INDIAN JOURNAL OF SCIENCE AND TECHNOLOGY

RESEARCH ARTICLE

OPEN ACCESS

Received: 10-03-2024

Accepted: 26-03-2024

Published: 16-04-2024

Citation: Reddy MVM, Krishnaiah S (2024) A Study on Durability and Microstructural Analysis for Macro Synthetic Fiber Reinforced Concrete with Supplementary Cementitious Materials. Indian Journal of Science and Technology 17(16): 1663-1671. https://doi.org/10.17485/IJST/v17i16.701

* **Corresponding author**.

mvmrphd@gmail.com

Funding: None

Competing Interests: None

Published By Indian Society for Education and Environment (iSee)

ISSN
Print: 0974-6846
Electronic: 0974-5645

A Study on Durability and Microstructural Analysis for Macro Synthetic Fiber Reinforced Concrete with Supplementary Cementitious Materials

M Venkata Maheswara Reddy[1]*, S Krishnaiah[2]

1 Research Scholar, Department of Civil Engineering, Jawaharlal Nehru Technological University, Anantapur (JNTUA), Ananthapuramu, 515002, Andhra Pradesh, India
2 Professor, Department of Civil Engineering, Jawaharlal Nehru Technological University, Anantapur (JNTUA), Ananthapuramu, 515002, Andhra Pradesh, India

Abstract

Background: Thermal cracking, delayed ettringite production and low tensile strength are three significant problems for high-strength concrete. **Objectives:** The current experimental study aims to determine the durability characteristics of concrete for application in pavements. To test how well the M40 grade of concrete absorbed chloride and water, the amounts of Supplementary Cementitious Materials (SCM) like Granulated Blast-Furnace Slag (GBFS) and fixed amounts of Fly Ash (FA) and Macro Synthetic Fiber (MSF) were optimized. **Methods:** One sample (S1) was made entirely of Ordinary Portland Cement (OPC) and five samples (S2, S3, S4, S5, and S6) made simply of SCMs, in which OPC was substituted with 20% FA+20% GBFS+1.5% MSF, 20% FA+25% GBFS+1.5% MSF, 20% FA+30% GBFS+1.5% MSF, 20% FA+35% GBFS+1.5% MSF, and 20% FA+40% GBFS+1.5% MSF, respectively, were cast in standard blocks with a volume of one cubic meter for this purpose. Field Emission Scanning Electron Microscopy (FESEM), Fourier-Transform Infrared Spectroscopy (FTIR), and X-ray Diffraction (XRD) investigations are used to examine the number of hydration products created at 28 days, which differ for different percentages of SCMs. **Findings:** Furthermore, 501, 520, 535, 565, and 590 coulombs are the measured Rapid Chloride Permeability Test (RCPT) values for the S2 to S6 samples. Similarly, the S1 sample is projected to have more than 2600 coulombs, showing a better endurance of samples based on SCMs. The microstructural characterization findings (i.e., XRD, FTIR and FESEM) suggested that GBFS and FA are promising SCMs for enhancing the strength and durability properties of the mix. **Novelty and applications:** This study validates the viability of using GBFS, FA, and MSF in pavement applications, yielding noteworthy environmental advantages and lowering dependency on OPC.

Keywords: Durability; Microstructural investigation; Macro synthetic fiber; Fly ash; Granulated blast furnace slag; Pavements

1 Introduction

Concrete is a composite material typically consisting of ordinary Portland cement (OPC), sand, gravel, chemical admixtures, and water. It is the second most consumed substance in the world, following water, and is more popular than all other materials[(1)]. Concrete possesses a satisfactory compressive strength. However, conventional concrete may be more suitable for engineering constructions that require vast sizes. Decreasing weight while maintaining high tensile strength is crucial for cost-effective structural design. Researchers developed fiber-reinforced concrete using supplementary cementitious materials (SCM)[(2,3)].

