Research Article

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A Comprehensive Microstructural Analysis for Enhancing Concrete's Longevity and Environmental Sustainability

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ABSTRACT

The environmental factors lessen the durability of concrete by changing the way the elements are linked together. To maximize the building's longevity, the concrete needs to be impermeable. Samples of Portland slag cement concrete of grade M20 that had been produced with and without admixtures as well as with and without reinforcement were taken into account for the current investigation. Samples were initially cured in potable water for 28 days, following which they were subjected to additional exposure in environments containing hydrochloric acid and sulphuric acid for challenging conditions for a subsequent 28-, 56- and 90-day period. All specimens exposed to various environmental circumstances showed a similar pattern of enriching in physical characteristics and diminishing in water absorption, therefore enriching the durability qualities, when the data gathered at intervals of 28, 56, and 90 days of curing were examined. SEM examination revealed a notable refinement in pore structure with aging, confirming age-related pore refinement. XRD analysis performed for phase identification revealed the presence of silica and calcium carbonate in the concrete specimens. This research intends to offer important insights about the appropriateness of Portland Slag Cement in strengthening the resilience and durability of concrete.

Keywords: Portland slag cement; Durability; Microstructural analysis.

1. INTRODUCTION

The mechanical strength of concrete mixtures is significantly increased by the addition of slag. Concrete specimens' compressive and flexural strengths have been discovered to be positively impacted by slag, a widely accessible waste from the metallurgical sector (Zhao *et al*. 2021). The stimulation of extra hydration products within the concrete matrix, particularly the creation of calcium silicate hydrate (C-S-H), is responsible for this advantageous impact. As a result, this procedure helps to create concrete buildings that have a higher density and greater overall strength, with the potential for enhancing mechanical strength, durability, and microstructural properties (Arasu *et al*. 2023). The utilization of slag as a supplementary cementitious material offers a sustainable approach to creating resilient and long-lasting concrete structures. To explore the full range of benefits and optimize mix designs for specific applications (Ojha *et al*. 2023).

Slag's ability to dramatically increase mechanical strength when added to concrete mixes has been repeatedly shown via research. Slag's pozzolanic and latent hydraulic capabilities increase the compressive and flexural strengths of the material. Slag-based concrete is a great option for load-bearing constructions because the production of calcium silicate hydrate (C-S-H) gel leads to a denser microstructure and enhanced strength (Goyal *et al*. 2023). Slag-based concrete has outstanding durability, outperforming conventional concrete in its resistance to alkali-silica reaction (ASR), sulfate assault, and chloride ion penetration (Yang *et al*. 2023). The improved hydration products and fine-tuned pore structure are responsible for its decreased permeability. Incorporating slag also supports environmental objectives because it uses industrial waste to lessen the carbon footprint of concrete manufacturing (Arasu *et al*. 2023).

Durability is a vital aspect of concrete performance. Slag-based concrete exhibits enhanced durability characteristics, including increased resistance to chloride ion penetration, sulfate attack, and alkali-silica reaction (ASR) (Mavroulidou *et al*. 2023). The use of slag mitigates the deleterious effects of aggressive environments on concrete structures, extending their service life. Advanced analytical techniques, including Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD), have provided crucial insights into the microstructural changes induced by slag (Zhang and Çopuroğlu, 2022). Studies have revealed a finer and more homogeneous microstructure in slag-based concrete, resulting in reduced porosity and enhanced resistance to deleterious agents. The formation of secondary hydration products, such as ettringite, contributes to the improved microstructure (Naveen *et al*. 2020).

In-depth microstructural analysis has played a vital role in understanding the mechanisms behind the improved performance of slag-based concrete. Advanced techniques such as SEM and XRD have revealed a finer and more homogenous microstructure (Li *et al*. 2022). The refined pore structure and secondary hydration products contribute to enhanced durability (Revilla *et al*. 2022). Slag-based concrete exhibits exceptional durability characteristics, making it resistant to a range of environmental challenges. By utilizing industrial byproducts like slag, the construction industry can reduce its carbon footprint, aligning with sustainability objectives (Dai et al. 2002).

