00
TiO_2	0.00	3.10	3.20	3.30	3.40	3.60	3.90

The increase in SiO_2 from 34.9% in the control to 39.96% at the maximum treatment of 4.8 kg/m² indicates that the ICW admixture provides an improved silicon environment that favored the nucleation of additional phases of C-S-H. This nucleation aligns with the findings of Lin et al. [39] who observed that crystalline admixtures promote the nucleation and development of C-S-H gel, improving concrete density and mechanical properties. Similarly, the increase in CaO content from 35.47% to 41.6% suggests enhanced hydration and supports findings from Zhang et al. [52], who noted that higher calcium content leads to the development of denser crystalline structures, enhanced mechanical properties, and crack-healing performance over time. Furthermore, the presence of TiO_2 in the spectra of the ICW-treated samples, without appearing in the control sample, implies the inclusion of new compounds that increase the density and impermeability of the matrix. This is consistent with the findings from de Souza Olivera et al. [36], who found that the addition of crystalline additives fosters the formation of stable impermeable matrices. Additionally, TiO_2, due to its photocatalytic and densifying effect, can assist in self-cleaning and improve durability over the long term by preventing chemical degradation in the presence of moisture or environmental pollutants [5, 9].

A fundamental characteristic of this study is the reductio n of CO_2 content—from 16.35 to 10.15%—which demonstrates the powerful effect of the ICW admixture in inhibiting CO_2 penetration, reducing carbonation potential, and increasing internal pH, all of which are necessary for protecting steel reinforcement. Several studies [39-40] suggest that this reduction is likely due to the increased precipitation of $CaCO_3$ inside the cracks—one of the main healing products of the concretes modified with ICW. Wu et al. [49] posit that the main self-healing mechanism is the precipitation of $CaCO_3$ on crack surfaces, which helps to block pore and increase impermeability. The decrease in aluminum oxide content from 5.39% to 0.65% suggests that aluminum compounds were consumed during the precipitation of secondary crystalline structures or within the densified matrix. This reduction could be due to the formation of AFt crystals, as identified by Hu et al. [42] in ICW-treated concretes. The presence of these ettringites within the microstructure contributes to matrix densification, reducing porosity and improving resistance to water penetration.

Additionally, the presence of magnesium compounds was recorded in hydration products and in ICW-treated concretes. According to Tsampali et al. [40], the incorporation of magnesium in C-S-H gels can alter their nature and potentially give rise to a magnesium silicate hydrate (M-S-H), which can significantly enhance concrete durability. The role of magnesium in the formation of M-S-H phases appears promising for improving the impermeability of ICW-treated concretes. One peculiar observation in this study is the introduction of titanium compounds through the ICW admixture, especially the increased concentration with higher ICW dosages. Departing from earlier research, this study postulates that TiO_2 compounds may chemically interact with $Ca(OH)_2$ during hydration to form stable and impermeable crystalline phases, in addition to densifying the matrix. This novel finding is in agreement with Songkhla et al. [53], who identified the contribution of titanium compounds to early strength gains and long-term durability in concrete.

3.5. Crystal Gel Formation

Crystallization in concrete usually occurs within confined spaces, such as pores and microcracks, where crystallization pressure develops against the pore walls. Crystallization pressure, which in most cases depends on factors such as crystal-liquid interfacial energy and the geometry of the pore space, may cause microcracks due to stresses during crystallization [21]. As shown in Figure 11, a crystal gel formed beneath the bottom surface of concrete specimens treated with the highest dosage of ICW (4.8 kg/m^2), whereas this phenomenon was absent in those treated with lower and middle dosages. The gel appeared as a semi-dry white crystalline mass, translucent in color, with sizes ranging from 2 to 4 mm. Upon exposure to air, the gel transitioned from dry to semi-dry sticky, demonstrating its agglomerative nature under certain conditions. This is likely due to the higher concentration of the ICW reacting with stored moisture on the top surface of the concrete for an extended period, resulting in the migration and subsequent precipitation of crystalline compounds into the lower areas.

