

Article

The Effect of Combining Waste-Derived Pozzolanic and Fibrous Materials with Functional Admixtures on Performance and Corrosion Resistance of Concrete

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Abstract

This study investigates the combined effect of waste-based metakaolin, cellulose fibres and functional waterproofing additive on the physical, mechanical, and durability-related properties of sustainable concrete. A total of 12 concrete mixtures were produced, varying in cellulose fibre content (0–2%), metakaolin waste replacement levels (up to 10% of binder), and functional waterproofing additive content (1%). The experimental program assessed workability, density, compressive and flexural strength, ultrasonic pulse velocity (UPV) and alkali–silica reaction (ASR) resistance. The presence of metakaolin due to high pozzolanic activity (1451 mg/g) and fine particle size enhance the formation of additional C–S–H phases. The incorporation of cellulose fibres (1–2 mm in length) improved crack-bridging ability and structural integrity, while functional waterproofing additive enhanced water tightness. Results demonstrated that the synergistic use of these materials led to improved mechanical performance (flexural strength varies from 4.87 MPa to 6.81 MPa; compressive strength varies from 24.01 MPa to 32.97 MPa) and enhanced notable ASR resistance (decrease in expansion varies from 0.209% to 0.029%). The findings highlight the potential of combining waste-derived pozzolanic and fibrous materials with functional admixtures to develop environmentally friendly and performance-optimized concrete composites.

Keywords: cellulose fiber; metakaolin waste; functional waterproofing additive; ASR resistance; sustainable concrete



Academic Editor: Salvatore Verre

Received: 16 January 2026

Revised: 9 February 2026

Accepted: 10 February 2026

Published: 13 February 2026

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1. Introduction

Concrete is one of the most important building materials, but its performance is still limited by its tendency to crack, moisture penetration, and progressive degradation over time. With the construction sector constantly raising the bar for resistance and sustainability, scientists are increasingly researching how alternative raw materials and chemical additives could improve the properties of cementitious composites while reducing their environmental impact. In recent years, particular attention has been paid to different kinds of fibres, industrial by-products, and modifying admixtures which not only offer functional advantages for new materials but also reduce the carbon footprint.

Cement modified with industrial waste, such as activated coal gangue, significantly reduces carbon emissions while maintaining structural integrity through fibre-reinforced

matrix synergy. This sustainable framework suggests the potential for integrating natural waste materials, such as cellulose fibres, as partial cementitious replacements to further enhance de-carbonization [1].

One promising direction is the use of cellulose fibres, especially those obtained from recycled paper. These fibres are lightweight, renewable, and capable of “bridging” microcracks in the early stages of concrete hardening. Their rough surface and favourable length-to-diameter ratio help to inhibit crack propagation and improve the behaviour of concrete after cracking—the same effects are observed when using other natural fibres, such as banana or vetiver [2–4]. Although cellulose fibres are shorter and more elastic than most plant fibres, their uniform distribution and compatibility with concrete matrices make them suitable for improving the structural integrity of sustainable concrete.

Another important component being studied in modern cementitious composites is metakaolin-based waste. Although it is an industrial by-product (in this case, formed during the production of foam glass granules), it can exhibit a high pozzolanic reaction. Previous studies have shown that metakaolin significantly contributes to the formation of secondary C–S–H phases, increases strength, and improves durability [5,6]. Thus, metakaolin obtained from waste provides a double benefit: it improves the properties of concrete and reduces cement consumption, which remains the main source of CO₂ emissions in concrete production.

The third group of materials, functional waterproofing additive, is particularly relevant for structures exposed to moisture and aggressive chemical environments. These materials react with unhydrated cement particles and moisture in the matrix to form insoluble crystals that fill capillaries and reduce water penetration. Numerous studies confirm their effectiveness in increasing water impermeability and long-term concrete durability [7,8]. Some authors also note that the formation and activation of crystals are strongly influenced by curing conditions [9,10], so their effectiveness may vary depending on the composition of the mixture and environmental factors.

Although each of these materials—cellulose fibre, metakaolin waste, and functional waterproofing additive—has been studied extensively individually, there is still a significant lack of knowledge about their interaction within the same concrete matrix. It is unclear whether cellulose fibres influence the ability of crystals to fill pores, how metakaolin waste affects the adhesion between the fibres and the matrix, how functional waterproofing additive improve densifying of concrete structure, and whether the combination of the three components can create a synergistic effect on strength, microstructure, or resistance to alkali–silica reaction (ASR) processes. Only a few studies have analyzed natural fibres in metakaolin-based systems [11,12], and even fewer studies have combined fibres and functional waterproofing additive.

Studies show that cellulose fibres in cementitious compositions lose their properties over time due to the effects of an alkaline corrosion environment. Although they effectively eliminate microcracks, the degradation of hemicellulose and lignin over time reduces their reinforcing effect [13].

Industrial waste such as metakaolin can improve the mechanical and physical properties: due to its high pozzolanic activity, it promotes the formation of additional C–S–H phases, reduces the amount of portlandite, and densifies the pore structure, improving strength and durability.

Functional waterproofing additives are another way to reduce water absorbability. They form insoluble crystals in pores and microcracks, which helps concrete maintain its impermeability even after repeated wetting and drying cycles [14]. It has also been found that advanced crystallization systems can reduce thermal stresses and improve resistance to freeze–thaw cycles [15], and their effect on moisture and heat transport depends on the

composition of the mixture and the curing conditions [16]. SEM studies under alkaline conditions confirm significant changes in the microstructure [17], but there is little data on mixtures with fibres and functional waterproofing additive.

The combined effect of these materials—fibres, metakaolin waste, and functional waterproofing additive—in a single concrete matrix has not yet been thoroughly researched. It is clarified that hydration products intensively generate on the fibre's surface when functional waterproofing additive is used in compositions to improve the flexural and compressive strength of concrete. Overall, the combined effect of these materials—cellulose fibres, metakaolin waste, and functional waterproofing additive—improves ASR resistance of the concrete matrix. Scientific research shows that 1% cellulose fibre, together with 8% metakaolin waste and 1% functional waterproofing additive, is recommended for use especially in cases of increased danger of alkaline corrosion conditions.

This study aims to fill this knowledge gap by investigating the combined effect of cellulose fibre, metakaolin waste, and functional waterproofing additive on the mechanical properties, durability, and microstructural development of sustainable concrete. By combining renewable fibres, reactive industrial waste, and a functional waterproofing additive, the aim of this study is to determine whether a balanced combination of these materials can result in stronger and more environmentally friendly ASR-resistant concrete compositions.

2. Materials and Methods

2.1. Materials

The experimental program employed CEM II A-LL 42.5N Portland limestone cement (AB Akmenės cementas, Naujoji Akmenė, Lithuania), which conforms to the requirements of LST EN 197-1:2011 [18]. Supplementary materials included metakaolin waste (MW) and a functional waterproofing additive (CA). Concrete specimens were produced using Sika WT-200P (Sika AG, Baar, Switzerland), a grey, powder-form functional waterproofing additive. A high-performance superplasticizer (in liquid form), MC-PowerFlow 3100 (MC-Bauchemie Müller, Bottrop, Germany), produced using the latest MC polymer technology, was used. Water with a pH of 7.8 was used for preparing the specimens.

Fine aggregate consisted of washed sand of the 0/4 fraction, complying with the requirements of standard LST EN 12620:2003+A1:2008 [19]. The mineralogical and petrographic composition of the quarry sand used is presented in Table 1.

Table 1. Mineral and petrographic composition of sand from used quarry.

	Quartz	Carbonates	Feldspars	Mineral Aggregates	Amphiboles	Mica
Declared/limit value	62.80%	18.00%	15.60%	1.80%	1.60%	0.20%

The metakaolin waste used in this study was generated as a by-product of the foam glass pellet manufacturing process and has shown promise as a pozzolanic material. Previous studies have demonstrated that such metakaolin waste can increase the mechanical strength and reduce the drying shrinkage of cementitious composites due to its high reactivity and fine particle size [20].

The particle size distribution of the three binder components—Portland cement (PC), metakaolin waste (MW), and functional waterproofing additive (CA)—is presented in Figure 1. The measurements were conducted over a size range of 0.10 to 500 µm, and the mean particle sizes are as follows: PC—14.21 µm; MW—4.79 µm; CA—73.19 µm.

