Performance Analysis of Sulphur Infiltrated-Concrete (SIC): A Novel Approach for Strength Improvement

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ABSTRACT

The growing demand for concrete due to rapid urbanization necessitates large-scale cement production, contributing significantly to CO_2 emissions. As a sustainable alternative, Sulphur-Infiltrated Concrete (SIC) utilizes molten sulphur, an oil and petroleum industry by-product, to fill voids in porous concrete. This study explores the potential of SIC, focusing on its production process, mix design, mechanical performance, and microstructural characterization. Concrete specimens (150×150×150 mm cubes) with varying water-cement (*w/c*) ratios (0.50-0.70) and cement contents (235.71-330 kg/m³) were infiltrated with 99% pure molten sulphur at 140 °C. Experimental trials evaluated compressive strength using destructive and non-destructive methods, including Rebound Hammer and Ultrasonic Pulse Velocity (UPV) tests. Microstructural analysis was conducted using Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD) at 1-day and 7-day curing periods. Durability was assessed through carbonation testing and water absorption studies. The results showed increased early compressive strength, even with reduced cement content, with Trial 3 (*w/c* = 0.70) demonstrating superior performance due to enhanced sulphur penetration. Microscopic analysis revealed reduced porosity and improved bonding between sulphur and cement matrix. Water absorption tests indicated minimal absorption, confirming enhanced impermeability. Cost-benefit analysis indicates long-term economic viability. SIC demonstrates promise as an eco-friendly alternative for precast construction applications, offering dual benefits of waste utilization and reduced cement consumption.

Keywords-carbonation; compressive strength; NDT; SEM; SIC; w/c ratio; XRD

I. INTRODUCTION

Modern infrastructure demands high-performance concrete with enhanced durability, strength, and sustainability to meet the challenges of aggressive environmental conditions and resource depletion [1]. Large-scale waste from oil and gas production plants contains sulphur as a major byproduct, making its effective utilization as a sustainable material essential [2]. Sulphur, a pale-yellow crystalline element, with symbol *S*, atomic number 16, is widely used in agriculture, petroleum, and pharmaceutical industries, and is increasingly valued as a binder due to environmental concerns related to cement production and depletion of raw materials [3]. Early attempts to use sulphur in construction date back to the 1920s when various industrial applications were explored [4]. This led to research on sulphur concrete durability in aggressive environments [5]. Sulphur-based concretes are particularly suited for roads, sidewalks, drainage systems, bridge decks, and acid-resistant structures, due to properties such as high strength, impermeability, rapid hardening, corrosion resistance, and recyclability [6]. Sulphur mortar exhibits superior chemical resistance compared to conventional cement mortar, particularly in acidic environments [7].

SIC is produced by introducing molten sulphur into porous Portland cement concrete, typically with a (*w/c*) ratio of approximately 0.70 [8]. This technique enables sulphur to penetrate the internal pore structure where it solidifies and bonds with the cement matrix [9]. Concrete's internal pore network significantly affects its mechanical performance, but infiltration with sulphur can substantially modify these properties due to its low melting point, low viscosity in the molten state, chemical stability, rapid solidification, and strong adhesion to cement paste [10].

Increased infiltration time enhances sulphur penetration and improves mechanical properties, such as modulus of rupture, with complete infiltration depending on specimen size and process parameters [11]. SIC demonstrates remarkable improvements in early-age strength development and interfacial bonding characteristics, with sulphur infiltration providing immediate structural enhancement even at reduced cement contents. The enhanced mechanical performance is directly attributed to effective void filling and strong adhesion between solidified sulphur and the cement paste matrix [12]. However, chemical analysis reveals that calcium and sulphur can leach from SIC when exposed to alkaline or neutral aqueous environments, forming soluble calcium polysulphides during infiltration that subsequently dissolve during moisture exposure [13].

Despite the promising attributes of SIC, systematic investigation of the relationship between *w/c* and sulphur infiltration efficiency remains limited, particularly regarding early-age strength development with reduced cement content. Most existing studies have focused on standard concrete mixes, with insufficient attention to optimizing the balance between porosity enhancement (through higher *w/c* ratios) and sulphur penetration depth. Furthermore, comprehensive microstructural characterization correlating pore structure modifications with mechanical performance improvements has not been adequately addressed in the literature. The potential for SIC to serve as a dual-benefit solution, both utilizing industrial waste and reducing cement consumption, warrants detailed investigation across varying mix proportions.