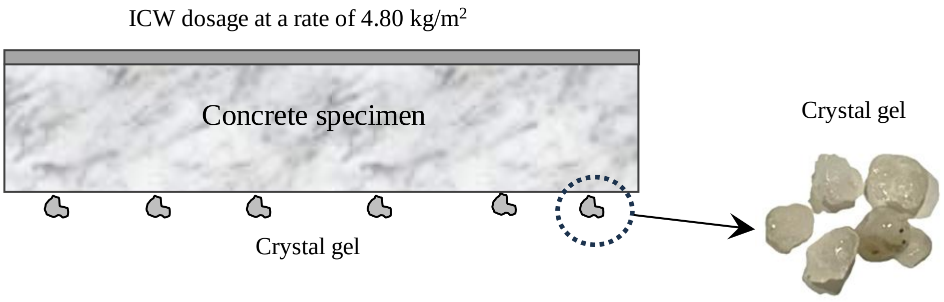

Figure 11. Crystal gel formation at the bottom surface of specimen

This migration of unreacted crystalline admixture, precipitating in a gel form on lower surfaces, is similar to findings from salt crystallization studies. Increased ion concentrations have been found to induce crystallization into pores in those studies, which might be responsible for efflorescence and subflorescence, and microcracking [22]. Table 4 and Figure 12 present the EDS results, proving the gel to be of a different composition than the bulk concrete matrix. The highest concentrations measured were for Si and Ti, at 67.54% and 6.15%, respectively, while K measured 11.00%. Ca and Fe were almost negligible. In other words, the composition is mainly silica-rich and potassium-rich phases that, though responsible for surface characteristics, do not contribute much to the mechanical properties of the concrete. Results in this regard are in agreement with other works concerning the action of nanoparticles, such as nano-SiO_2 [33], whose high doses modified the matrix without adding more resistance. Besides, the absence of calcium hydroxide in the gel composition indicates a wrong silica-to-calcium ratio, which led to the formation of a non-structural gel phase that would barely develop strength or durability. The presence of TiO_2 at 6.60% in the evolution may relate to the formation of titanium-rich phases, which can alter further the optical and surface properties without mechanical advantage, as noted by Faisal and Patra [66]. These results align with previous research indicating that crystalline admixtures in concrete can promote crystal growth within the matrix, effectively filling pores and cracks [33, 34]. However, this study underscores a critical downside of excessive ICW dosage. While optimal dosages enhance concrete's self-healing abilities by promoting crystallization in the pores [39-43], this gel did not form in specimens treated with lower dosages, suggesting that the excess ICW admixture likely migrated due to gravity, settling in the lower regions and forming non-structural, silica-rich gels.

Table 4. The EDS quantitative calculation of the crystal gel

Element (%)	Crystal gel	Compound (%)	Crystal gel
C	10.59	CO_2	10.51
Na	0.31	Na_2O	16.37
Mg	0.43	MgO	0.00
Al	3.70	Al_2O_3	0.57
Si	67.54	SiO_2	54.46
S	0.23	SO_3	0.81
K	11.00	K_2O	10.68
Ca	0.00	CaO	0.00
Fe	0.00	FeO	0.00
Ti	6.15	TiO_2	6.60