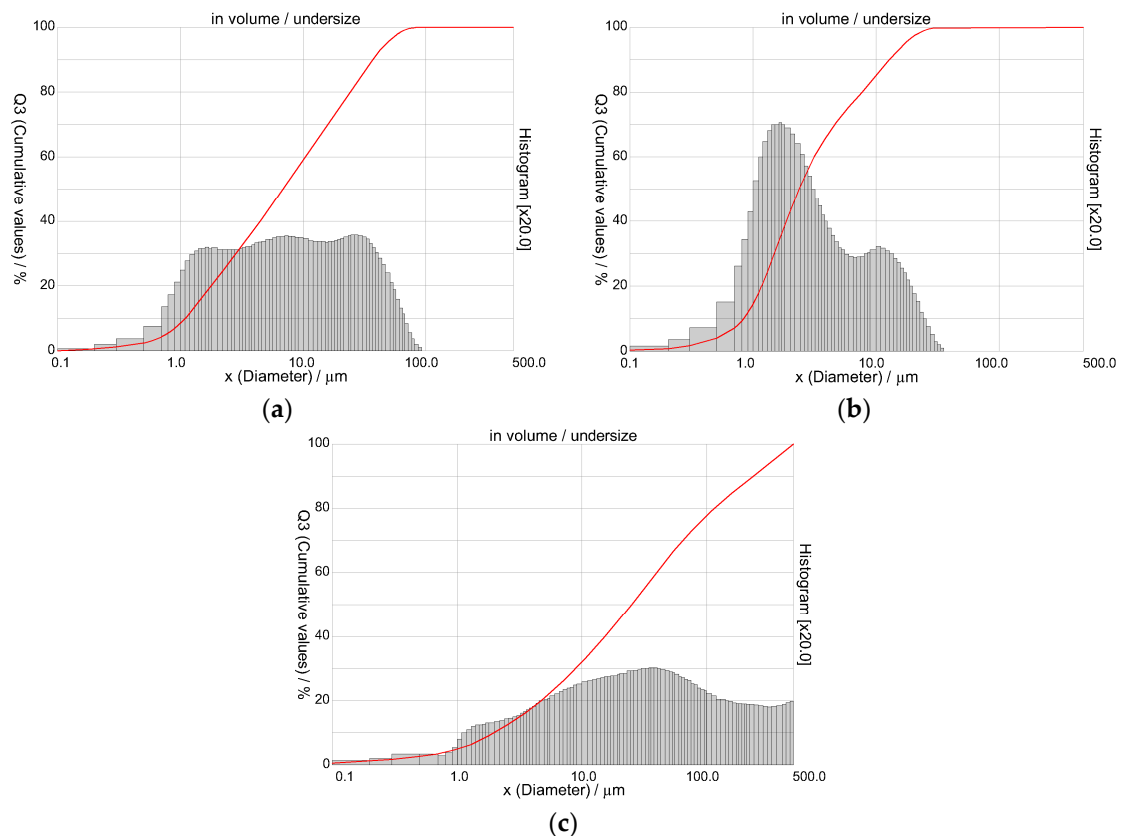


Figure 1. Particle size distribution of (a) Portland cement, (b) metakaolin waste, (c) functional waterproofing additive.

As seen in Table 2, the metakaolin waste exhibited the highest specific surface area ($7043 \text{ cm}^2/\text{g}$), which is more than 80% higher than that of cement, confirming its high fineness and thus its potential for rapid reactivity [21,22]. In contrast, the functional waterproofing additive had the lowest particle density and largest average particle size, indicating a fundamentally different functional role in the composite matrix.

Table 2. Cement, metakaolin waste, and functional waterproofing additive main properties.

Properties	PC	MW	CA
Specific surface area, cm^2/g	3819	7043	3407
Particle density, kg/m^3	3130	2700	2390
Bulk density, kg/m^3	1210	3802	3406

Figure 2 presents microstructural images of cellulose fibres obtained from recycled paper, highlighting their morphological characteristics at different magnifications.

Morphological analysis of the cellulose fibres revealed that their average length ranged from 1.0 to 1.1 mm, indicating relatively short fibre geometry suitable for uniform dispersion in the cementitious matrix. The average diameter of the fibres was found to be between 0.045 and 0.05 mm, corresponding to a high aspect ratio, which may contribute positively to crack-bridging and mechanical performance enhancement in concrete [23,24].

The chemical composition of the materials is provided in Table 3. The metakaolin waste contains 50.4% SiO_2 and 20.9% Al_2O_3 , indicating a strong pozzolanic potential [25,26]. Its low CaO content (5.54%) further supports its classification as a silico-aluminous pozzolanic material rather than a hydraulic one. On the other hand, the functional waterproofing additive is rich in CaO (49.59%) and contains a total alkali oxide content ($\text{K}_2\text{O} + \text{Na}_2\text{O}$) of 5.6%,

which is consistent with materials designed to form insoluble crystalline phases that block pores and enhance the water-tightness of concrete [27,28]. However, its limited reactive silica and alumina content suggests that its pozzolanic activity is negligible or absent.

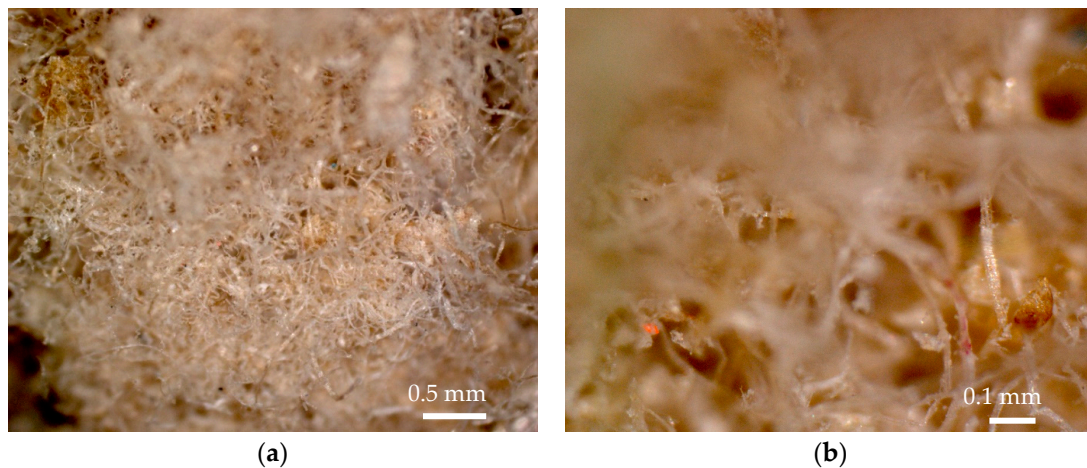


Figure 2. Cellulose fibres obtained from recycled paper, shown in micro scale: (a) $\times 30$ magnification, (b) $\times 112.5$ magnification.

Table 3. Chemical composition of Portland cement, metakaolin waste and functional waterproofing additive.

Chemical Composition of Portland Cement, Mass %										
SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	K ₂ O	SO ₃	Na ₂ O	P ₂ O ₅	MgO	TiO ₂	Cl
14.8	3.47	2.61	61.6	1.12	4.56	0.097	0.09	3.37	0.249	0.04
Chemical composition of Metakaolin waste, mass %										
50.4	20.9	0.61	5.54	0.51	0.12	11.2	0.08	2.22	0.33	0.02
Chemical composition of Functional waterproofing additive, mass %										
5.74	0.93	0.68	49.59	0.22	1.06	5.38	0.13	0.97	0.08	-

2.2. Mix Design

A total of 12 batches of concrete mixtures were prepared to investigate the influence of cellulose fibres, metakaolin waste, and a functional waterproofing additive on the properties of cementitious composites. The compositions of the mixtures are presented in Table 4. The mixing procedure was as follows: dry components—cement, sand, metakaolin waste, and, where applicable, functional waterproofing additive and cellulose fibres—were mixed for 5 min. Subsequently, water and a chemical admixture (0.4%) were added, followed by an additional 5 min mixing phase. This method of preparing the mixture ensures even distribution of dry powders and fillers before hydration begins. Scientific literature sources also refer to this technique when forming concrete specimens [29,30]. The water-to-cement ratio (W/C) was maintained at 0.3 for all batches. The batches were grouped according to the variable additive contents.

Table 4. Concrete compositions, kg/m³.

Batches	I	II	III	IV	V
Cement, kg	346.8			319.52	
Sand, kg			1793		
Cellulose fibre, kg	0	31.5	21	31.5	42
Metakaolin waste, kg	0			27.28	
Chemical admixture, kg			5.21		
W/C			0.3		

In Batches I to V, the cellulose fibre content ranged from 0% to 2% by weight of the total dry materials (cement, sand, and metakaolin, where applicable), specifically 0%, 1%, 1.5%, and 2%, respectively. The water, sand, and total binder content (cement + metakaolin) remained constant in all batches. In Batches III–V, 8% of the cement was replaced by metakaolin waste (by weight of binder). A chemical admixture was added at a constant dosage of 0.4%, and the water-to-cement (W/C) ratio was kept at 0.3 across all mixes.