The processes underlying the enhanced performance of slag-based concrete have become increasingly clear thanks to thorough microstructural study. A finer and more homogeneous microstructure has been discovered using cutting-edge methods including SEM and XRD (Wang *et al*. 2022). Durability is improved because of the improved pore structure and secondary hydration products (Kadhar *et al*. 2024). Microstructural analysis plays a crucial role in understanding the behavior of slag-based concrete. SEM and XRD studies have revealed that slag incorporation leads to refined pore structures and the formation of stable hydration products. This contributes to improved concrete density and reduced permeability (Boudache *et al*. 2023).

The microstructural investigation provides important insights into the changes brought on by nano-silica (Yuan *et al*. 2023). It illustrates the development of a finer, denser microstructure with a higher concentration of C-S-H gel. Improved mechanical qualities and longevity are a result of this revised structure (Liang *et al*. 2023). The addition of nano-silica to PSC has several useful ramifications. It improves early-age and long-term strength, making it appropriate for applications requiring high-performance concrete or speedy building. Additionally, PSC enhanced with nano-silica may have advantages in infrastructure projects like bridges and marine constructions due to its improved durability and decreased permeability (Kim, 2022).

1.1 Research Significance

Making concrete to perform better in various adverse situations has undergone a paradigm shift in recent years. Through the use of cutting-edge cement and aggregate substitutes, research is being done to alter the characteristics of concrete. Micro-fillers sprang to prominence as a cement substitute at a certain point in time. For this purpose, a wide variety of materials entered the market. However, because these materials are not as reactive at first, the early age strength could not be achieved with them. However, concrete's performance improved, and as a result, these materials remain at the top. According to a new school of thought, the practical difficulties noted above can be mitigated by adding minute amounts of nano-fillers such as micro silica, alumina and minute clay. Particularly, in the early years, nanofillers have more pozzolanic action than microfillers. The fact that the nano-fillers serve as both a binder and a filler is what makes this observation more intriguing. In this connection, research has been done to determine how adding steel fibers to concrete that contains micro sand may affect its mechanical and other qualities. A suitable conclusion has been drawn from the experimental study.

2. MATERIAL PROPERTIES

2.1 Cement

The features of the cement sample under examination can be used to learn vital details about its performance and quality. The cement's 2% fineness is within allowable bounds, suggesting an appropriate particle size distribution. The cement's 2.68 specific gravity indicates that it has a typical density. The cement's standard consistency of 32% suggests that just a small amount of water is necessary to give it the proper workability, which is a benefit for building. Additionally, the cement has sufficient working time, allowing for correct placement and completion during building operations, as shown by the starting setting time of 113 minutes and the total setting time of 248 minutes. At 3 days, 7 days, and 28 days, the cement mortar's compressive strength was 12.08, 17.51, and 29.63 MPa, respectively. According to these findings, the cement strengthens with time and satisfies or surpasses the conventional strength criteria for a variety of building applications.

2.2 Fine Aggregate

The characteristics of the sand sample offered provide important information about its appropriateness for engineering and building applications. It has a modest density that is characteristic of building sands; 2.65 relative density to be exact. It is excellent for a variety of concrete and mortar applications due to its fineness modulus of 2.72, which denotes a balanced particle size distribution. Due to its evenly distributed particle sizes, it is known as medium sand and provides adaptability for building projects. According to Bureau of Indian Standards (BIS) grading specifications for concrete manufacture, being categorized as Zone II is appropriate. The mix should be designed with moisture absorption in mind for uniformity and workability, as indicated by the 4.05% water absorption. Last but not least, with a loose bulk density of 1667 kg/m^3 and a dry-rodded bulk density of 1833 kg/m^3, these properties are essential for figuring out the amount of sand to use in concrete mixtures and guaranteeing correct compaction. When employed in line with specific project needs and mix designs, these characteristics define the sand's appropriateness for building.

2.3 Coarse Aggregate

The provided material exhibits promising characteristics for a range of applications. Its high relative density of 2.988 suggests that it is dense and compact, which can make it suitable for applications where structural integrity and strength are essential. The low water adsorption rate of 0.58% is indicative of its resistance to moisture penetration, making it an attractive choice for projects requiring materials with good moisture resistance properties, such as in outdoor construction or damp environments. Additionally, the volumetric density of 1528 kg/m^3 falls within a typical range for construction materials, contributing to its overall stability and durability in various settings. These properties collectively imply that the material may be well-suited for tasks such as structural components in construction, where high-density materials are favored for load-bearing capacities. Its resistance to moisture makes it an ideal candidate for outdoor structures or environments with fluctuating humidity levels. However, the suitability of this material for specific applications should be assessed in the context of project requirements, as other factors like cost, availability, and environmental considerations also play crucial roles in material selection. Nonetheless, these properties offer an excellent starting point for considering this material in construction and engineering projects, with the potential to provide both strength and resilience