This study systematically evaluates SIC performance across three *w/c* ratios of 0.50, 0.60, and 0.70, with corresponding cement contents of 330, 275, and 235.71 kg/m³, respectively. The study focuses on sulphur penetration depth and early-age compressive strength development. Mechanical characterization employs both destructive testing and non-destructive techniques, including Rebound Hammer and UPV at 1-day and 7-day curing periods. Microstructural analysis through SEM and XRD investigates pore structure, hydration products, and sulphur-cement matrix bonding mechanisms. Durability assessment via carbonation testing and water absorption studies establishes the long-term viability and optimal mix proportions for sustainable precast construction applications.

II. MATERIALS AND EXPERIMENTAL PROCEDURE

A. Selection, Collection, and Analysis of Sulphur

Elemental sulphur for SIC requires a minimum 99% purity and moisture content below 0.5%. For infiltration, the present study used the light yellow sulphur granules in the molten state at approximately 140 °C. The analytical report of sulphur used in the present study is presented in Table I.

TABLE I. ANALYTICAL REPORT OF SULPHUR

Constituents	% By weight			Avg.
	1	2	3	
Moisture (%)	0.120	0.190	0.210	0.173
Ash content (%)	0.013	0.013	0.012	0.012
Acidity as H_2SO_4 (%)	0.060	0.070	0.030	0.053
Chloride as Cl (%)	0.007	0.007	0.008	0.007
Purity (%)	99.97	99.98	99.93	99.96

B. Mix Design and Casting of Concrete

The concrete mix design for the SIC trials was based on three different cases. Each case involved a different *w/c* ratio and three different cement contents. Trail 1 used OPC (i), with a *w/c* ratio of 0.5 and a cement content of 330 kg/m^3; Trail 2 used OPC (ii), with a *w/c* ratio of 0.6 and a cement content of 275 kg/m^3; and Trail 3 used OPC (iii), with a *w/c* ratio of 0.7 and a cement content of 235.71 kg/m^3. The water content was kept constant at 165 kg/m^3, as presented in Table II.

TABLE II. MIX PROPORTION OF CONCRETE WITH DIFFERENT CEMENT AND *w/c* RATIOS

Sample	Cement (kg/m^3)	Sand (kg/m^3)	Agg.20 mm (kg/m^3)	Agg.10mm (kg/m^3)	Water (kg/m^3)	*w/c*	*a/c*
OPC (i)	330	953	585	440	165	0.50	5.99
OPC (ii)	275	976	578	472	165	0.60	7.37
OPC (iii)	235.71	992	578	492	165	0.70	8.74

The SIC testing procedure involves casting samples as per the mix design, demolding, curing for 24 h for 1 day and 168 h for 7 days, then oven drying at 110°C for 24 h to ensure complete internal drying and prevent polysulfide formation. Dried specimens are then infiltrated with molten sulphur at 140°C, as portrayed in Figure 1.

C. Testing of Concrete Specimens (OPC)

Compressive strength tests were carried out on the concrete samples of the three aforementioned cases using both destructive and non-destructive methods, with and without sulphur infiltration. Rebound Hammer tests were conducted to assess compressive strength at 1 day and 7 days of curing. UPV tests were performed to evaluate concrete quality. Carbonation tests using phenolphthalein indicator assessed durability, with pink colour indicating non-carbonated concrete, while colorless concrete indicated carbonated concrete. Water absorption tests were performed using distilled water to determine the porosity and permeability characteristics of the specimens. Microstructural characterization was conducted through SEM to examine the morphology of hydration products, pore structure, and interfacial transition zones. XRD analysis was employed to identify crystalline phases and assess the degree of cement hydration in all concrete specimens.