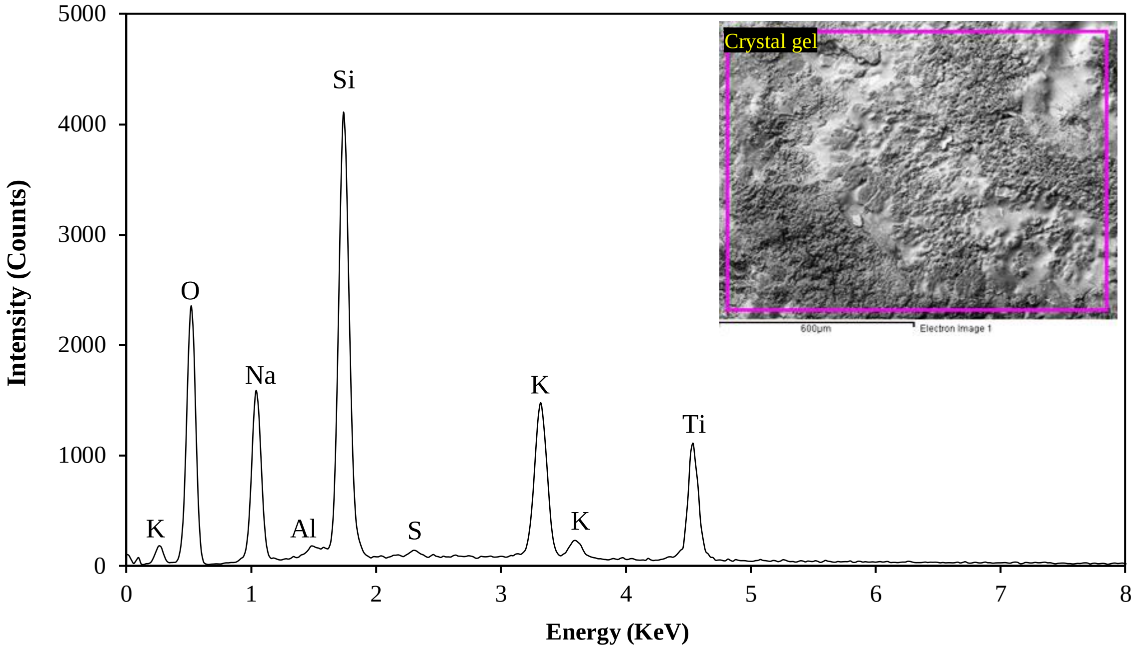

Figure 12. The EDS result of the crystal gel

Using various solvents, such as hexane, and deionized water in performing solubility tests, it was found that the gel was insoluble in both hexane and ethyl acetate because there was no change in the pH of these respective solvents. However, when tested in deionized water, as represented in Figure 13, the crystal gel dissolved, increasing the pH of the water from 6 and 12. The increase in pH indicates the release of alkaline components from the gel, possibly calcium hydroxide or other basic compounds, thereby reinforcing the hypothesis that the gel was composed of crystalline hydration products formed by the ICW admixture in the presence of water [34].

Figure 13. Crystal gel was tested in deionized water

Further analysis by Fourier Transform Infrared Spectroscopy (FTIR) showed that this crystal gel shared functional groups with most acrylic acid polymers, which are generally applied in waterproofing admixtures and acrylic-based coatings, as depicted in Figure 14. These findings suggest that excessive ICW use leads to undesirable side effects, where the crystalline compounds fail to integrate effectively into the matrix, forming a gel with limited structural contribution. The FTIR spectra of the crystal gel and acrylic acid polymer show similarities in transmittance patterns across various wave numbers. The crystal gel formed by the concrete matrix appears similar to acrylic acid polymers and therefore provides evidence that the formation of the crystal gel structure has involved acrylic-based compounds. However, transmittance values are slightly higher for the crystal gel compared to acrylic acid polymer, showing that there is a combination of water molecules and organic materials. In the fingerprint region, which is the region below 1500 cm^{-1}, the transmittance values of the crystal gel are different from those of the acrylic acid polymer. The peaks in the region around 1400 cm^{-1} down to 900 cm^{-1} show the presence of complex inorganic compounds, probably silicate-based or calcium-silicate hydrates C-S-H, which are not seen in acrylic acid polymers [39-40, 42]. These lower transmittance values at 1300 cm^{-1} and below, especially around the region of 1000 cm^{-1}, are related to the stretching vibrations of Si-O and Ti-O, which are indicative of silicate and titanium compounds within the crystal gel. These results are in agreement with the obtained XRF and EDS, where high contents of SiO_2 and TiO_2 were recorded, confirming that the crystal gel is indeed a complicated combination of silicates and probably modified polymers or organic compounds [40].