2.4 Curing

Curing is an essential step in the production of concrete buildings that affects their overall performance, strength, and longevity. Three different techniques are used throughout the curing process to obtain different results: conventional, hydrochloric acid, and sulfuric acid curing techniques. The most widely used technique of curing concrete is known as conventional curing, which entails maintaining freshly put concrete moist at a set temperature for a predetermined amount of time. This method ensures that cement particles are properly hydrated, resulting in the strongest possible strength development and the least amount of surface cracking. Contrarily, the precast concrete industry frequently uses the specialized method of hydrochloric acid curing to speed up the curing process. A diluted hydrochloric acid solution is sprayed on or submerged in the concrete during this process. This concrete's strength is maintained while the curing process is sped up by the acidic environment's accelerated chemical reactions. To avoid excessive reinforcement corrosion, it does, however, require strict supervision and monitoring. Another fast curing technique is sulfuric acid curing, which is frequently employed in scientific settings. To measure early-age strength development, concrete samples are exposed to sulfuric acid vapor, which dramatically accelerates the process.

3. EXPERIMENTAL INVESTIGATION

3.1 Saturated Water Absorption

The long-lasting of concrete depends partly on the saturation water absorption. Using ASTM C642, the water absorption of concrete cubes was assessed. 150 mm cube concrete examples were cast, and they were given 28, 56 and 90 days to cure in water for each mix percentage. The cube samples were taken out of the curing tank after the curing period and kept at 105 °C for twenty-four hours in a hot air oven. The dried specimens were put back on display when they had cooled to normal temperature. The dry weight of the samples was determined and recorded. The dried specimens were then kept in a water container. Before weighing the item, the surface was frequently wiped with a dry towel. The weight was monitored for two consecutive observations or at least 48 h, whichever occurred first, before stopping. The weight of the specimen was noted as its wet weight.

3.2 Porosity

The water displacement method is used for porosity determination to evaluate void areas in materials. The original dry weight of the sample (W-dry) is noted. The sample is moved around in a graduated cylinder with a known beginning water volume (V-initial). The new water volume (V-final), which has stabilized, is noted. By deducting V-initial from V-final, the volume displaced is computed. This reveals material voids in relation to the entire volume. Porosity is important to geology and material science because it sheds light on interior structure. The equipment and sample are properly cleaned and dried to bring the process to a close.

3.3 Acid Resistance Test

Acid resistance testing evaluates materials, especially concrete and building materials, to see how well they can endure being exposed to acidic conditions. This test evaluates a material's ability to withstand the corrosive effects of acids, which can occur in a variety of sectors, including infrastructure, wastewater treatment, and chemical processing. The material is tested by being exposed to particular acidic solutions under controlled circumstances, often sulfuric or hydrochloric acid. The bodily changes and weight loss of the substance are tracked over time. The findings offer insightful information on the material's resilience and appropriateness for applications where exposure to acids is anticipated, assisting engineers and designers in making decisions that will preserve the structural integrity of structures and components over the long term.

3.4 Sorptivity Test

As a result of capillary action on a porous media, water rises from a single surface. In 2012, Hall and Hoff developed this testing procedure. This is the simplest and most successful method for measuring the amount of water that enters through capillary action. Water curing was performed on concrete samples 0.15 m cubes for 28, 56 and 90 days. After the curing, the specimen was oven-dried for three days at 100°C. After three days of the oven-drying process, the specimen was cooled to room temperature. To prevent water infiltration, epoxy was coated on the cube specimen's surfaces except the top and bottom faces. The specimen's starting weight was noted at time 0. The specimen was made to submerge in water up to 10 mm. The value of sorptivity was calculated using best fitting slope of the line composed.