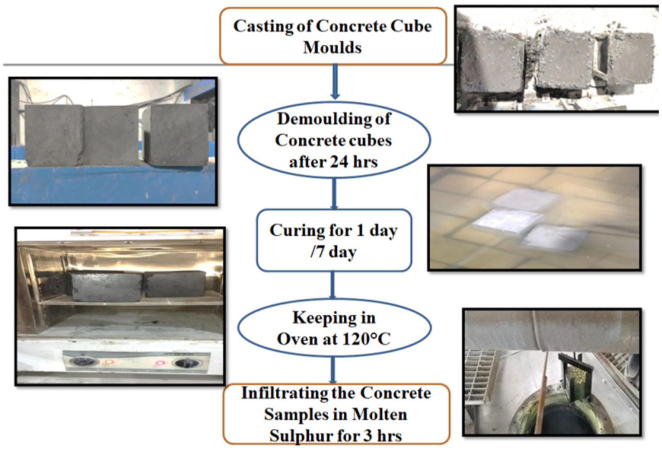

Fig. 1. Methodology for casting and curing the concrete specimens.

III. RESULTS AND DISCUSSION

Based on the adopted methodology, the following results were obtained from various tests conducted on SIC.

A. Compressive Strength Test Results for SIC Using Destructive Testing Method

Trail 1, OPC (i), compressive strength results at 1 and 7 days of curing and 1 day oven drying are as shown in Figure 2, while Trail 2, OPC (ii), compressive strength results at 1 and 7 days of curing and 1 day oven drying are as displayed in Figure 3.

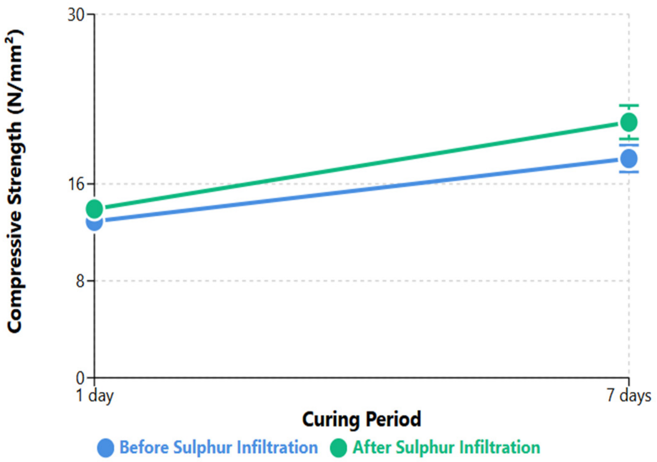

Fig. 2. Compressive strength development of OPC (i) specimens, with *w/c* of 0.50 and cement content of 330 kg/m³, before and after sulphur infiltration with standard deviation error bars.

For OPC (ii), the specimens exhibited lower compressive strength at both 1 day and 7 days of curing compared to Trial 1 OPC (i). OPC (iii) also demonstrated a lower strength at both 1 day and 7 days of curing. Compared to the first two cases, the compressive strength increased with an increase in *w/c* ratio, as the higher porosity associated with larger w/c ratios allowed greater infiltration of molten sulphur into the concrete matrix.

Trail 3, OPC (iii), compressive strength results at 1 day of curing and 1 day oven drying are presented in Figure 4. Sulphur infiltration significantly enhanced early-age compressive strength by 4.70 N/mm² (59.3%) at 1 day of curing, with the improvement reducing to 1.83 N/mm² (9.6%) at 7 days as normal cement hydration progressed.

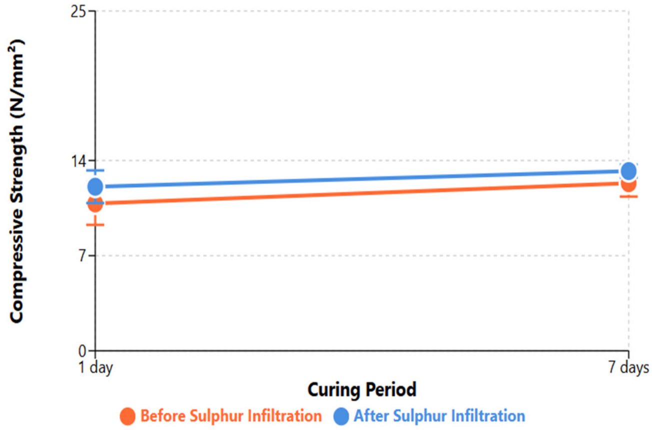

Fig. 3. Compressive strength of development OPC (ii) specimens, with a *w/c* ratio of 0.60, and cement content of 275 kg/m³, before and after sulphur infiltration with standard deviation error bars.