Batches VI to IX in Table 5 were formulated to investigate the potential synergistic effect between cellulose fibres and metakaolin waste on the properties of cementitious composites. In these mixtures, the cellulose fibre content was varied from 0% to 2% by weight of total dry materials (cement, sand, and metakaolin where applicable), specifically 0%, 1%, 1.5%, and 2% in batches VI, VII, VIII, and IX, respectively. A constant 8% of cement (by mass of the binder) was replaced with metakaolin waste in batches VII to IX. The functional waterproofing additive was excluded from these formulations to isolate the influence of fibres and metakaolin. A chemical admixture was incorporated at a fixed dosage of 0.4%, and the water-to-cement (W/C) ratio was maintained at 0.3 across all compositions. The water and sand contents remained constant throughout.

Table 5. Concrete compositions, kg/m³.

Batches	VI	VII	VIII	IX
Cement, kg	346.8		319.52	
Sand, kg			1793	
Cellulose fibre, kg	0	21	31.5	42
Metakaolin waste, kg	0		27.28	
Functional waterproofing additive, kg			3.39	
Chemical admixture, kg			5.21	
W/C			0.3	

Batches X to XII in Table 6, referred to as the red group, were designed to evaluate the combined effect of cellulose fibres, metakaolin waste, and a functional waterproofing additive on the properties of cementitious composites. These were the only formulations that incorporated all three types of supplementary materials simultaneously. Each mix contained a constant 10% replacement of cement with metakaolin waste (by binder mass) and 1% of a functional waterproofing additive (by weight of total dry materials). The cellulose fibre content varied across the mixes at 1%, 1.5%, and 2% by weight of total dry materials in batches X, XI, and XII, respectively. The water, sand, and total binder contents were held constant across the group. A chemical admixture was added at a uniform dosage of 0.4%, and the water-to-cement (W/C) ratio was maintained at 0.3 in all mixes.

Table 6. Concrete compositions, kg/m³.

Batches	X	XI	XII
Cement, kg		312.12	
Sand, kg		1793	
Cellulose fibre, kg	21	31.5	42
Metakaolin waste, kg		34.1	
Functional waterproofing additive, kg		3.39	
Chemical admixture, kg		5.21	
W/C		0.3	

The experimental design enables the evaluation of the impact of cellulose fibres alone, the interaction between fibres and metakaolin, and the combined effect of fibres, metakaolin, and functional waterproofing additive. All other parameters, including aggregate content, water content, and chemical admixture dosage, were kept constant to ensure comparable test conditions.

2.3. Methods

The structural and chemical characterization of the samples was carried out using X-ray fluorescence (XRF) and X-ray diffraction (XRD) techniques. The chemical composition metakaolin waste was analyzed using a Bruker S8 TIGER wavelength-dispersive X-ray fluorescence (WD-XRF) spectrometer (Bruker, Mannheim, Germany). The instrument was equipped with a Rh-target X-ray tube, operated at an anode voltage of up to 60 kV and a current of up to 130 mA. Sample preparation involved pressing the powders into pellets, which were subsequently analyzed in a helium atmosphere using the SPECTRA Plus QUANT EXPRESS method.

Structural analysis was conducted using X-ray diffraction (XRD), employing a Smart Lab diffractometer (Rigaku, Tokyo, Japan) fitted with a rotating Cu anode X-ray source rated at 9 kW. XRD scans were acquired over a 2θ range of 5° to 75° , using the Bragg–Brentano geometry. During data collection, the sample stage rotated at a speed of 0.02° per minute, while the detector scanned at 1° per minute. Phase identification was carried out using PDXL software, Version 1.8.0.3 (Rigaku), referencing the ICDD Powder Diffraction File (PDF+), 2021 edition.

Particle size distribution was determined using a Cilas 1090 LD laser diffraction particle size analyzer (Cilas, Orléans, France) capable of measuring particle sizes in the range of $0.01\ \mu\text{m}$ to $500\ \mu\text{m}$, with air as the dispersing medium. All laser particle size analyzers have been designed with lasers that provide the highest resolution, accuracy, and precision across the entire range.

Microstructural imaging was performed using a Helios Nanolab 650 dual-beam scanning electron microscope (SEM) (FEI Company, Hillsboro, OR, USA) equipped with focused ion beam (FIB) technology. SEM observations were conducted in secondary electron imaging mode, at an accelerating voltage of 15 kV and an electron beam current of 0.1 nA. Compared to an optical microscope, a scanning electron microscope allows users not only to distinguish much smaller surface structure formations (magnification up to 100,000 times) but also to obtain significantly better contrast due to the large depth of field (up to $30\ \mu\text{m}$).

Six samples of the same mixture were used to determine each indicator.

Ultrasonic pulse velocity (UPV) measurements were conducted in accordance with LST EN 12504-4:2021 [31]. This non-destructive testing method enables the assessment of concrete by measuring the propagation speed of ultrasonic waves, which reflects its

internal quality, homogeneity, and structural integrity. As such, UPV serves as a critical tool for both quality assurance and long-term condition monitoring of concrete structures. The ultrasonic pulse velocity (UPV) was calculated according to Formula (1):

$$UPV = \frac{l}{\tau'}, (\text{m/s}) \quad (1)$$

where l is the length of the sample and τ' is the signal propagation time, s .

Concrete density was determined following LST EN 12390-7:2019 [32], while compressive strength and flexural strength were evaluated according to LST EN 12390-3:2019 [33] and LST EN 12390-5:2019 [34], respectively.

The alkali–silica reactivity (ASR) of concrete specimens was evaluated based on expansion testing following the RILEM AAR-2 methodology. According to this procedure, hardened mortar specimens (dimensions: $40 \times 40 \times 160$ mm) are initially cured in water at 80°C for 24 h and subsequently immersed in a 1 M NaOH solution at 80°C . Expansion is measured over time to assess the potential reactivity of aggregates.

3. Results and Discussion

3.1. Parameters of Tested Materials

X-ray diffraction (XRD) analysis was conducted on four materials in Figure 3: hydrated cement, cellulose fibre, metakaolin waste, and a functional waterproofing additive.