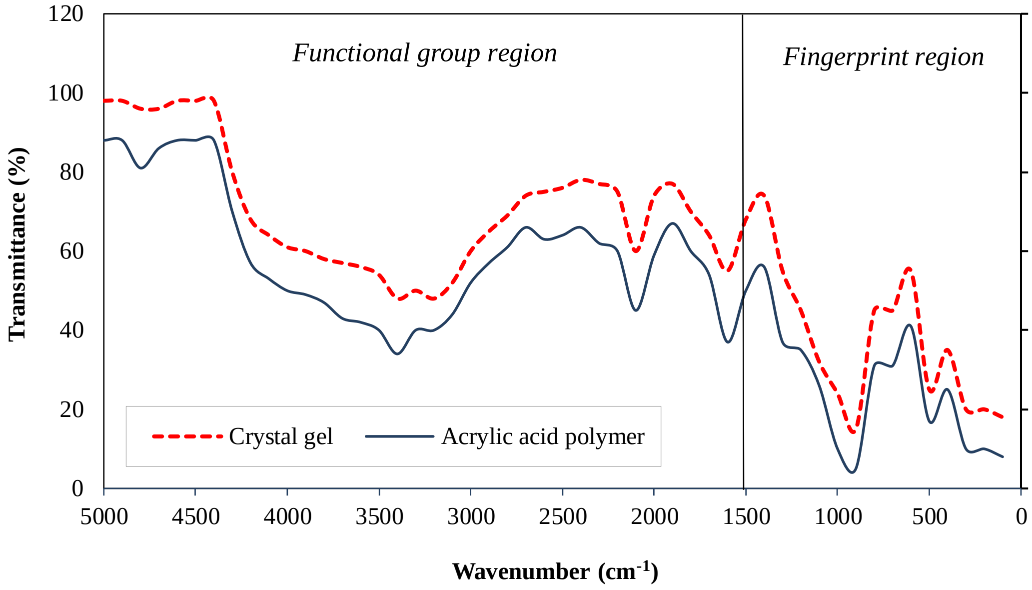

Figure 14. FTIR spectrum between crystal gel and acrylic acid polymer

Some previous studies have emphasized the critical problem of using correct crystalline admixture dosages to avoid undesired crystallization effects. Liu et al. [37] demonstrated that crystalline admixtures could accelerate cement hydration, producing more hydration products, thereby improving concrete compactness. However, this only occurs when admixture are applied at an optimum dosage rate, while higher dosages, as used in this study, can be inefficient in crystallization. Excess admixtures were also reported by Elsamawy et al. [38] to form larger crystals that block the pores too aggressively or fail to provide good bonding with the matrix. Solubility tests support the hypothesis that the crystal gel formed is further composed of hydration products sensitive to water. This was evidenced by the gel dissolving in the deionized water and the pH skyrocketing, indicating the release of alkaline components. Zhang et al. [52] have also reported similar effects, pointing out the gels disturb the chemical environment within concrete, which could result in potential negative changes in permeability and long-term durability. In the present work, the formation of crystal gel is directly related to the high dosage of ICW and the time of exposure to moisture, while the latter drives silicate and calcium-rich compounds to migrate and crystallize [67]. With such a peculiar chemical composition and properties, this gel is likely to engage in complex interactions with the ICW materials and the paste matrix when interacting with the environment. Further research is needed regarding the implications for concrete durability and performance.

4. Discussion

4.1. Mechanism of Action

This might be explained by their peculiar mode of action, since ICW admixtures indeed are very effective in improving the performance of concrete. The application of the ICW admixture—usually in dry powder form—leads to its penetration into the fresh concrete matrix. When the concrete starts setting and hydrating, the ICW admixture reacts with calcium hydroxide—a by-product of cement hydration—and water in the concrete [42]. This will consequently yield insoluble crystalline material in the network of pores in the concrete [45]. The crystals grow and expand further to fill in the capillary pores and micro-cracks, effectively blocking pathways through which water or other aggressive agents could potentially pass [34, 36, 38, 43].