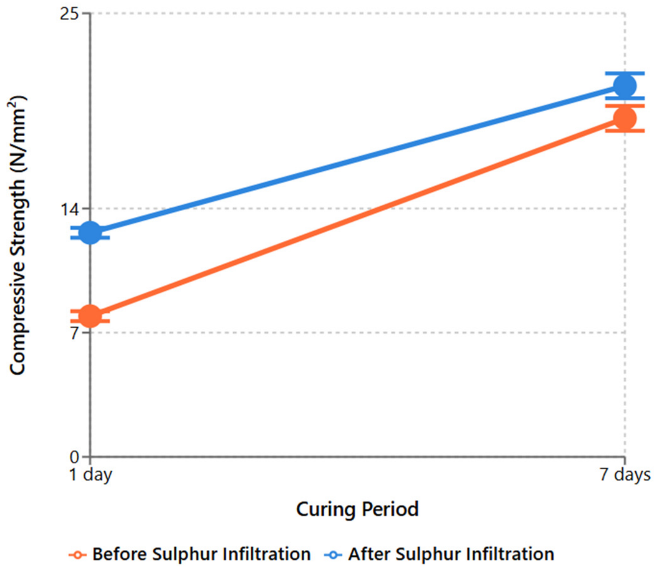

Fig. 4. Compressive strength development of OPC (iii) specimens, *w/c* of 0.70 and cement content of 235.71 kg/m³, before and after sulphur infiltration with standard deviation error bars.

B. Compressive Strength of SIC Using the Rebound Hammer

Rebound Hammer test was conducted in accordance with IS: 13311 (Part 2)–1992 at 1 and 7 days post-sulphur infiltration. For Trial 1, compressive strength increased from 13.5 to 20.0 N/mm², for Trial 2 from 14.0 to 18.9 N/mm². In contrast, in Trial 3, compressive strength decreased from 16.8 to 15.8 N/mm². The higher 1-day compressive strength in Trial 3 suggests that greater porosity enabled deeper sulphur penetration, increasing early-age surface hardness, as illustrated in Figure 5.

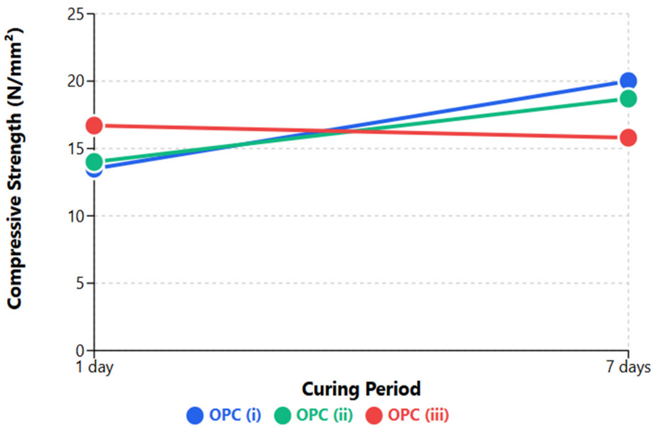

Fig. 5. Effect of curing duration on compressive strength of molten sulphur-infiltrated OPC concrete for three formulation variants.

C. Quality Assessment Using the UPV Test

UPV test was conducted in accordance with IS 13311:1992 at 1 and 7 days of curing to assess internal structure development. All trials showed progressive densification: For Trial 1, pulse velocity increased from 3.12 to 3.27 km/s, for Trial 2, pulse velocity increased from 3.05 to 3.43 km/s, and Trial 3 recorded the highest pulse velocity of 3.59 km/s, decreasing slightly to 3.40 km/s at 7 days. The increasing pulse velocities in Trials 1 and 2 indicate improved material homogeneity and compaction with curing time.