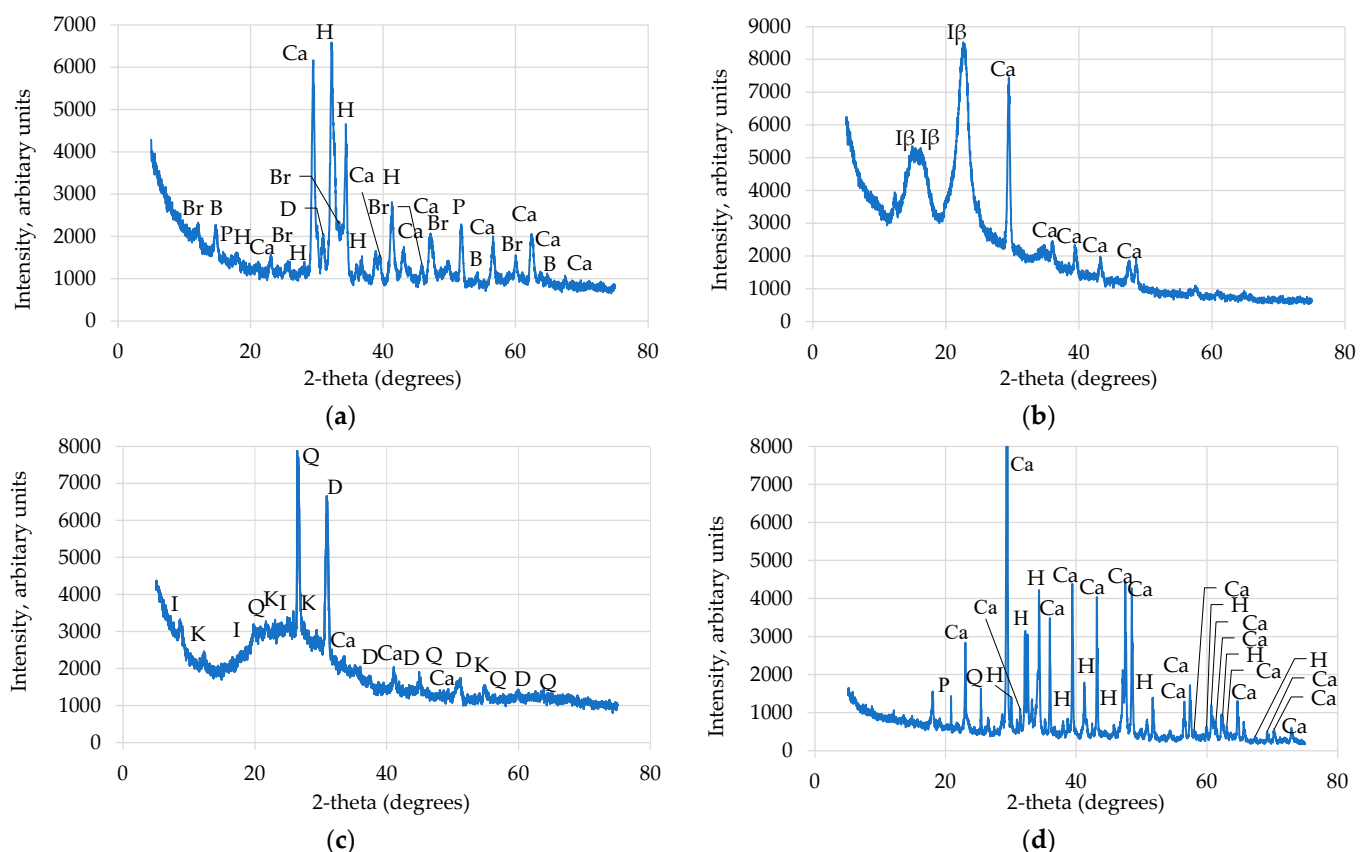


Figure 3. X-ray diagram of (a) cement; (b) cellulose fibre; (c) metakaolin waste; (d) functional waterproofing additive. Br—brownmillerite; B—bassanite; P—portlandite; H—CSH (hatrurite); D—dolomite; Ca—calcite; Iβ—cellulose; Q—quartz; K—kaolinite; I—illite.

The hydrated cement sample exhibited a complex crystalline profile dominated by calcium silicate hydrate (CSH, 60%), which is a principal hydration product, accompanied

by significant amounts of brownmillerite (15.3%), calcite (13%), bassanite (6.2%), and portlandite (4.2%). A minor presence of dolomite (1%) was also observed. This mineral assemblage reflects a mature hydration process in a Portland cement system after 28 days of curing.

The cellulose sample, on the other hand, was characterized by sharp and intense peaks attributed to crystalline cellulose I β (96.2%), with a small fraction of calcite (3.8%), indicating high structural order and purity with only minimal inorganic impurities.

The metakaolin waste sample revealed a notably different mineral composition, consisting primarily of dolomite (34.5%), quartz (23%), and calcite (23%). Residual kaolinite (6.3%) and illite (11%) indicated that the thermal treatment did not completely dehydroxylate all clay components, suggesting partial transformation and limited pozzolanic reactivity. Metakaolin waste still contains a considerable amount of amorphous phase, which is typical for thermally activated aluminosilicates. This amorphous phase significantly contributes to the material's reactivity, providing additional sites for pozzolanic reactions.

The functional waterproofing additive displayed broad and distinct peaks associated with CSH, confirming its role as a hydration-promoting agent [35]. Minor signals of calcite and quartz were also present, likely originating from filler materials or by-products of early hydration. Overall, the XRD results highlight the distinct mineralogical fingerprints of each component and their potential contributions to composite cementitious systems in terms of hydration behaviour and structural performance.

Scanning electron microscopy (SEM) analysis was performed to evaluate the morphological characteristics of the dry components used in the cementitious mixtures, as shown in Figure 4. The SEM image of Portland cement (Figure 4a) revealed angular and irregularly shaped particles of varying sizes, which is characteristic of ground clinker and limestone constituents. These features are known to influence the packing density and initial hydration kinetics of the binder system. The cellulose fibres (Figure 4b), obtained from recycled paper, displayed elongated, ribbon-like structures with a rough surface texture, which may promote mechanical interlocking with the cement matrix and improve crack-bridging efficiency. The metakaolin waste (Figure 4c) shows a highly porous, finely textured morphology, composed of angular particles and a granular microstructure. Similar SEM image results showing an irregular and rough surface of metakaolin waste particles have also been obtained by other researchers [36]. This morphology, along with its high specific surface area, supports its classification as a reactive pozzolanic material capable of contributing to secondary hydration product formation. In contrast, the functional waterproofing additive (Figure 4d) demonstrated a coarser and more compact granular structure with distinguishable crystalline features. These characteristics suggest its role is primarily physical, contributing to pore blocking and water-tightness enhancement rather than participating in pozzolanic reactions.

The observed morphological differences confirm the distinct functional contributions of each material: Portland cement as the primary hydraulic binder, metakaolin waste as a chemically reactive additive [37], cellulose fibres as physical reinforcement [38], and the functional waterproofing additive as a densifying component improving durability-related properties [39].

Metakaolin waste demonstrated a high pozzolanic activity of 1451 mg/g, indicating its strong potential as a supplementary cementitious material [40]. Its elevated SiO₂ content enables an active reaction with Ca(OH)₂ in the cementitious matrix, leading to the formation of additional calcium silicate hydrate (C–S–H) and other hydration products. These compounds contribute to pore refinement, increased density, and enhanced mechanical and durability-related properties of sustainable concrete. Therefore, metakaolin waste can

be effectively utilized to improve both the performance and environmental footprint of cement-based composites [41].

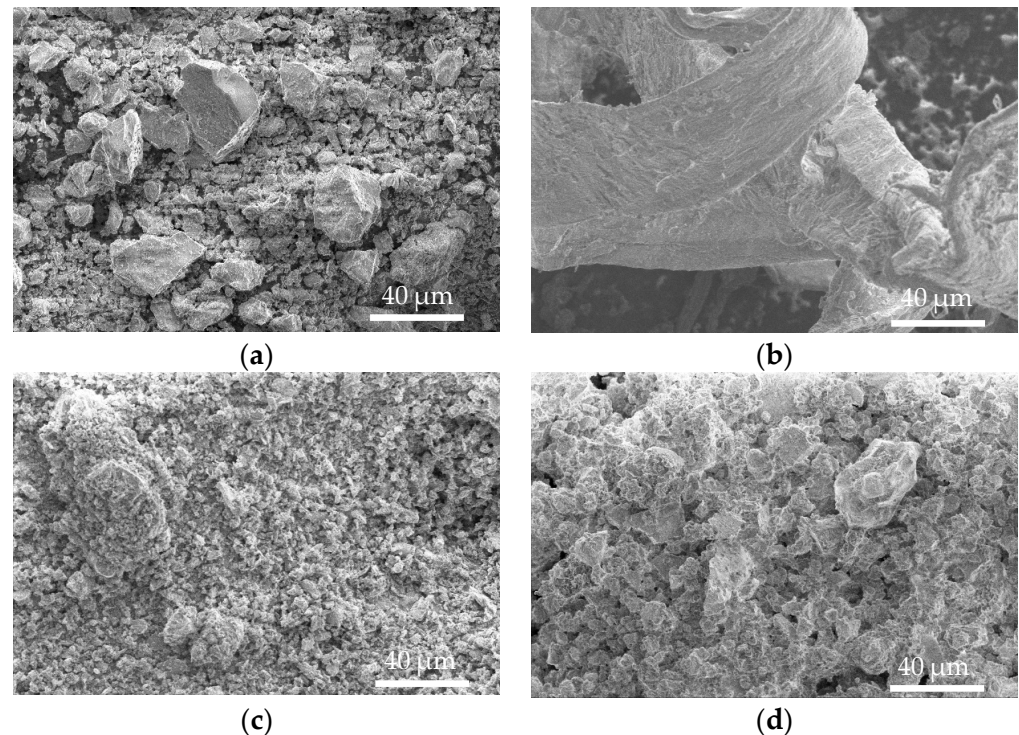


Figure 4. Microstructure 1000 $\times$ magnification of (a) cement; (b) cellulose fibre, (c) metakaolin waste; (d) functional waterproofing additive.

3.2. Density and UPV

Figure 5 shows density and ultrasonic pulse velocity graphs of concrete modified with different materials before and after corrosion.

The results in Figure 5 show that both density and ultrasonic pulse velocity slightly decrease after corrosion in all mixtures, but samples with cellulose fibre, metakaolin, and functional waterproofing additive retain higher values, indicating improved durability compared to the reference concrete. The highlighted values indicate the best-performing result within the entire group of mixtures. These values were pointed out to show which mixture achieved the most suitable density and ultrasonic pulse velocity in the comparison.