Examining the impact of SCM on the fresh and hardened properties and the durability of concrete has been extensively explored and advocated[(4)]. The researchers have admitted to utilizing granulated blast furnace slag (GBFS) and fly ash (FA) to improve the freshness and hardness of concrete[(2–8)]. When OPC, GBFS, and FA are mixed, the calcium-silicate parts in OPC clinker, such as C_3S and C_2S, start to dissolve. This creates calcium-silicate-hydrate (C-S-H) and calcium hydroxide (CH), also known as portlandite, as byproducts of secondary hydration[(9)]. Like OPC hydration, the interactions between GBFS and FA result in fresh calcium-silicate-hydride (C-S-H) gels with binding capabilities. Using GBFS and FA as substitutes for OPC improves the strength and longevity of concrete by forming a more compact matrix, thereby extending the lifespan of concrete structures. The primary elements influencing the inclination to recycle and utilize slag are economic and environmental considerations, as well as their impact on enhancing the characteristics of concrete. The global development of steel mills and thermal power plants is a significant focus. Huge depots are being constructed to hold GBFS and FA in plants as steel and power production rises, causing environmental harm. Using GBFS and FA instead of cement decreases cement usage and pollutants. Utilizing GBFS and FA in concrete eliminates depot running expenses and frees up space held by GBFS and FA, providing economic benefits[(10,11)]. In an experimental study, Kim et al.[(12)] examined the effects of FA and GBFS on microstructure, porosity, crystalline phase formation, and setting time. They discovered that these factors had less impact on strength development and a denser microstructure. XRD patterns show that while crystalline calcite was formed, calcium in fly ash did not contribute to creating the C-S-H bond.

Concrete has a low tensile strength despite its adequate compressive strength. Direct tension or bending-induced tension occurs in numerous structural components[(13)]. Thus, fiber-reinforced concrete has been suggested to address this deficiency in concrete. The incorporation of fibers into concrete started in the 1960s and has persisted till the present time. Commonly utilized fibers in concrete are steel, polypropylene, glass, and carbon fibers[(13–15)]. Using fibers in concrete often enhances tensile and flexural strength (FS) along with ductility. Using macro-synthetic fibers (MSF) in the concrete mixture is feasible and cost-effective to enhance strength and corrosion resistance while minimizing fracture extension. Researchers have said that utilizing MSF can enhance several properties such as toughness, modulus of rupture, bond strength, impact resistance, the performance of synthetic fibers, long-term tensile strength, modulus of elasticity, spalling behaviour, abrasive erosion resistance, fire resistance, and post-cracking behaviour. It can decrease plastic shrinkage, crack propagation, fractal dimension, and fracture toughness. MSF is said to enhance the stability of concrete in hot temperatures.

1.1 Research Gaps

Previous literature includes multiple experimental studies on the impact of different fibers and FA on concrete. This paper examines the combined influence of MSF, GBFS and FA on durability and microstructural performance, which has yet to be studied. MSF possesses attributes like a high melting point, exceptional thermal stability, and flame resistance, which can enhance the diverse qualities of concrete. Moreover, the fibers' high tensile strength enhances the strength and ductility of concrete. The study utilized MSF at fixed concentration of 1.5% by volume. The GBFS content ranged from 0 to 40% by weight of OPC, in addition to a constant 20% FA. To the author's knowledge, the combined use of MSF, GBFS, and FA has yet to be studied in terms of durability. Different types of microstructural studies were carried out, including X-ray diffraction analysis (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray (EDX).

1.2 Research Significance

This study makes significant contributions to the corpus of literature. It closes a considerable research gap by thoroughly examining how GBFS, FA, and MSF affect durability and microstructural characteristics. Adding these SCM to fiber-reinforced concrete is part of the expanded analysis of this work. This has implications for creating high-performance building materials and environmentally friendly building methods. These contributions broaden the comprehension of sustainable building materials, providing practitioners and scholars with insightful information.

2 Materials and Methodology

In this study, OPC 53 grade (IS: 269-2015)[16,17] with a specific gravity of 3.14 was used. The OPC took 65 minutes to set up initially and 220 minutes to set up in the end. The soundness was 0.6 mm, and its specific surface area was 295 m^2/kg. % of GBFS from 20 to 40 and FA with a fixed dosage of 20 % is replaced with OPC by weight for making SCM concrete. The GBFS and FA were sourced from the JSW, Hyderabad, and the Ramagundam thermal power plant. GBFS and FA correspond to specific gravities of 2.86 and 2.17. This investigation utilised GBFS with a Blains-specific surface area of 388 m^2/kg (IS 16714 & 3812)[18,19]. The OPC, FA and GBFS chemical properties were examined by X-ray fluorescence and are displayed in Table 1. A maximum size of 20 mm of crushed stone aggregate with a specific gravity of 2.64, bulk density of 1.68 kg/liter, and water absorption of 0.26% were used. Zone-II river sand, with a specific gravity of 2.58 and a bulk density of 1.82 kg/liter, was used as a fine aggregate following IS 383-2016 requirements[20]. Macro-synthetic fibers (MSF) with a tensile strength of more than 80 MPa, a modulus of elasticity of more than 7000 MPa, and an aspect ratio of 36 were acquired from local vendors and used to create fibre-reinforced concrete with GBFS and FA. Master Glenium Sky 8609 (IS 9103)[21] is a superplasticizer obtained from M/s. Master Builders Solutions India Pvt. Ltd. was utilized in this investigation.