D. Influence of w/c Ratio on Sulphur Infiltration and Strength Attribution

The *w/c* ratio controls sulphur penetration through capillary porosity. Visual examination indicated progressively deeper sulphur infiltration with increasing *w/c* ratio, with OPC (iii) exhibiting enhanced penetration compared to OPC (i) and OPC (ii), though complete infiltration was not achieved in 150 mm cubes. Strength attribution analysis revealed that at 1-day, sulphur contributed 37% of total strength in OPC (iii) (a gain of 4.70 N/mm² from 7.93 N/mm² baseline), while cement hydration contributed 63%; at 7-day, sulphur's contribution decreased to 9% (a 1.83 N/mm² gain from 19.00 N/mm² baseline), with hydration dominating at 91%. The 59.3% early-age strength improvement in OPC (iii), compared with 13.3% in OPC (i), confirms that higher *w/c* ratios amplify sulphur's contribution through greater void volume. UPV data support this, with OPC (iii)-SIC achieving the highest velocity (3.59 km/s) despite the lowest cement content, confirming enhanced homogeneity from sulphur void-filling. While direct porosity measurements were not performed, the *w/c* ratio serves as an indirect indicator of initial porosity, with the systematic correlation between *w/c* ratio, strength improvement, and UPV values providing indirect evidence of the porosity-infiltration-performance relationship.

E. Carbonation

Carbonation tests using phenolphthalein indicator were conducted on concrete samples after sulphur infiltration. All specimens from Trials 1, 2, and 3 turned pink, indicating no carbonation. This confirms that molten sulphur infiltration preserved the concrete's high pH and protective qualities against steel corrosion.

F. Impact of Distilled Water and Water Absorption

The SIC samples were immersed in distilled water for one month to assess water absorption. Specimen weights were recorded before and after immersion. The results showed that SIC specimens absorbed less than 8% water by weight, demonstrating that molten sulphur effectively sealed surface voids and restricted moisture penetration. The OPC (iii) sample with a higher *w/c* ratio (0.70) showed slightly greater water absorption due to higher initial porosity, which allowed limited water ingress despite sulphur infiltration. The minimal mass increase confirms that SIC samples exhibit low water absorption characteristics.

G. Microstructural Analysis

1) SEM Analysis

SEM was conducted to investigate the microstructural characteristics of three OPC concrete specimens designated as OPC (i), OPC (ii), and OPC (iii). The analysis focused on evaluating the morphology of hydration products, pore structure distribution, and interfacial transition zone characteristics.

a) Specimen OPC (i)

The SEM micrograph revealed a relatively dense microstructure with well-formed calcium silicate hydrate (C-S-H) gel. The presence of needle-like ettringite crystals and hexagonal portlandite (CH) plates indicated proper cement hydration. The pore distribution, as illustrated in Figure 6, appeared relatively uniform with minimal micro-cracks, suggesting good compaction during casting.

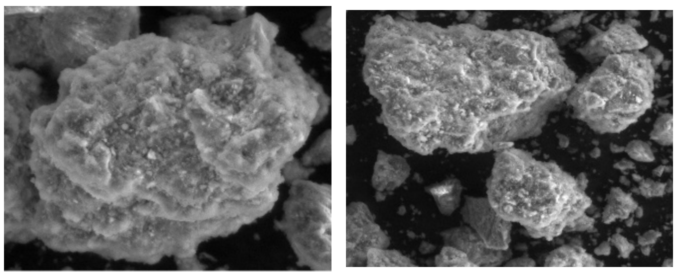

Fig. 6. SEM image of OPC (i) concrete specimen.

b) Specimen OPC (ii)

The microstructure exhibited moderate densification with visible capillary pores distributed throughout the cement matrix. Hydration products showed characteristic fibrous morphology of C-S-H gel interspersed with crystalline phases. The interfacial transition zone between aggregate and cement paste displayed adequate bonding, though some minor gaps were observed, as presented in Figure 7, potentially affecting mechanical performance.