The decreased density of concrete specimens after alkaline corrosion indicates internal expansion and the appearance of cracks and voids [42]. Metakaolin waste helps reduce the risk of ASR: it can react with alkalis, reducing the amount available to react with silica and helping to prevent expansion and cracking [43,44]. The functional waterproofing additive promotes the formation of insoluble crystals such as calcite that fill cracks and can appear after alkali corrosion, restore the matrix and increase density and UPV (since it is directly dependent on density) of the concrete [45].

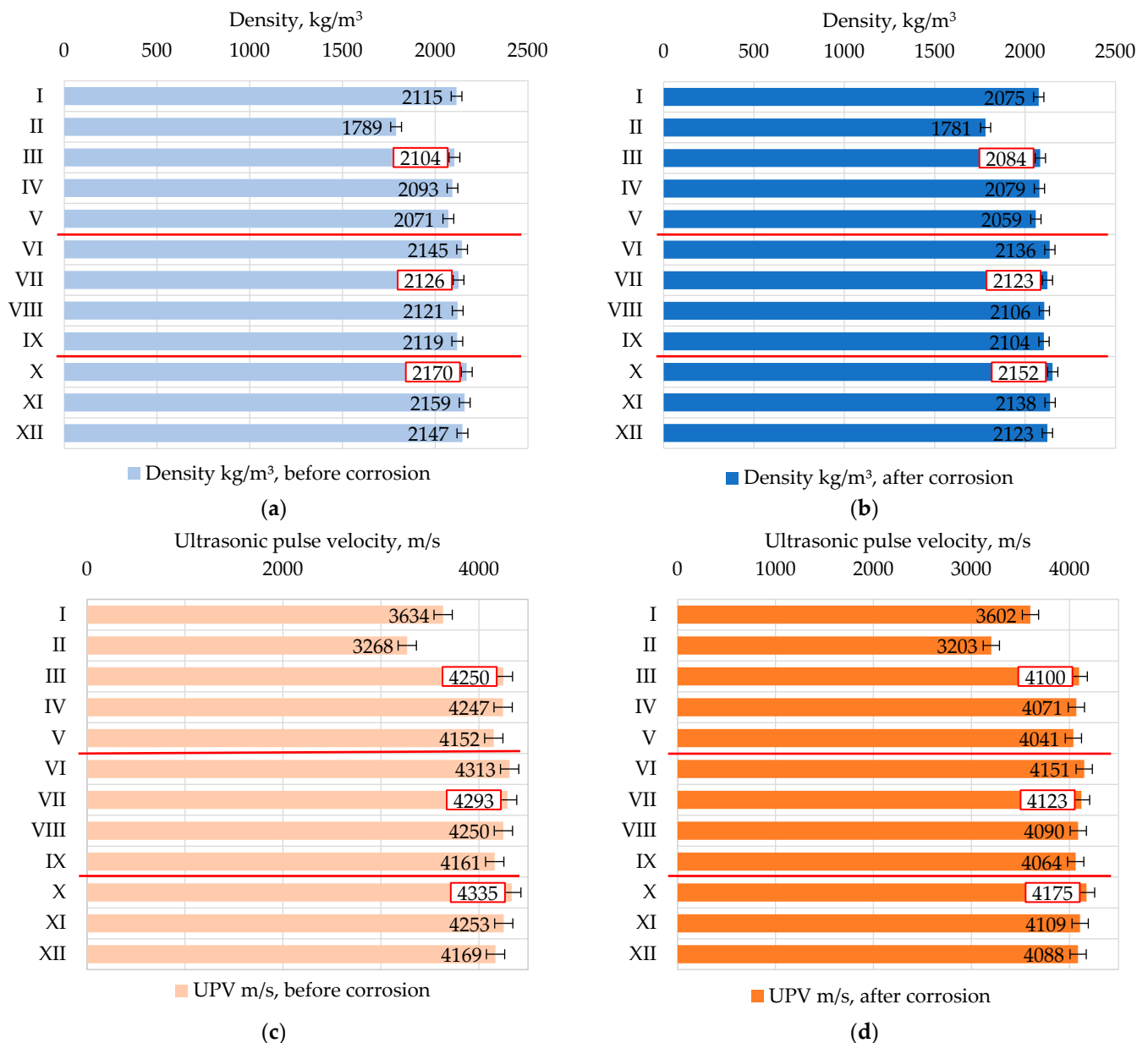


Figure 5. Density and ultrasonic pulse velocity of the hardened modified concrete before corrosion (a,c) and after corrosion (b,d). Composition sets are separated by red lines.

3.3. Flexural and Compressive Strengths

Figure 6 shows graphs with flexural and compressive strength values of concrete modified with different materials before and after corrosion. Graphs in Figure 6 show that concrete with 1% cellulose fibre, 8% metakaolin waste and 1% functional waterproofing additive has 20% increased flexural strength and 2.3% increased compressive strength compared to sample I without any additives. The highlighted values mark the mixtures that achieved the highest flexural and compressive strength within the entire group.

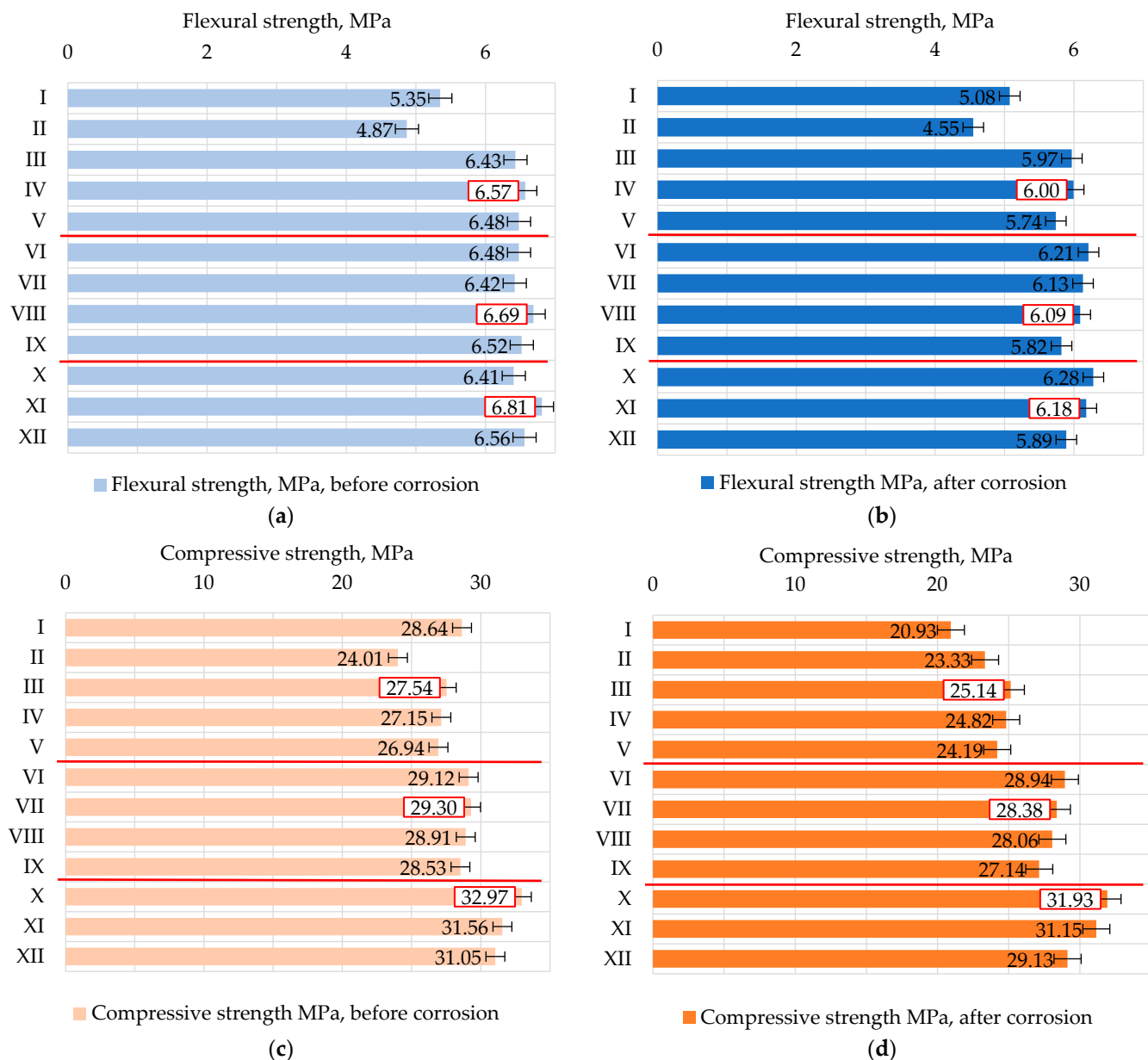


Figure 6. Flexural and compressive strength of the hardened modified concrete before corrosion (a,c) and after corrosion (b,d). Composition sets are separated by red lines.