3.5 Rapid Chloride Penetration Test (RCPT)

When assessing the permeability and durability of concrete buildings, the Rapid Chloride Penetration Test (RCPT) is an essential evaluation technique. It gauges the speed at which chloride ions (often found in de-icing solutions or maritime environments) can pierce the concrete's surface and make their way to the reinforcing, where they might cause corrosion. A concrete specimen is exposed to an electrical field during the RCPT, which forces chloride ions into the substance. It is a useful method for determining concrete's resistance to chloride ion infiltration because the test lasts only a few days on average. The findings of the RCPT assist engineers in assessing the susceptibility of concrete structures to chloride-induced corrosion and in making educated judgments about the design of the concrete mix and preventative actions to maintain the durability and security of the infrastructure.

3.6 SEM

SEM is a useful instrument for testing and investigating on physical objects. It makes it possible to examine concrete specimens' microstructures in great detail. The cementitious matrix, aggregate distribution, pore structure, and the existence of any fractures or other flaws in the concrete may all be revealed by SEM. SEM may provide light on the hydration process in concrete analysis, which is essential for comprehending the growth of concrete strength and durability. It can also assist researchers in examining how different admixtures and additives affect the characteristics of concrete. To better understand how concrete deteriorates over time, researchers can employ SEM to investigate concrete samples that have been exposed to extreme climatic conditions or chemical assaults. Using this knowledge, stronger concrete mixtures and building methods may be created.

3.7 XRD

An effective analytical method for examining the crystalline structure and mineralogical makeup of the components found in concrete mixes is X-ray Diffraction (XRD). When using X-rays to analyze concrete samples, diffraction patterns created by the X-rays' interactions with the sample's crystalline components are recorded and analyzed. When used to identify phases like calcium hydroxide (CH), calcium hydroxide (C-S-H), and numerous crystalline phases including quartz, calcite, and others, XRD can offer useful information on the types and quantities of minerals present. Understanding the characteristics of concrete, such as its strength, durability, and sensitivity to various environmental variables, requires knowledge of these data.

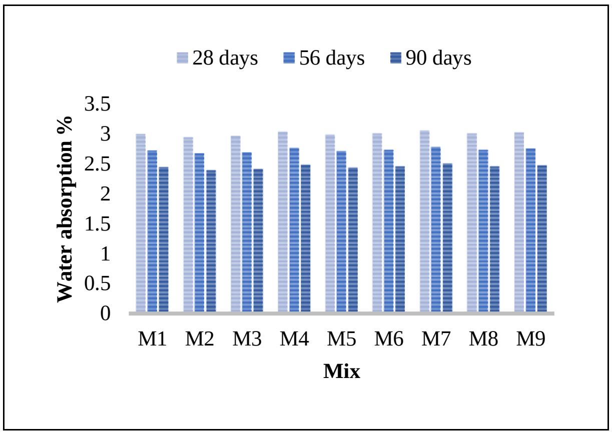

Fig. 1: Graphical representation of saturated water absorption

4. RESULTS AND DISCUSSION

4.1 Saturated Water Absorption

The results demonstrate that all three mix designs had relatively low water absorption percentages at 28 days, ranging from 2.44 to 2.99%, in the event of normal curing (M1, M2, and M3). This proves that traditional curing, whether it involves the use of admixtures or not, produces concrete that has good resistance to moisture infiltration. A modest decrease in water absorption percentages was seen as the curing time increased to 90 days, indicating that the concrete's durability and moisture resistance would continue to increase with time. There was a notable increase in the percentage of water absorption when the specimens were treated to more aggressive curing techniques using hydrochloric acid (M4, M5, and M6) and sulphuric acid (M7, M8, and M9) in comparison to standard curing. This rise in permeability can be attributed to the acids' corrosive properties, which may have led to some surface degradation, microcracking, or alterations in the pore structure of the concrete, all of which enhanced permeability. These results highlight the need to choose a suitable curing technique for concrete mix designs based on particular requirements and environmental circumstances. Fig. 1 shows the graphical variation of the water absorption percentages of various concrete mixtures at different curing durations.

4.2 Porosity

The effective porosity percentages for various concrete mix designs subjected to different curing conditions at 28 days, 56 days, and 90 days reveals critical insights into the material's durability and resistance to harmful substance penetration. In the context of normal curing (M1, M2, and M3), all three mix designs displayed relatively low effective porosity percentages at 28 days, ranging from 5.76 to 5.82%. This signifies that conventional curing methods, whether with or without admixtures, generate concrete with excellent resistance to harmful substance ingress. As the curing period extended to 56 days and 90 days, there was a consistent reduction in effective porosity, indicating an improvement in the concrete's durability and resistance to permeation over time. When the specimens were subjected to more aggressive curing methods involving hydrochloric acid (M4, M5, and M6) and sulphuric acid (M7, M8, and M9), there was a noticeable increase in effective porosity percentages compared to normal curing. This aligns with the corrosive nature of the acids, potentially causing surface deterioration and heightened susceptibility to permeation. Fig. 2 shows the graphical variation the porosity percentages of various concrete mixtures at different curing durations.