Table 1. Constituents of OPC, GBFS and FA

Elements (wt.%)	OPC	GBFS	FA
CaO	60.76	37.34	15.98
Al_2O_3	5.01	14.42	22.3
SiO_2	23.4	37.73	60.2
Fe_2O_3	4.46	1.8	0.17
MgO	3.95	8.71	1.15

The concrete mix design was developed following IS 10262-2019[22]. The mix design upholds a consistent water-binder ratio of 0.36, a binder content of 450 kg/m^3, a river sand content of 758.40 kg/m^3, a coarse aggregate content of 1026.80 kg/m^3 and an admixture content of 0.9 kg/m^3. Based on the assumption that the fresh concrete contains 1.5% entrapped air, the volume of aggregates for the design concrete was determined. The mix proportion (kg/m^3) are displayed in Table 2.

Table 2. Mix Proportions (kg/m^3)

Mix ID	W/C	Cement (kg/m^3)	Macro synthetic fibres (MSF) %	Cementitious material (kg/m^3)		Fine Aggregate (kg/m^3)	Coarse Aggregate (kg/m^3)	Admixture (kg/m^3)
				GBFS	FA			
SP1	0.36	450	1.5	0.0	0.0	758.40	1026.80	0.90
SP2	0.36	270	1.5	90	90	758.40	1026.80	0.90
SP3	0.36	248	1.5	112	90	758.40	1026.80	0.90

Continued on next page

Table 2 continued

SP4	0.36	225	1.5	135	90	758.40	1026.80	0.90
SP5	0.36	203	1.5	157	90	758.40	1026.80	0.90
SP6	0.36	180	1.5	180	90	758.40	1026.80	0.90

2.1 Durability Properties

Using the rapid chloride penetration test (RCPT), the specimen's resistance to salt assault was assessed at 28 days of age, following ASTM C1202. Three specimens with a thickness of 50 mm and a diameter of 100 mm were subjected to a 60 V potential for six hours. The total charge that flowed through the specimens was determined to assess the mixture's fixed concentration of 1.5% by volume. The GBFS content ranged from 20 to 40% by weight of OPC, in addition to a constant 20% FA measured chloride permeability, respectively.

To determine the absorption of concrete, a water absorption test was run on one face of 100-mm cubic specimens under water. Following BS EN-1811(Part 2), each specimen's measured absorption will be determined by expressing the mass gain from immersion as a percentage of the specimen's dry mass.

2.2 X-ray diffraction analysis (XRD)

X-ray diffraction analysis (XRD), a non-destructive examination, was carried out using a Philips 1140 to identify the elements in a specimen. With a step size of 0.01, the continually evaluated range was 10–70°. Selected sample types were synthesized in powder form under 40 kV/30 mA working conditions and were tested at the central analytical laboratory, BITS-Pilani, Hyderabad Campus.

2.3 Scanning electron microscope (SEM)

The optimized specimens were broken into small pieces after being cured for 28 days. The about 6.3 mm chunks with fibers and aggregate were coated in gold-palladium and examined under an electron microscope. SEM (APEROS, FEI) were used for the investigations. In order to see how GBFS and FA affected the binder and aggregate in this investigation, the two materials were placed in the center of the SEM to display the morphology. In order to ascertain the elemental composition, EDX analysis was carried out at various sample locations and were tested at the central analytical laboratory, BITS-Pilani, Hyderabad Campus.