In similar studies where concrete was modified with metakaolin waste, tests in alkaline conditions showed clear improvements: ASR was reduced because metakaolin waste lowers the alkalinity of the matrix and suppresses alkali–silica reaction, preventing expansion and cracking that would reduce strength [46]. Cellulose fibre acts as mechanical reinforcement by controlling crack propagation, but it is vulnerable to alkaline environments [47], so additives like metakaolin waste and a functional waterproofing additive are used to prevent its degradation and improve both flexural and compressive strength. However, in our case, the cellulose fibres have an additional role. The uneven surface of the cellulose fibres keeps adsorbed water and is suitable for additional hydration products precipitation. The fibre, because of its hydrophilicity, absorbs water and releases it gradually in surrounding area. This water can later participate in cement hydration reactions and create a strong bond between the fibres and cement matrix.

3.4. SEM Analysis

Figure 7—SEM images of I concrete specimens without admixtures before and after corrosion. Figure 7a shows the structure of the hardened concrete surface at a magnification of $\times 2000$, and Figure 7b shows the concrete surface at a magnification of $\times 5000$ before corrosion. Figure 7c and d show the microstructure of concrete sample I after corrosion, with a rougher surface and ASR gel formation.

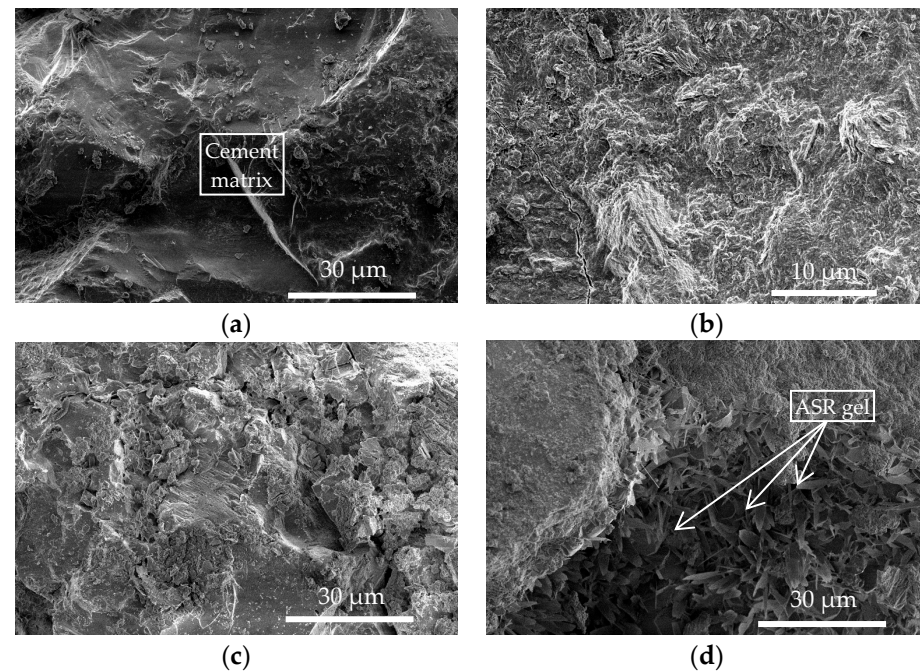


Figure 7. Sample of hardened concrete of the I composition microstructure before corrosion: (a) magnification $\times 2000$; (b) magnification $\times 5000$. After corrosion: (c) magnification $\times 2000$; (d) magnification $\times 2000$.

In the cement matrix, ASR gel forms in the damaged areas after alkali-induced degradation [48]. The ASR occurs between the alkali-affected cement paste and amorphous silica, which is found in significant amounts in the fine aggregate, such as sand.

Figure 8 shows SEM images of concrete specimens of the composition VII with admixtures before corrosion. The selected composition demonstrated the most advantageous mechanical and physical properties, combined with an optimized composition and appropriate rheological characteristics. Figure 8a–e shows cellulose fibre (CF) covered with hydration products; similar results were observed in another studies, when SEM images showed how cellulose fibres are covered with hydration products [49,50]. Figure 8f shows ettringite crystals covered with calcium silicate hydrate (C–S–H) gel.

SEM images show that concrete containing cellulose fibre, metakaolin waste, and functional waterproofing additive develops a very dense structure. This forms during the hydration process, as the dry cellulose fibre absorbs water and simultaneously catches a large amount of other hydration products, resulting in a strong bond between the fibre and matrix and a compact overall concrete structure.

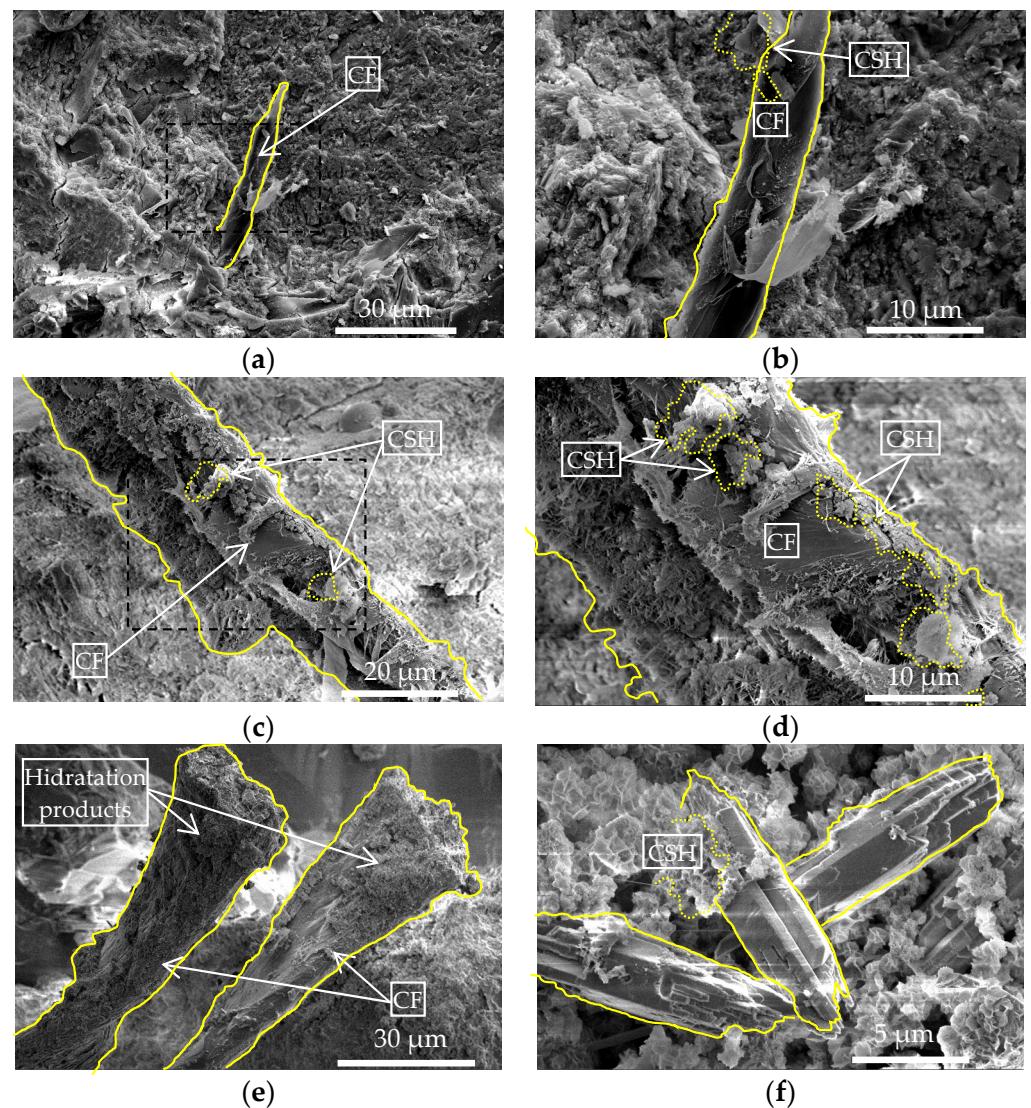


Figure 8. Hardened concrete sample with 1% cellulose fibre, 8% metakaolin waste and 1% functional waterproofing additive microstructure before corrosion: (a) magnification $\times 3500$; (b) magnification $\times 10,000$; (c) magnification $\times 5000$; (d) magnification $\times 10,000$; (e) magnification $\times 2000$; (f) magnification $\times 10,000$.