This crystalline growth does not adversely affect the breathability of the concrete so that water vapour can still pass through, which helps eliminate internal moisture build-up that can lead to problems, including freeze-thaw damage [2, 20, 42, 44]. However, it is the liquid form of water—which could cause corrosion of reinforcement or other forms of degradation—that is effectively barred [3, 23]. This is also supported by the XRF and EDS analyses, which reveal increased silicon and calcium content with increasing dosage of ICW, corresponding to the formation of more substantial amounts of C-S-H and other silicate phases with less solubility and greater resistance to water penetration. At the same time, there was a reduction in the concentration of reactive elements such as Al and Fe; it was assumed that the elements became incorporated into more stable and less reactive forms due to crystalline networks being formed. This stabilization decreases the risk of deleterious chemical reactions that may weaken the concrete structure over time. In the case of further treated samples examined with ICW, the small increment in the content of titanium (Ti) suggested that other protective phases may form for the enhanced durability of concretes, which may also be resistant to UV radiation and chemical attacks [9].

4.2. Influence of ICW Dosage

From the results, it is quite evident that three integral components of concrete—water absorption, structural integrity, and chemical composition—can be heavily influenced by different dosages of ICW admixture. With an increasing dosage, the ICW-treated samples showed a considerable reduction in their water absorption rates. The control sample, with no treatment of ICW, had a 6.33% water absorption rate, which decreased progressively down to 3.60% at 4.8 kg/m², the highest dosage of ICW. This reduction in water absorption, which fell to as low as 57% of the control value, clearly indicates that higher doses of ICW are more effective in reducing the permeability of concrete. Indeed, it can be attributed to the fact that higher-dosed ICW forms a denser crystalline network inside the concrete matrix that tends to block most of the capillary pores and pathways through which water could easily penetrate.

A similar trend was observed in pulse velocity. The control specimen recorded a pulse velocity of 3548 m/s, and this was significantly improved with higher dosages, recording as high as 5012 m/s at 4.8 kg/m². The pulse velocity increased with dosage in a way that indicates the concrete becomes more homogeneous, denser, and characterized by few internal voids and micro-cracks as the dosages of ICW increase. This densification process enhances the structural integrity of concrete, improves its performance under load, and makes it more resistant to such environmental degrading factors as freeze-thaw cycles or chemical attack [33, 43-44]. In addition, changes in the elemental composition due to the use of different dosages of ICW are demonstrated by XRF and EDS analyses. With the increase in the dosages of ICW, the contents of Ca and Si increased, although other potentially reactive elements such as Al and Fe showed lower concentrations. This trend, in fact, confirmed that with higher dosages of ICW, more stable and durable compounds like C-S-H would be formed that are of prime importance to the strength and impermeability of concrete [36, 38-39].

4.3. ICW Optimum Dosage

In this study, concrete performance and durability under different dosages of ICW were discussed. The results indicated that ICW optimization of dosage is extremely critical for cost efficiency, material performances, and achieving the desired waterproofing effect [39, 42, 56]. At the optimal dosage—from about 2.4 to 4.0 kg/m²—there was a marked improvement in the waterproofing capabilities of the concrete specimens without adverse side effects. Such a dosage range seems to be the window where crystalline formation inside the concrete matrix is substantial enough to reduce permeability and improve durability, without excessive material cost increase or other negative side effects [33-34, 36-37, 39]. It is further supported by XRF and EDS analyses, since in all those optimal dosages, concentrations of key elements such as silicon dioxide (SiO_2) and calcium oxide (CaO) were in a state of relatively good balance. This balance is very important in building an effective crystalline network within the concrete for better water resistance while still maintaining the required structure of the concrete [42, 45]. Furthermore, at those levels, there was no evidence of any unwanted negative phenomena such as reduction in mechanical properties and/or formation of unwanted compounds, and hence the characteristics required from the concrete mix were retained.