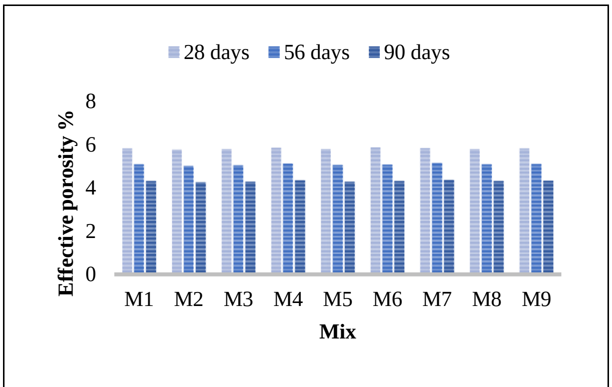

Fig. 2. Graphical representation of porosity

4.3 Acid Resistance Test

In the case of normal curing (M1, M2, and M3), all three mix designs exhibited relatively low weight loss percentages at 28 days, ranging from 4.59 to 4.66%. This suggests that conventional curing methods, whether with or without admixtures, result in concrete with good resistance to environmental factors. Over the extended curing periods of 56 days and 90 days, there is a trend of gradual reduction in weight loss percentages, indicating an improvement in the concrete's durability and resistance to deterioration over time. Conversely, when the specimens were subjected to more aggressive curing methods involving hydrochloric acid (M4, M5, and M6) and sulphuric acid (M7, M8, and M9), there was a noticeable increase in weight loss percentages compared to normal curing. This is consistent with the corrosive nature of the acids, which may have led to surface erosion and increased the concrete's susceptibility to deterioration. Fig. 3 shows the graphical variation in the percentage of loss in weight of various concrete mixtures at different curing durations.