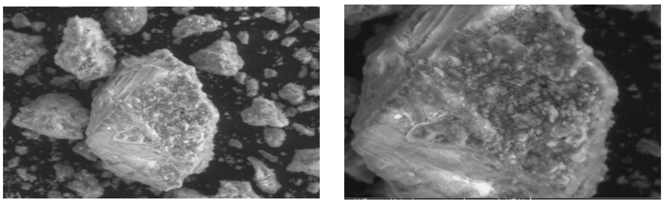

Fig. 7. SEM image of OPC (ii) concrete specimen.

c) Specimen OPC (iii)

OPC (iii) specimen demonstrated a heterogeneous microstructure with distinct variations in pore size distribution. The cement matrix revealed irregular morphology with both dense and porous regions. Hydration products were evident, though the degree of hydration appeared variable across different zones. Some microcracks were visible at the interface, as shown in Figure 8, indicating potential weak zones that could influence durability and strength characteristics.

The comparative SEM analysis revealed microstructural differences among the three specimens, attributed to variations in mixing procedures, curing conditions, or *w/c* ratios. These microscopic observations correlate with the macroscopic mechanical properties and provide valuable insights into concrete quality assessment and performance prediction.

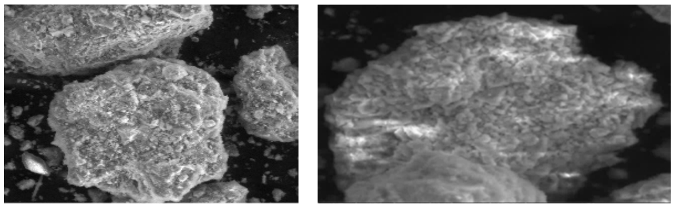

Fig. 8. SEM image of OPC (iii) concrete specimen.

2) XRD Analysis

XRD analysis was performed on all three OPC concrete specimens to identify the crystalline phases and assess the degree of cement hydration. The diffraction patterns were recorded over a 2θ range, and the results are presented in Figures 9-11 for specimens OPC (i), OPC (ii), and OPC (iii), respectively.

a) XRD Pattern of OPC (i)

The diffractogram of the OPC (i) specimen exhibited characteristic sharp peaks indicating the presence of well-crystallized hydration products. The prominent peaks correspond to portlandite ($Ca(OH)_2$), ettringite (calcium sulfoaluminate hydrate), and calcium silicate hydrate (C-S-H) phases. The high-intensity peaks at specific 2θ angles suggest complete cement hydration with minimal unhydrated cement particles. The baseline intensity remained relatively low, indicating reduced amorphous content and good crystallinity of the hydrated matrix, as shown in Figure 9.

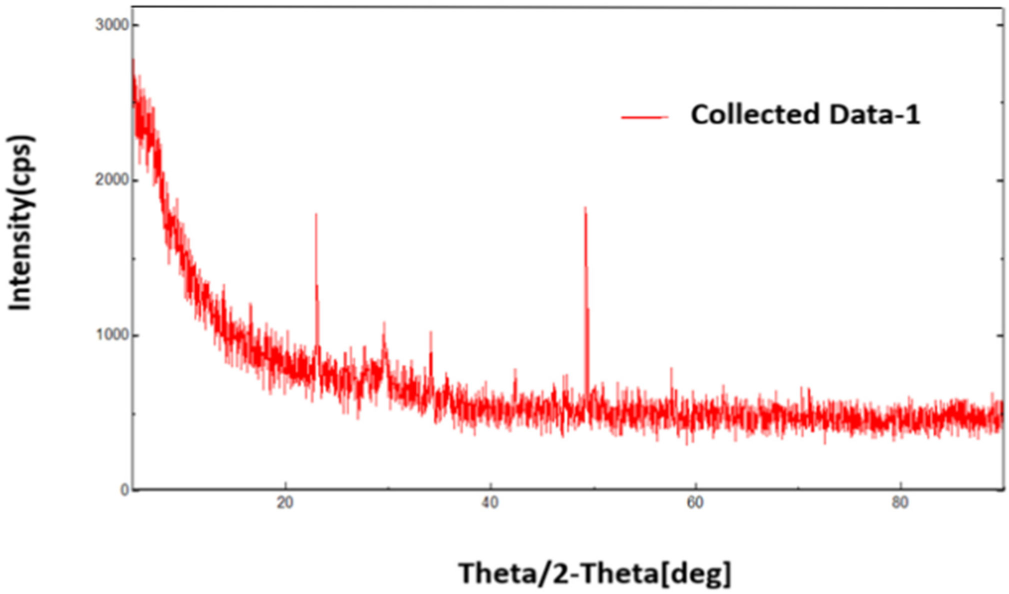

Fig. 9. XRD pattern of OPC (i) concrete specimen.