Figure 9 shows SEM images of concrete specimens with admixtures after corrosion. Figure 9a,b shows cellulose fibre (CF) covered with different hydration products, while Figure 9c,d shows cellulose fibre (CF) heavily covered with CSH. Cellulose fibre with well-crystallized ettringite needles (AFt) arranged in different directions after corrosion is visible in Figure 9e,f. These AFt needles were also detected using SEM analysis in a similar study investigating the impact of high-alkali biofuel fly ash on concrete [51].

SEM observations after alkali corrosion testing indicate that the cellulose fibres remain structurally unaffected and contribute to maintaining the overall dense structure of the concrete. Concrete mixtures containing cellulose fibres, metakaolin waste, and functional waterproofing additive exhibit only minor microstructural changes following alkaline exposure. The images show slight cellulose fibre shedding, yet the cellulose fibres remain present with attached hydration products.

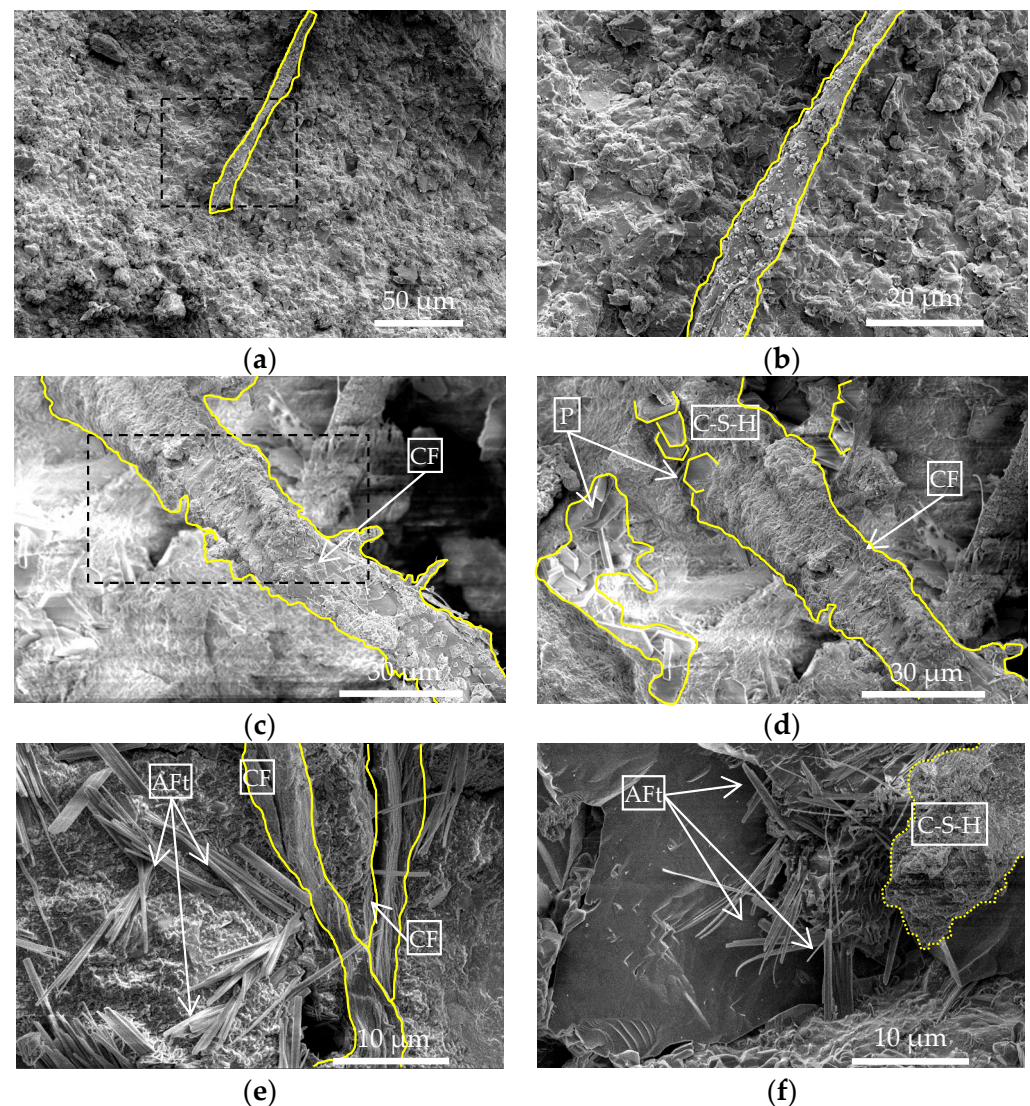


Figure 9. Hardened concrete sample with 1% cellulose fibre, 8% metakaolin waste and 1% functional waterproofing additive SEM images after corrosion: (a) magnification $\times 1500$; (b) magnification $\times 5000$; (c) magnification $\times 3500$; (d) magnification $\times 3500$; (e) magnification $\times 5000$; (f) magnification $\times 5000$.

3.5. XRD Analysis

The XRD analysis of two different composition samples, I and VII (1% cellulose fibre, 8% metakaolin waste and 1% functional waterproofing additive), before and after corrosion is presented in Figure 10, which shows that quartz remains the dominant crystalline phase in all specimens, and its peak intensity is largely unaffected by the corrosion exposure. More notable changes occur in phases associated with cement hydration and carbonation. In the I specimen, corrosion leads to a visible reduction in the intensity of portlandite, calcite, and ettringite peaks, indicating dissolution of calcium-rich phases and a partial loss of crystalline hydration products.

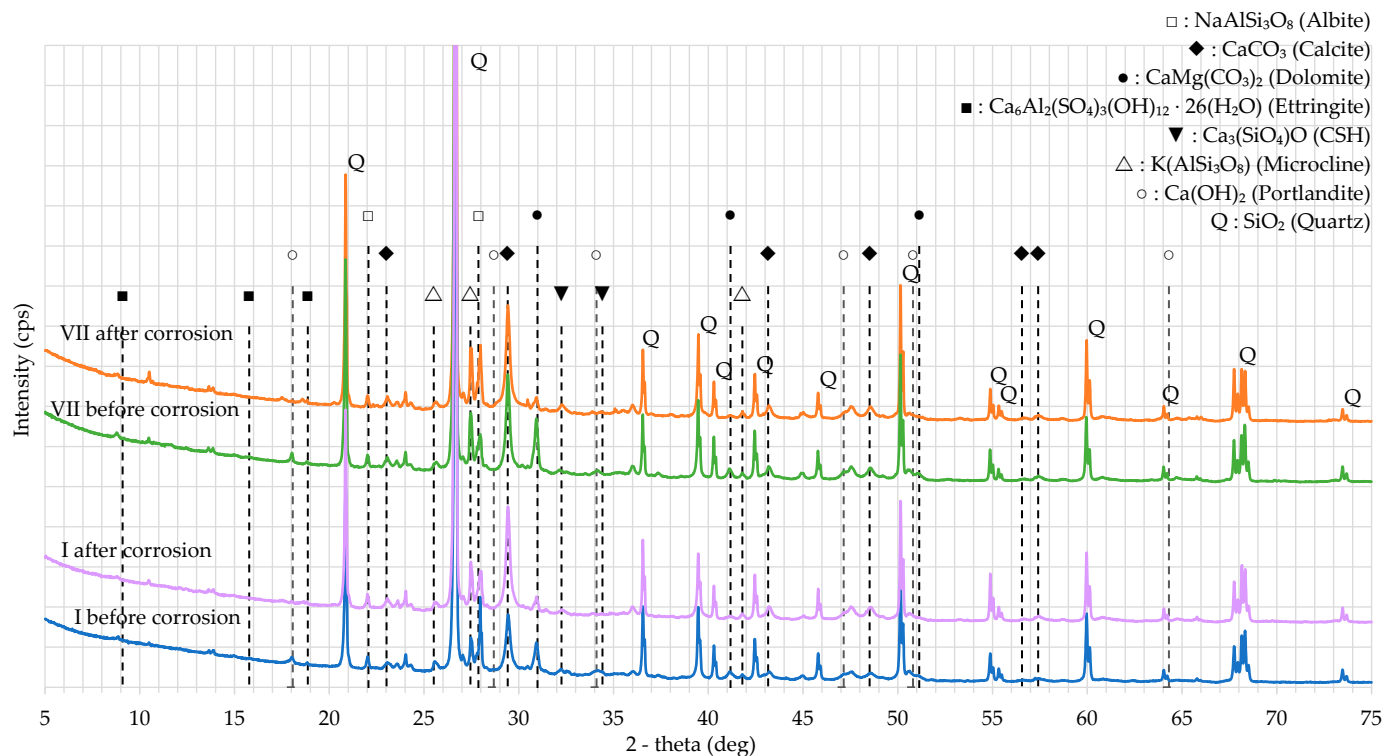


Figure 10. I and VII specimens XRD diffractogram results before and after corrosion.