2.4 Fourier transform infrared spectroscopy (FTIR)

FTIR spectroscopy was used to examine the influence of MSF, FA and GBFS-based concrete at the central analytical laboratory, BITS-Pilani, Hyderabad Campus. The selected samples were blended at a weight ratio 1:100 to create pellets. The IRAffinity 1S(SHIMADZU) FTIR spectrometer was used to scan the mid-infrared spectra ranging from 500 to 4000cm^{-1}.

3 Results and Discussions

3.1 Rapid chloride penetration test (RCPT)

The Rapid Chloride Permeability Test, often known as the RCPT, is a standard procedure utilised to evaluate the durability of fiber-reinforced concrete with SCM by measuring the amount of chloride ions that can penetrate the material. To do this, a solution of 0.3 M sodium hydroxide and a solution of 3% sodium chloride is applied to the concrete sample, and then a 60 V direct current is passed through an electrically conductive wire for six hours. The values in coulombs for the mix proportions added to selected samples are then determined. Every single day for the next 28 days is approached in the same manner. Using the RCPT, the chloride ion penetration depths into concrete discs are depicted in Figure 1. The figures illustrate the differences in penetration when the Water-Binder (W/B) ratio is 0.36, and the percentages of mineral additive substitution in OPC are altered[(23)]. After twenty-eight days, the mixture with zero per cent replacement can achieve a substantial degree of chloride ion penetration. Which rises to a moderate level after fifty-six days.

In comparison, the mix S2 that contains a replacement of 20% FA, 20% GBFS, and 1.5% MSF demonstrates a low level of chloride ion penetration after 28 days have passed. At the 28-day mark, the introduction of chloride ions into concrete is restricted, which indicates the high level of efficacy achieved by the considerable replacement of cementitious materials in OPC. Replacing constant 20% FA with composite cementitious materials that contained 25%, 30%, 35%, and 40% of GBFS led to moderate and low levels of chloride ion penetration in the specimens. Overall, the S2 mix successfully produced positive results across all grades.

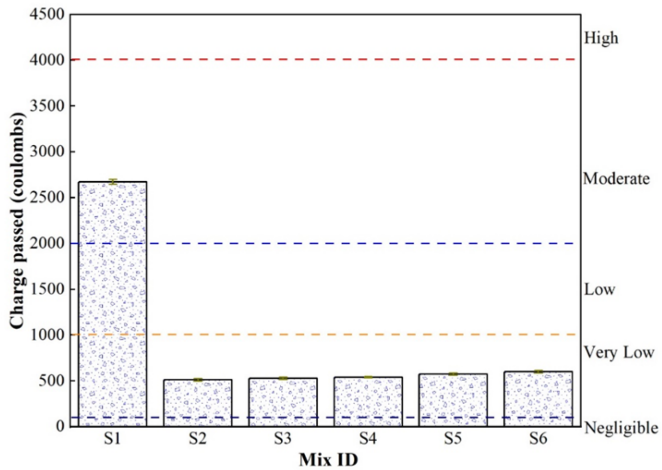

Fig 1. RCPT values of S1 to S6 samples

3.2 Water Absorption

For the purpose of determining the permeability/absorption of a cube size of 100mm, an RCPT test was carried out in conjunction with a water absorption test. Several well-known elements can contribute to high durability and postpone corrosion. Two of these aspects are low permeability and ion chloride penetration rate. A representation of the water penetration depth data at 28 days is shown in Figure 2. These findings provide evidence that the RCPT values are accurate. There is a more significant difference between the water absorption depths of S4 and that of S1. Compared to S1, the water penetration depths of the following samples, denoted by S2, S3, S5, and S6, are lower. The variable water absorption might be ascribed to the increase in porosity when the concentration of MSF is further increased. Pore obstruction and decreased capillary porosity are likely to be blamed for the decrease in water depth that may be observed in Substance S2. The results of the durability tests that were carried out in this research are supported by this outcome [6]. There does not appear to be any noticeable difference between S2 and S1 regarding water absorption.