b) XRD Pattern of OPC (ii)

The XRD pattern of OPC (ii) specimen, as depicted in Figure 10, demonstrated a similar diffraction pattern with characteristic peaks of major hydration products, though slight variations in peak intensities compared to OPC (i) suggest differences in crystalline phase proportions. Portlandite peaks confirmed adequate hydration, while the C-S-H gel contribution to the amorphous hump indicated proper cement matrix development. Minor unreacted clinker phase peaks (alite and belite) suggested incomplete hydration in certain regions.

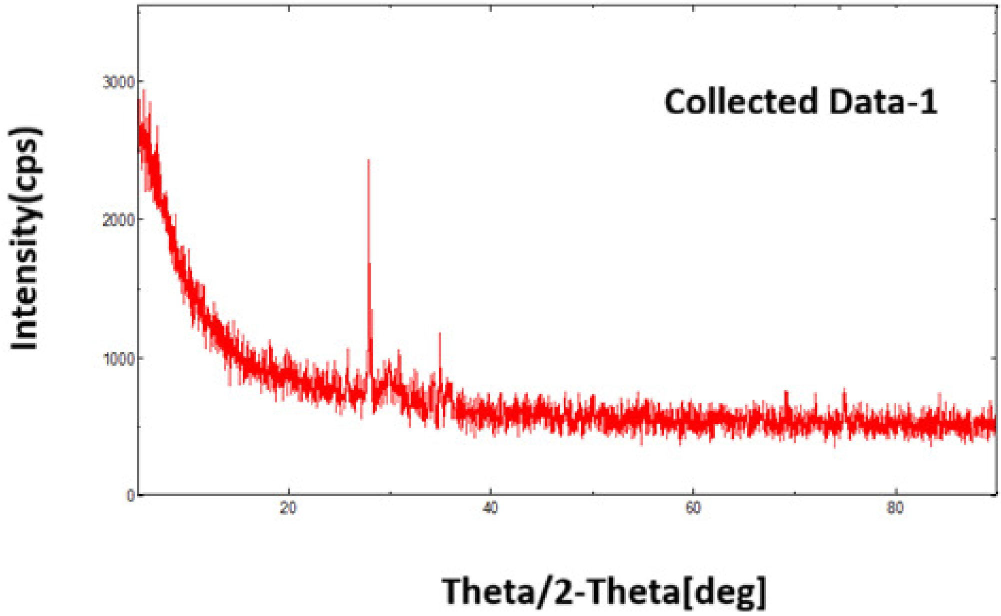

Fig. 10. XRD pattern of OPC (ii) concrete specimen.