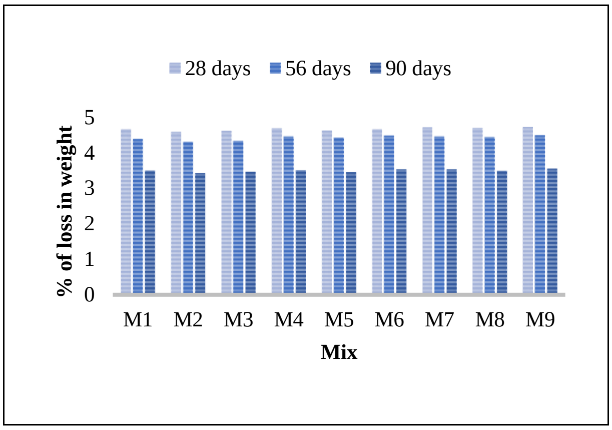

Fig. 3: Graphical representation of acid resistance test

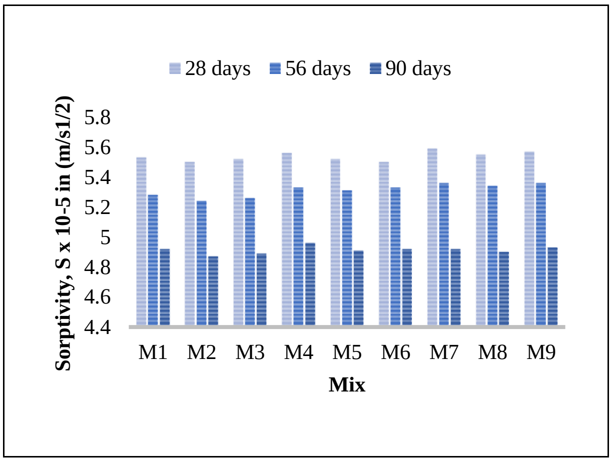

Fig. 4: Graphical representation of sorptivity test

4.4 Sorptivity Test

In the case of normal curing (M1, M2, and M3), all three mix designs displayed relatively similar sorptivity values at 28 days, ranging from 5.50 x 10^{-5} to 5.53 x 10^{-5} m/$s^{1/2}$. This indicates that conventional curing methods, whether with or without admixtures, result in concrete with comparable moisture absorption properties. Over the extended curing periods of 56 days and 90 days, there is a trend of gradual reduction in sorptivity values, suggesting an improvement in the concrete's resistance to moisture ingress over time. When the specimens were subjected to more aggressive curing methods involving hydrochloric acid (M4, M5, and M6) and sulphuric acid (M7, M8, and M9), there was a slight increase in sorptivity values compared to normal curing. This could be attributed to the corrosive nature of the acids, potentially causing minor surface alterations that may have increased moisture absorption. Fig. 4 shows the graphical variation the sorptivity of various concrete mixtures at different curing durations.

4.5 Rapid Chloride Penetration Test

In the case of normal curing (M1, M2, and M3), all three mix designs exhibited relatively similar total charge period values at 28 days, ranging from 2136 to 2157 Coulombs. This suggests that conventional curing methods, whether with or without admixtures, yield concrete with comparable resistance to the initiation of reinforcement corrosion. Over the extended curing periods of 56 days and 90 days, there is a trend of gradual reduction in total charge period values, indicating an improvement in the concrete's resistance to corrosion over time. When the specimens were subjected to more aggressive curing methods involving hydrochloric acid (M4, M5, and M6) and sulphuric acid (M7, M8, and M9), there was a slight increase in total charge period values compared to normal curing. This suggests that these acid-based curing methods did not significantly accelerate the corrosion of embedded reinforcement, at least within the tested timeframes. Fig. 5 shows the graphical variation in the total charge period (in Coulombs) for various concrete mixtures at different curing durations.

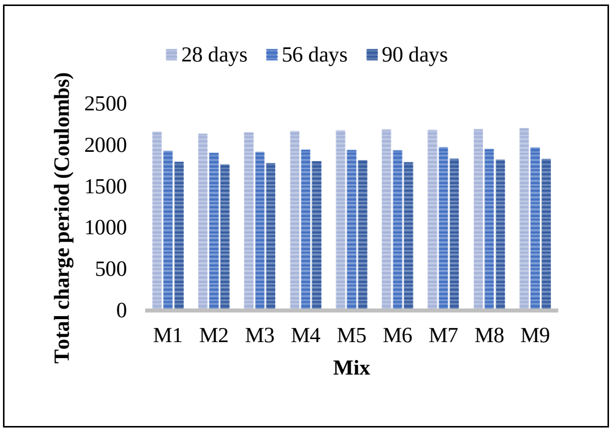

Fig. 5: Graphical representation of Rapid chloride penetration test (RCPT)

4.6 SEM

In order to better understand the composition, shape, and pore structure of concrete specimens, scanning electron microscopy (SEM) was used to examine their microstructure. Under typical curing, SEM analysis of conventional concrete reveals a well-distributed matrix of hydrated cement particles with a few unhydrated ones, indicating effective hydration. A similar microstructure, with efficient dispersion and equivalent pore distribution, can be seen in concrete that has been added to it. The microstructure remains the same in the absence of an admixture. However, when concrete surfaces are subjected to concentric hydrochloric acid curing, corrosive effects can be seen in the form of etching, increased porosity, and cementitious material leaching. Concentric sulphuric acid curing of concrete results in noticeable etching, widespread porosity, and considerable leaching, all of which represent the corrosive effect of sulphuric acid. For evaluating concrete durability and long-term performance in practical applications, SEM examination gives critical information on microstructural changes resulting from curing techniques and additive use. Figures 6, 7 and 8 shows the SEM images of M1, M4 and M6.