4.4. Effects of High ICW Dosage

Clearly, when the dosage of ICW increases to its maximum value, 4.8 kg/m², an abrupt change in the behavior of concrete occurs. This dosage showed crystal gel masses on the bottom surface of the concrete specimens. These gel masses had a white, semi-transparent, soft, easily deformed appearances ranging from 2 to 4 mm in size. Overdosing of ICW may result in crystalline gel formation where reactive compounds like SiO_2 and CaO become supersaturated and interfere with the normal hydration process, leading to unreacted compounds. These gels tend to form weak zones in the concrete matrix, probably inducing micro-cracking, increased porosity, and loss of durability over time [33]. For practical applications, such effects may result in decreased environmental resistance and poor long-term performance, making careful dosage control warranted to avoid the risk of this happening. Over-application of ICW increases material costs and other costs for mitigating side effects. Dosage optimization supports goals for performance and economic efficiency [33, 39, 42, 56]. In addition, the crystal gel precipitate at the bottom of the specimens shows evidence of some kind of phase separation in the concrete caused by excessive use of ICW, yielding an inferior material overall. This could result in non-uniform distribution of waterproofing characteristics and early deterioration when exposed prematurely to actual service conditions, especially in basements, water tanks, and marine environments.

5. Conclusions

This research work examines the impact of different dosage levels of ICW admixture on various concrete properties and performance. From the experimental results obtained, the dosages of ICW significantly affect water absorption, pulse velocity, and microstructural characteristics of concrete. The findings obtained are summarized below:

- The ICW admixture effectively reduces water absorption in concrete. In the case of an ICW dosage at 4.8 kg/m², water absorption was drastically decreased by 43% compared with the control specimen. This result shows that the incorporation of the ICW admixture enhances the impermeability of concrete by crystallization within pores and micro-cracks, blocking the water pathways.
- Pulse velocity increases with an increase in the dosage of ICW. It shows a maximum improvement of 41% at 4.8 kg/m². This indicates a denser and more compact concrete matrix because of crystalline growth that promotes ICW, filling the voids and reducing porosity.
- XRF and EDS analyses revealed major changes in the chemical composition of the concrete as a function of the dosage of ICW. In particular, higher dosages increased the amount of silica (SiO_2) and potassium oxide (K_2O) while the calcium oxide content, CaO, also increased, indicating further crystalline phase formation. However, an excessive dosage promoted crystalline gel formation at the bottom of the specimen which was rich in silica and scarce in calcium. These observations clearly indicate that, although ICW is beneficial at an optimum dosage, excess dosage of ICW facilitates the formation of crystals not related to the structure at all and, therefore, provides no strength to concrete but poses a potential threat to its durability.
- The improvement brought by the ICW admixture generally comes from crystalline structures formed inside the concrete matrix, which act as barriers to water ingress and increase density. The study identifies that ICW dosages between 2.4 kg/m² and 4.0 kg/m² offer the right balance between reduced water absorption and improved structural integrity of concrete, without undesired reactions such as the formation of crystalline gels that do not contribute to the structural strength. On the other hand, with higher dosages, the unreacted components migrate and settle, yielding crystalline formations that may compromise the concrete's structural integrity.

6. Declarations

6.1. Author Contributions

Conceptualization, W.N., C.C., and G.S.; methodology, S.J., C.C., and G.S.; validation, S.J., C.C., and G.S.; formal analysis, S.J., W.N., and G.S.; investigation, W.N. and G.S.; resources, W.N. and C.C.; data curation, S.J. and C.C.; writing—original draft preparation, W.N. and G.S.; writing—review and editing, S.J., C.C., and G.S.; supervision, W.N.; project administration, G.S.; funding acquisition, C.C. All authors have read and agreed to the published version of the manuscript.

6.2. Data Availability Statement

The data presented in this study are available in the article.

6.3. Funding and Acknowledgement

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6.4. Conflicts of Interest

The authors declare no conflict of interest.

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