In contrast, the VII mixture exhibits a more stable phase composition: the differences between its diffractograms before and after corrosion are considerably smaller than in the I sample. Most characteristic peaks remain well-defined, suggesting improved resistance to decalcification and reduced transformation of hydration products. Carbonate phases in the modified mixture also appear less affected, which points to a denser or more chemically stable microstructure.

Overall, the results indicate that the VII formulation provides better mineralogical stability under corrosive conditions compared to the unmodified I mixture.

3.6. Expansion

The alkali–silica reaction (ASR) expansion tests of concrete specimens were carried out in accordance with the RILEM AAR-2 testing methodology. The essence of the RILEM AAR-2 method is as follows: hardened specimens ($40 \times 40 \times 160$ mm) are immersed in water at 80°C , then stored in a 1 M NaOH (sodium hydroxide) for 14 days, after which their expansion is measured. The methodology states that if the aggregates contain slowly reacting reactive rocks, the storage duration should be longer. Therefore, in this study the storage time in 1 M NaOH solution was extended to 56 days. According to the RILEM AAR-2 method, aggregates are considered reactive when the expansion of concrete specimens after 14 days in 1 M NaOH solution exceeds 0.054%. When the test is continued up to 56 days, the critical expansion threshold is 0.1%, as defined by the ASTM C441 testing method.

Figure 11 presents the ASR expansion test results of hardened modified concrete specimens. After 14 days, the expansion exceeded 0.054% in compositions I, II, III, VI, and VI. After 56 days, several mixes (I, II, and VI) exceeded the 0.1% threshold, indicating increased ASR.

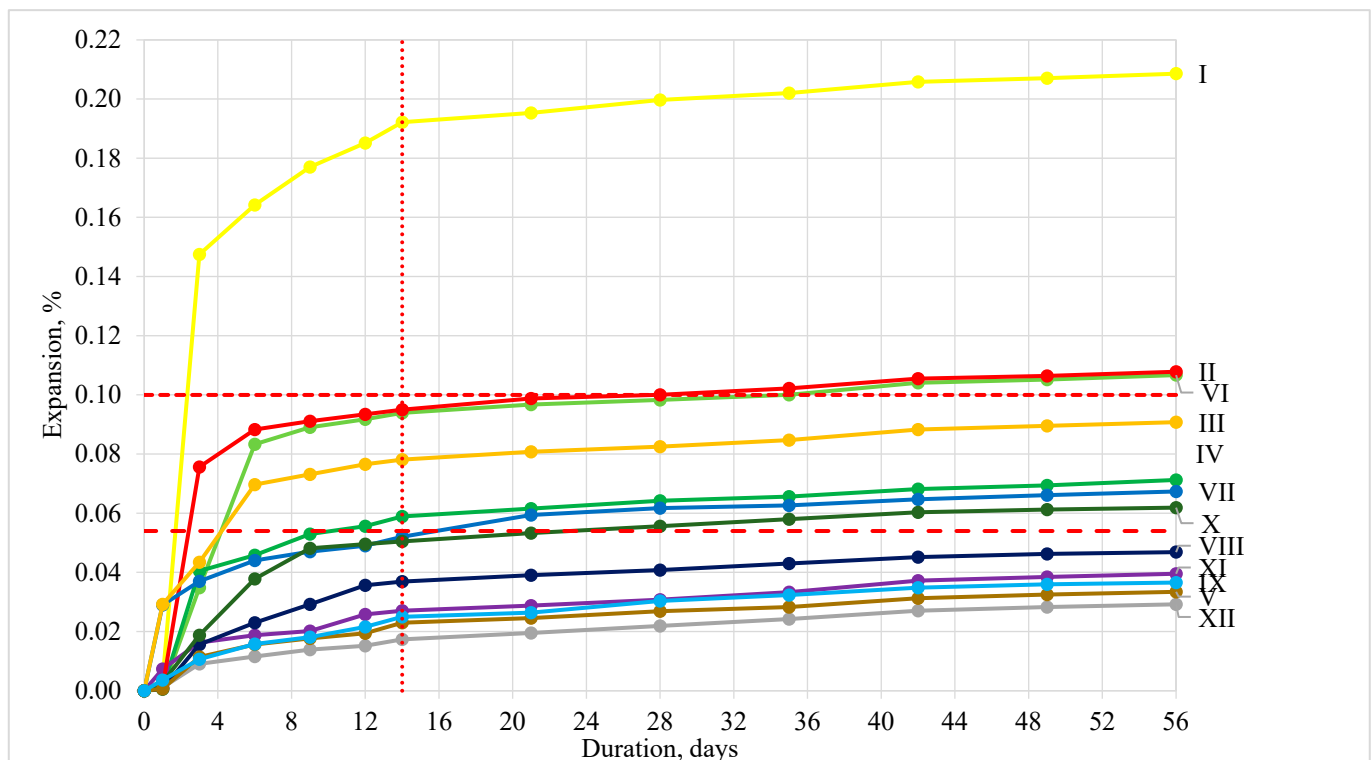


Figure 11. Expansion values of concrete specimens with different admixtures after 56 days, when kept in a 1 M NaOH solution at +80 °C. The red dotted horizontal lines indicate the reference expansion limits (0.054% at 14 days and 0.10% at 56 days). The vertical red dotted line marks the 14-day evaluation point.

Concrete expansion was tested by placing the specimens in a 1 M NaOH solution at 80 °C. Batch VII did not exceed an expansion of 0.054% after 14 days and remained below 0.1% after 56 days; this result can be considered favourable. This positive result may be associated with the combined effect of metakaolin, functional water-proofing additive, and cellulose, promoting a denser microstructure and limiting alkali–silica reaction development.

4. Conclusions

A concrete was developed, modified with 8% and 10% metakaolin waste, 1% cellulose fibre, and 1% functional waterproofing additive. This composition ensured improved structural and performance properties of the mixture, reduced the demand for primary raw materials, and enabled the utilization of secondary raw materials in concrete production.

1. Flexural strength is increased by 23% and 27% in comparison to pure cement specimens when 8% and 10% of metakaolin waste are substituted for cement and cellulose fibres in amounts varying from 1% to 2%. However, adding a 1% functional waterproofing additive also enhances specimen density by up to 2.6%, ultrasonic pulse velocity by up to 19%, and compressive strength by up to 15% due to increased crystallization of hydration products on the surface of cellulose fibre and pore filling. The functional water-proofing additive contains a large amount of CaO, and possibly actively participates in the hydration process by forming additional hydration products, such as CSH.
2. Combined utilizing of metakaolin, functional waterproofing additive and cellulose fibre positively influences concrete microstructure, displaying increased exceptional bonding with the cement matrix. An additional factor that can improve the cement hydration process is the fact that cellulose fibre is hydrophilic. The wet surface

of cellulose fibre is the suitable area on which precipitation of cement hydration products occurs most visibly. As the hydration process continues, the cellulose fibre acts as a source of additional water. Cellulose fibre, because of its hydrophilicity, collects water and releases it gradually into the surrounding region, thus ensuring increased reactions between cement minerals, metakaolin and CaO- rich functional waterproofing additive on the fibre surface.

3. In a 1 M NaOH solution at 80 °C, batch VII's (with minimal 1% of cellulose fibre in composition) expansion stayed below 0.054% after 14 days and did not surpass 0.1% after 56 days, indicating that it performed excellent in the expansion tests. These findings suggest a low susceptibility to the alkali–silica interaction. This improved performance is most likely attributable to the combined use of metakaolin, a functional waterproofing additive, and an optimal amount of cellulose fibres, which led to a denser concrete structure and helped prevent ASR development.

Overall, cellulose fibre is a promising additive for improving the durability and flexural strength of concrete, especially when combined with metakaolin waste and functional waterproofing additive. Optimal fibre content depends on the mix design, with 1–1.5% providing the best balance between mechanical performance and expansion test results.

Author Contributions: Conceptualization, V.V. and D.N.; methodology, V.V., D.N. and I.P.; investigation, V.V., D.N. and I.P.; resources, V.V., D.N. and I.P.; writing—original draft preparation, V.V., D.N. and I.P.; supervision, V.V., D.N. and I.P. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

Conflicts of Interest: The authors declare no conflicts of interest.

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