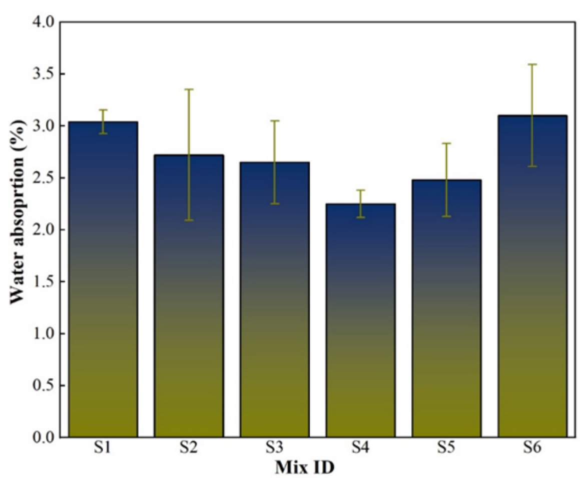

Fig 2. Water absorption (%) of S1 to S6 samples

3.3 XRD analysis

XRD analysis was performed on specimens to show how adding MSF to a concrete mixture affected phase changes. Figure 3 displays the findings of the chosen samples after 28 days. The primary peaks are silicon dioxide SiO_2, calcite $Ca(CO)_3$, and portlandite $Ca(OH)_2$. The results indicate that adding MSF had no discernible effect on $Ca(CO)_3$ or $Ca(OH)_2$. The XRD figure demonstrates that following the activation of the antacid system and the expansion of the GBFS, the mullite and quartz tops of the initial fly debris were still present. The following are the causes of the peaks that can be found at different angles:

- 27° are caused by calcium hydroxide ($Ca(OH)_2$).
- 54° are caused by calcium oxide (CaO)
- 28° and 32° are caused by tricalcium silicates (alite) (C_3S).
- 47° are caused by dicalcium silicates (blite) (C_2S).
- 23° and 28° are caused by calcium carbonate ($CaCO_3$).
- 21° and 24° are caused by magnesium oxide (MgO);
- 21° are caused by calcium sulphate ($CaSO_4$).

Therefore, Portlandite is the primary hydrated product in the XRD pattern. Alite is the primary chemical that reacts in cementitious systems [4]. The foregoing observations are further validated using FTIR analysis of the materials soaked for 28 days.

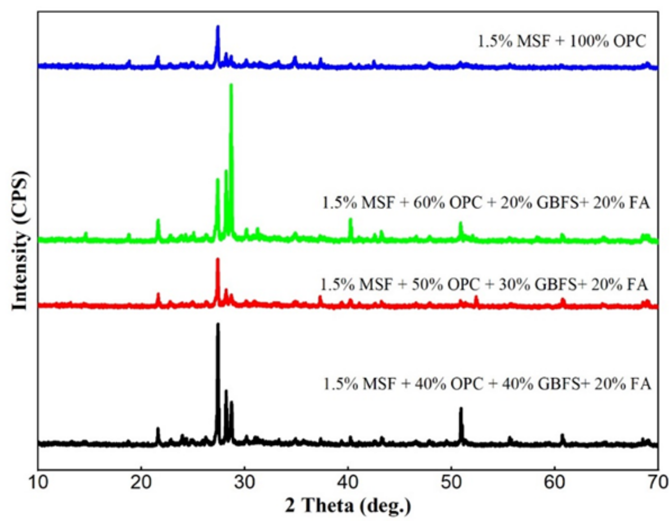

Fig 3. XRD analysis

3.4 FTIR analysis

An FTIR analysis was conducted on the concrete powder sample to determine the functional groups of the products generated during hydration. Figure 4 displays the FTIR spectra of the samples after 28 days of hydration in transmittance mode. During the experiment, identical infrared bands were seen in all samples with consistent wave number values, albeit with varying intensities. The IR peak at 3646 cm^{-1} corresponds to the O–H bond stretching in $Ca(OH)_2$. The peak at 1660 cm^{-1} is caused by the v_2 bending of the absorbed and bound water (H_2O). The peak at 1420 cm^{-1} is caused by the ν_3 stretching of C-O in $CaCO_3$. The peak at 1448 cm^{-1} is caused by the Si-O stretching of calcium silicate hydrate (CSH). The peak at 770 cm^{-1} is caused by the ν_2 stretching of C-O in $CaCO_3$. The 3646 cm^{-1} and 976 cm^{-1} peaks suggest hydrated products such as $Ca(OH)_2$ and C-S-H, respectively. The two peaks are crucial for evaluating the hydration of the cementitious phases. Upon thoroughly examining Figure 4, it is evident that the peaks at 3646 and 980 cm^{-1} exhibit greater intensities in OPC compared to other samples. The increased presence of $Ca(OH)_2$ and C-S-H in the conventional sample, which consists of 100% OPC, is causing this phenomenon.