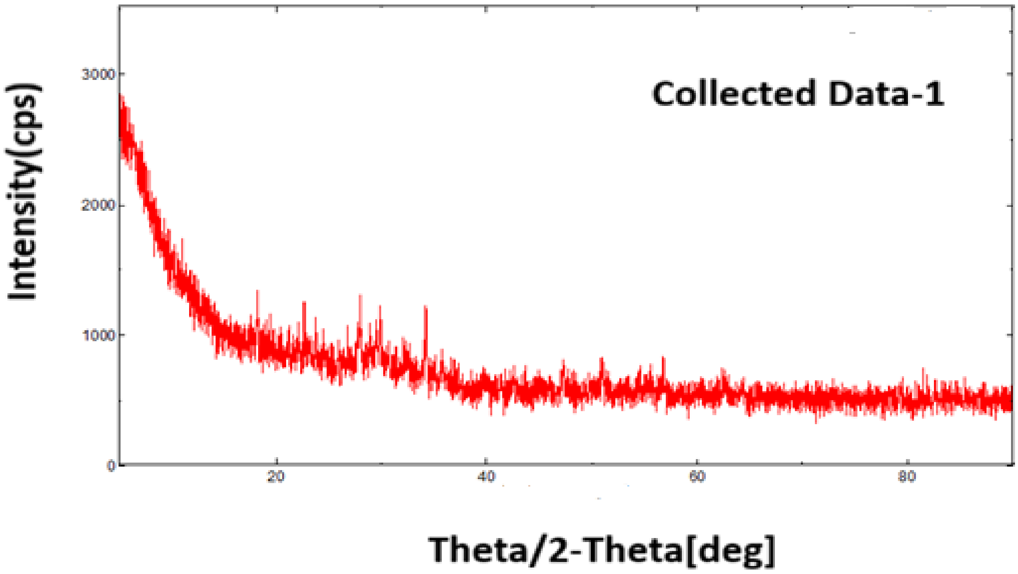

Fig. 11. XRD pattern of OPC (iii) concrete specimen.

c) XRD Pattern of OPC (iii)

The diffractogram of OPC (iii), as shown in Figure 11, demonstrated distinct crystalline phases with peak intensity variations compared to OPC (i) and OPC (ii). Characteristic peaks for calcium hydroxide, ettringite, and calcite were identified. The elevated baseline indicated higher amorphous C-S-H gel content, the primary strength-contributing phase. Peaks corresponding to residual unhydrated cement suggested variable hydration kinetics due to differences in curing conditions or *w/c* ratio.

XRD patterns confirmed typical OPC hydration products with variations in peak intensities, indicating different hydration degrees. OPC (i) showed complete hydration, while OPC (iii) exhibited partial hydration with higher amorphous content. These differences correlate with SEM observations and influence mechanical performance and durability.

IV. CONCLUSIONS

This study evaluated Sulphur-Infiltrated Concrete (SIC) performance across varying water-cement (*w/c*) ratios of 0.50-

0.70, and cement contents of 235.71-330 kg/m³. Based on specimens cured at 1 day and 7 days, the following conclusions are drawn:

- Standard cube specimens (150×150×150 mm³) revealed incomplete sulphur infiltration to the center, indicating the need for a smaller size for optimal sulphur infiltration.
- OPC (iii) with lower cement content (235.71 kg/m³) and higher *w/c* ratio (0.70) achieved superior compressive strength at 1-day curing, demonstrating that increased porosity enables enhanced sulphur infiltration with strength gains of 59.3% compared to 13.3% in lower *w/c* ratio specimens, though higher initial cement content provides greater baseline strength.
- Non-destructive Rebound Hammer testing yielded results comparable to conventional destructive compression testing, validating its reliability for SIC assessment.
- Carbonation testing confirmed no immediate deterioration, with phenolphthalein indicator showing pink coloration in all specimens.
- Water immersion studies demonstrated minimal mass gain (<8% absorption), confirming low permeability and structural integrity throughout the immersion period.
- Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD) analyses confirmed microstructural variations in pore distribution and hydration phases that directly correlated with mechanical performance differences among specimens.

The novelty of this research lies in quantifying the individual contributions of sulphur infiltration compared to contributions of cement hydration to overall strength at different curing ages. The sulphur contributes 37% at 1-day and 9% at 7-day in OPC (iii), while cement hydration contributions increase from 63% to 91%. This study adds to existing knowledge by demonstrating, for the first time, that reduced cement content (28.6% reduction) with optimized *w/c* ratio (0.70) achieves superior early-age strength through a systematic investigation of the *w/c*-porosity-infiltration relationship. The systematic correlation between *w/c* ratio, sulphur penetration depth, and mechanical performance provides practical guidance for SIC mix design. These findings establish SIC as a viable, sustainable alternative for precast construction, offering dual benefits of industrial waste valorization and reduced cement consumption.

For future studies, testing smaller specimens (100×100×100 mm³) would ensure complete infiltration. Future research should also investigate long-term durability, sulphur-concrete interfacial bonding using SEM-EDX, and optimized infiltration rates for industrial scalability. While initial durability indicators are promising (zero carbonation, <8% water absorption), anticipated degradation mechanisms, including sulphur/calcium leaching, thermal softening, and interfacial cracking, require validation through wet-dry cycling, thermal exposure, and chemical resistance testing across multiple climate zones.

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