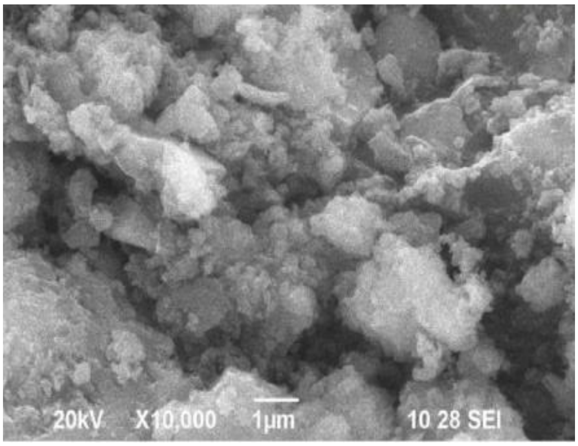

Fig. 6: SEM image of M1 mix

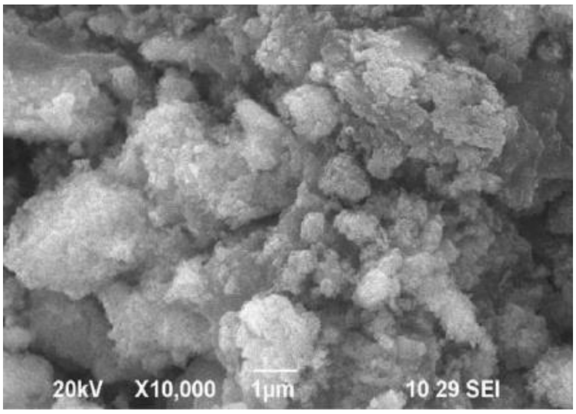

Fig. 7: SEM image of M4 mix

4.7 XRD

The minerals and other crystalline materials are identified using X-ray Diffraction (XRD). XRD can offer supplementary data to assist the fundamental data. The silica phase and calcite phase of the powdered concrete samples may be identified using the XRD analysis. A little powder sample is put into a sample container, and the surface is smoothed down. After that, the holder is put into the X-Ray diffractometer. The samples were scanned using an X-Ray diffractometer with Cu-K radiation at 40 kV / 20 mA, CPS = 1k, width 2.5, speed 2°/min, and scanned with an angle of 2 from 3 - 70°. During the course of the three seconds, the analysis is conducted in 0.04 degree steps.

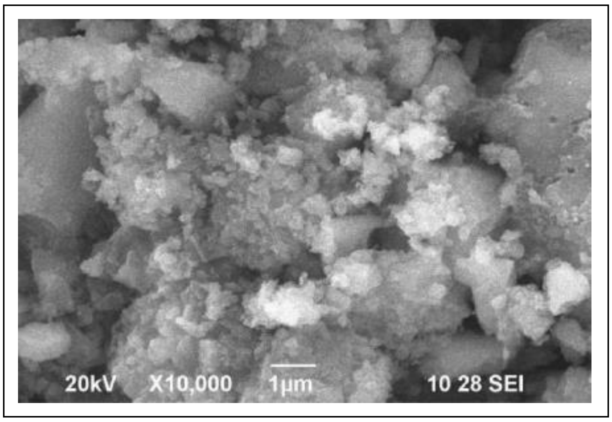

Fig. 8: SEM image M6 mix

The crystalline phases and mineral makeup of the concrete specimens from various mix designs and curing circumstances were further examined using X-ray diffraction (XRD) analysis. Understanding the concrete's long-term performance and durability is made easier thanks to this non-destructive method, which offers insights into the material's mineralogical makeup. In Conventional Concrete (Normal Curing), when conventional concrete is subjected to X-ray diffractometry (XRD) examination, it exhibits dominating peaks that are indicative of the presence of hydrated calcium silicate phases, such as calcium hydroxide ($Ca(OH)_2$) and calcium silicate hydrate (C-S-H). The production of solid crystalline phases and successful cement hydration are both indicated by these peaks. The XRD pattern of concrete with additive displays comparable prominent peaks connected to hydrated calcium silicate phases, indicating effective cement hydration (concrete with admixture, normal curing). The admixture does not considerably change the composition of the crystals.

The minerals and other crystalline materials are recognized using the X-ray Diffraction (XRD) technique. To support fundamental information, the XRD can offer further data. The XRD analysis is a useful technique for figuring out the silica phase and calcite phase of the powdered concrete samples. A small amount of powder sample is put into a sample container, and the surface is smoothed down. The holder is then inserted into the X-ray diffractometer. Then the samples are scanned using Cu-K radiation at 40 kV / 20 mA, CPS = 1k, width 2.5, speed 2° / min, and scanned with an angle of 2 from 3 - 70°. For three seconds, the analysis is conducted in 0.04-degree steps. Figures 9, 10 and 11 show the SEM images of M1, M4 and M6 mixes.