FTIR and XRD analysis results show that using SCM is highly successful in lowering the early degree of hydration and reducing the mass concrete's peak core temperature. Additionally, FESEM analysis is performed to validate the results of FTIR and XRD [4].

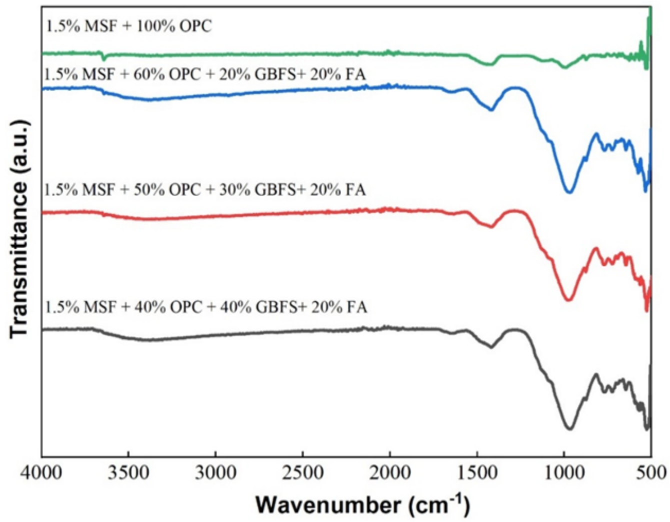

Fig 4. FTIR analysis

3.5 SEM Analysis

FESEM analysis helps determine the kind and amount of product production and porosity in the system. Figure 5 displays the microstructures of the fracture surfaces of concrete samples with various SCM that were cured for 28 days. Upon examining the fracture surfaces of 28-day hydrated concrete samples, it was noted that the microstructure of Figure 5**a** is denser and has a higher concentration of hydrated products than the other samples. The Figure 5**b** exhibits greater product production and relatively lower porosity than the Figure 5**d** sample. The Figure 5 **c**, consisting of 20% FA and 20% GGBS, shows the lowest product production and increased porosity.

The phenomena can be explained by the reduced OPC concentration in samples compared to conventional sample shown in Figure 5**a**.

The microstructural study corroborates the findings from the XRD and FTIR investigations. FESEM study confirms that using SCM decreases product formation (hydration degree), resulting in a lower peak core temperature in mass concrete. XRD, FTIR, and FESEM observations show that the presence of SCM slows the hydration rate at an early stage. However, they react gradually over a more extended period, leading to a substantial enhancement in strength and durability, as detailed in the subsequent sections [4].

4 Conclusions

This study presents concrete's durability and microstructural behaviour with SCM and a volume fraction of MSF. The exposure duration is 28 days, and the parameters considered assess the RCPT and water absorption tests. The investigation also presents a microstructural analysis using XRD, FTIR, SEM, and EDAX. Drawing from the principal findings, it concludes that:

- The durability of SCM & MSF-based samples falls into the very low category, according to RCPT values obtained at 28 days. In contrast, the concrete constructed with 100% OPC without SCM & MSF falls into the moderate category. This suggests that using SCMs & MSF can significantly increase the longevity of M40 grade concrete.
- The results of the water absorption test prove the validity of RCPT values by showing that all water absorption for SCM & MSF-based concrete is lower than that of conventional concrete, which can be related to the pore-blocking effect of MSF fibres.
- The SCM & MSF-based system shows less hydrated product development in 28 days of cured samples, according to the XRD and FTIR studies. This is evident because the binders have less hydration, which supports the hydration phenomenon.
- The SEM pictures reveal a continuous matrix that denotes a dense microstructure and a homogenous matrix for secondary CSH, unreacted FA, and GBFS. The EDS analysis supplemented the experimental findings on the strength evolution of each sample and the SEM pictures.

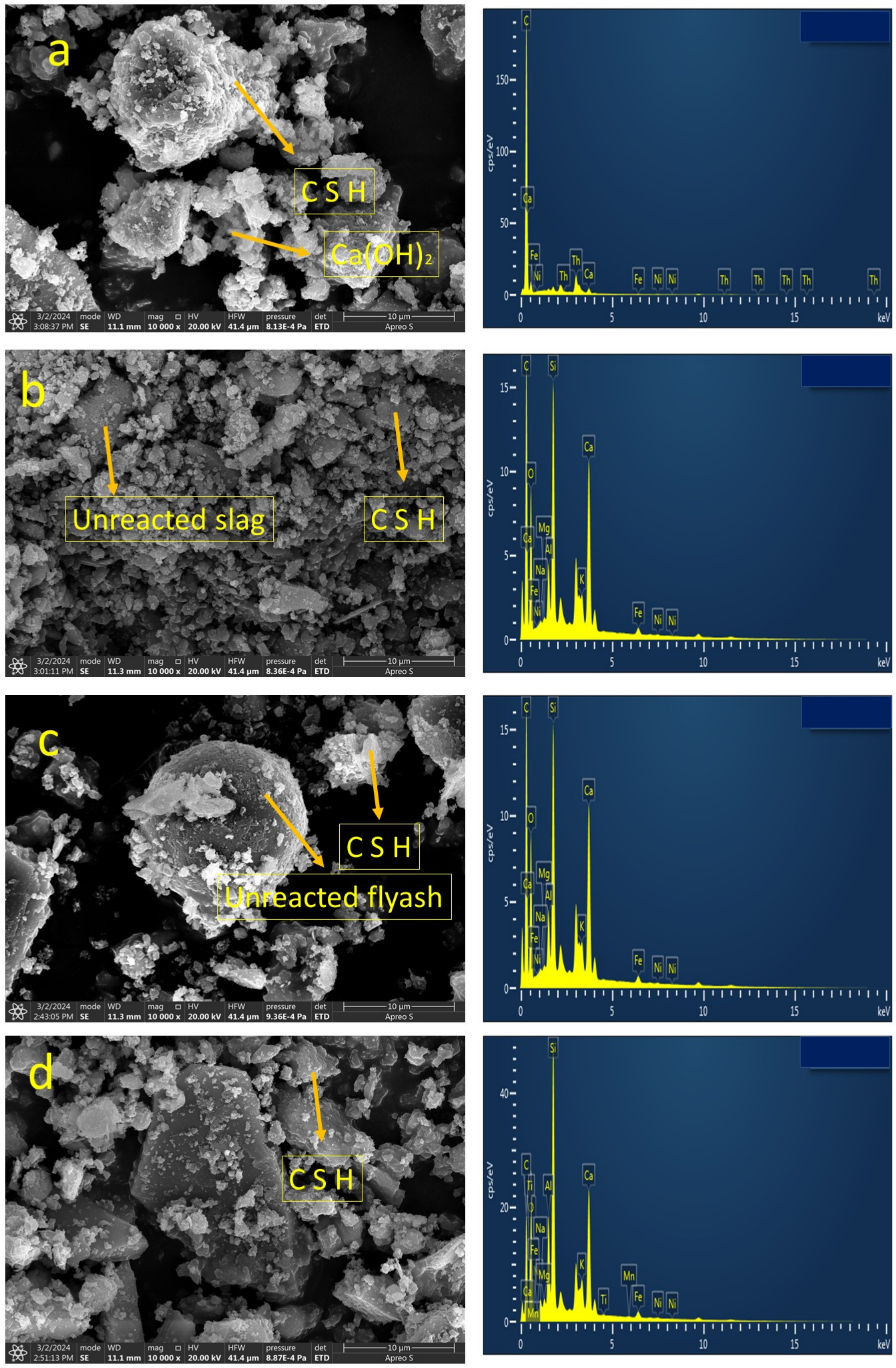

Fig 5. SEM analysis (a-100% OPC; b-20%FA+20%GBFS+1.5%MSF; b-20%FA+30%GBFS+1.5%MSF; d- b-20%FA+40%GBFS+1.5%MSF

The cost and convenience of operation of implementing SCM & MSF-based M40 grade concrete will have a significant impact on the industry. Utilizing SCM also conserves natural resources by reducing the issues associated with landfilling, air and water pollution, and raw cement material waste.

5 Recommendations

Based on the current study findings, several problems and possibilities for future research in this area have arisen. Researchers are invited to conduct pilot studies on the particular uses of fibre reinforced concrete including FA and GBFS for various civil engineering applications. Furthermore, as part of this research, life cycle assessments (LCAs) have to be done to quantify the environmental effect of FA and GBFS in building projects, giving critical insights into their sustainability benefits.

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