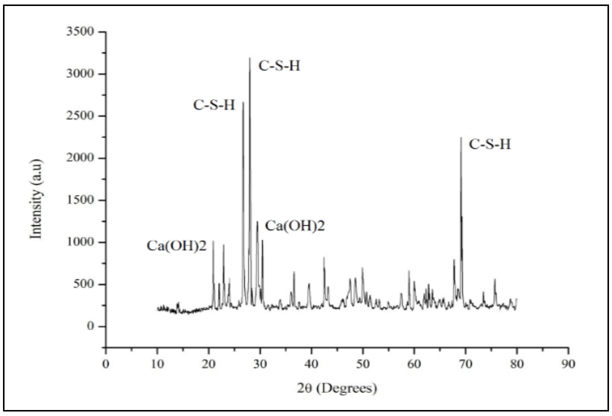

Fig. 9: XRD image M1 mix

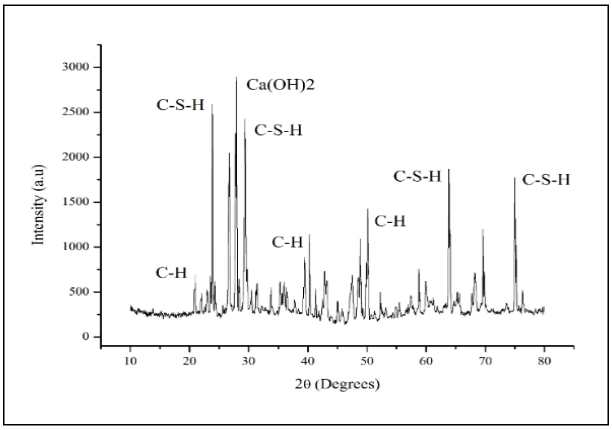

Fig. 10: XRD image M4 mix

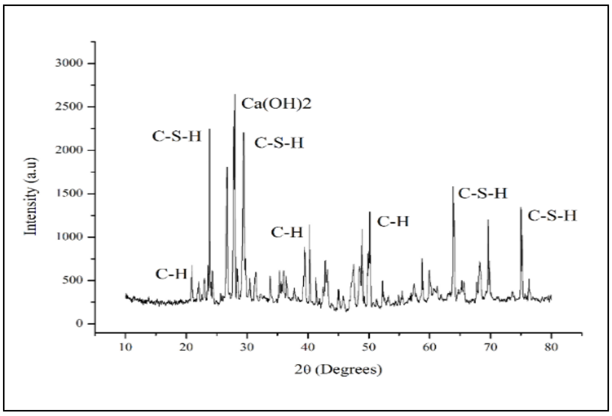

Fig. 11: XRD image of M6 mix

5. CONCLUSION

A thorough experimental examination of a variety of concrete mixtures, made using various curing techniques and additives, has provided important insights into the durability traits and microstructural alterations of these combinations:

The water absorption test results indicate that normal curing generally leads to lower water absorption over time compared to acid-based curing methods.

- The effective porosity test results indicate that normal curing generally leads to lower effective porosity values, suggesting better resistance to moisture penetration compared to acid-based curing methods over the tested durations.

- The acid resistance test results indicate that normal curing generally results in lower percentages of weight loss, suggesting better resistance to acid-induced deterioration compared to acid-based curing methods over the tested durations.

- The sorptivity results suggest that normal curing generally leads to slightly lower sorptivity values, indicating better resistance to moisture ingress compared to acid-based curing methods over the tested durations.

- The Rapid Chloride Penetration Test (RCPT) results indicate that normal curing generally results in lower total charge period values, suggesting better resistance to chloride ion penetration, a critical factor in reinforcing steel corrosion, compared to acid-based curing methods over the tested durations.

- SEM analysis of conventional concrete under typical curing reveals well-distributed hydrated cement particles, while admixture-added concrete exhibits a similar microstructure; in contrast, the acid curing leads to corrosive effects and increased porosity, emphasizing SEM's importance in assessing concrete durability.

- X-ray diffraction (XRD) analysis is essential for identifying concrete's crystalline materials. It aids in understanding mineral composition, confirming effective hydration, and assessing concrete durability.

- The sample results of scanned X-ray diffractometer utilizing Cu-K radiation at 40 kV / 20 mA, CPS = 1k, width 2.5, speed 2° / min, and scanned with an angle of 2 from 3 - 70°. For three seconds, the analysis is conducted in 0.04-degree steps and the results were analyzed.

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