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Research Article

Effect of elevated temperature on concrete incorporating zeolite, silica fume and fly ash as replacement for cement

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Abstract

In this study, the effect of utilizing zeolite and industrial by-products, silica fume, and Class C fly ash, as partial replacement for cement is investigated at room and elevated temperatures of 200°C, 400°C, and 600°C. Four different high-strength concrete mixes of M50 concrete grade with varying proportions of zeolite, silica fume, and Class C fly ash are tested and observed for workability, compressive strength, and microstructural characteristics using Scanning Electron Microscope analysis, and the optimum mix has arrived. The results indicate that the addition of zeolite, silica fume, and fly ash resulted in lesser early-age strength development but the strength after 28 days of curing is found satisfactory and the desired target strength is achieved. Temperature rise resulted in degradation of concrete properties for both conventional and optimum mix but the concrete cubes with 10% of zeolite, 7.5% of silica fume, and 10% of fly ash have higher retainment of strength at elevated temperatures compared with conventional mix. The results from the microstructural study are found to correlate with strength test results. Incorporation of 10% zeolite, 7.5% silica fume, and 10% fly ash improved mechanical and microstructural characteristics at room and elevated temperature, however, the replacement is effective for 28 days of curing.

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1. Introduction

The building industry has been investigating sustainable alternatives to Portland cement because of its negative environmental impact and large carbon dioxide emissions throughout its manufacturing process. Supplementary Cementitious Materials (SCMs) such as fly ash, zeolite, and silica fume have garnered attention in concrete mixes as a way to improve the material's physical properties and reduce its environmental effect. This introduction examines the use of these SCMs in the specific context of elevated temperatures, which is a crucial consideration for the lifetime and functionality of concrete structures in high-temperature industrial environments or fire scenarios.

Zeolite, a naturally occurring mineral with a high concentration of aluminium oxide (Al_2O_3) and reactive silicon dioxide (SiO_2), is a pozzolanic substance that may strengthen cement pastes by pozzolanic interaction with calcium hydroxide ($Ca\ (OH))_2$. It reacts more readily with fly ash and silica fume, and it works best in mixes with a lower water to cementitious material ratio [1]. Zeolite mixtures have shown reduced heat of hydration [2], lower drying shrinkage, and increased resistance to sulphate attack and the alkali-silica reaction in

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comparison to fly ash blends [3]. The incorporation of zeolite into concrete leads to the development of a material that possesses enhanced interfacial microstructure characteristics between the aggregate and the cohesive cement paste [4]. However, they may also need more water due to high surface area [5] and can be less workable [6, 7]. Concrete containing zeolite is observed to have a satisfactory performance at elevated temperatures [8]. Research conducted in the past suggests that the recommended percentage of cement replacement for zeolite is 20%, 15% and 10% [5].

When silicon and ferrosilicon alloys are made, minute amorphous silicon dioxide particles are released into the air, forming silica fume. Because of its potent pozzolanic reactivity and capacity to modify pore structure, it is frequently used to improve the mechanical characteristics and durability of concrete [9]. Furthermore, it has been shown that adding nano silica fume to concrete improves post-fire compressive strength while also greatly reducing temperature shrinkage deformation and mass loss at high temperatures [10]. The optimized percentage for replacing cement with silica fume is 7.5-10% in combination with 10-15% of zeolite and 10% of metakaolin [11]. Another SCM with pozzolanic properties is fly ash, which is produced as a byproduct of burning coal in power plants. It contains both silicon dioxide and calcium oxide, with the latter contributing to its cementitious properties. Fly ash will notably reduce the heat of hydration in concrete and improve its long-term strength development. The concentration of calcium in fly ash may influence its reactivity; fly ash with low calcium content will more obviously slow down the evolution of heat [12]. When exposed to high temperatures, concrete containing various SCMs may function differently. Research has indicated that zeolite and granulated blast furnace slag (GBFS) mortars outperform fly ash and silica fume mortars when it comes to heat resistance [13]. At higher temperatures, the formation of a greater quantity of secondary hydration products leads to improved mechanical properties in concrete that contains zeolite [14]. Moreover, SCMs may affect the mineralogy of cement pastes. Zeolites form during adiabatic curing when combined with a high fly ash content, can immobilize sodium and increase the material's overall stability [15].

In conclusion, adding fly ash, zeolite, and silica fume to concrete mixes in place of some of the cement is a practical way to improve the material's performance and sustainability, especially at high temperatures. The selection and proportioning of these SCMs have a notable impact on the mechanical properties, durability, and thermal behaviour of the finished concrete [16, 17, 18, 19]. Based on the literature review, it is noticed that the studies examining the combined effect of zeolite, silica fume, and fly ash at elevated temperatures are limited and scanty. Hence, the present study focused on studying the performance of concrete incorporating zeolite, silica fume, and fly ash at room and elevated temperatures.

2. Research Significance

The addition of class C fly ash may help the concrete to attain compressive strength in the 28-days curing period. Silica fume and class C fly ash as the cement replacement can enhance the microstructure of the concrete. Examining the properties of the high-strength concrete incorporated with zeolite, silica fume, and class C fly ash can provide advancement in the construction. The endurance of the concrete during the fire accident may improve in the building. The major objectives of the present study are:

- To find the optimum mix for concrete incorporating zeolite, silica fume, and class C fly ash as a replacement for cement
- To study the effect of elevated temperature on mechanical and microstructural properties of concrete incorporating zeolite, silica fume, and class C fly ash.

3. Materials and Mix Design

The preliminary investigation is performed to study the properties of materials utilized in the casting of concrete specimens. Ingredients include coarse aggregate, fine aggregate, cement, zeolite, fly ash, silica fume, and superplasticizer.

3.1. Coarse Aggregate

Within the domain of construction and civil engineering, coarse aggregate denotes a class of granular materials employed in concrete and various other structural applications. These materials commonly encompass sizable particles, including crushed stone, gravel, or recycled concrete, which are combined with cement and fine aggregates to produce concrete mixes. The physical properties of the coarse aggregate are determined as per IS 2386 – part 3 (1963) [20] and IS 383 (2016) [21].

3.2. Fine Aggregate

The process of crushing solid granite stone results in the production of manufactured sand. This crushed sand is characterized by a cubic shape with smoothed edges, having undergone comprehensive washing and sorting procedures, rendering it appropriate for use in construction. The particle size of M-Sand not exceeding 4.75 mm is used in the present study. The shape of the M sand is granular and the determined properties of M-Sand as per IS 2386 – part 3 (1963) [20] and IS 383 (2016) [21] are given in Table 1. Table 1 gives the properties of coarse aggregate and fine aggregate used for the present study.

Table 1. Properties of coarse aggregate and fine aggregate

Material	Properties	Values
Coarse aggregate	Specific gravity	2.74
	Fineness modulus	6.57%
	Water absorption	0.5%
Fine aggregate	Specific gravity	2.35
	Fineness modulus	3.33
	Water absorption	0.5%

3.3. Cement

When combined with water, cement acts as a binding agent for coarse and fine aggregate. The manufacturing process of cement primarily relies on calcium-rich limestone as its fundamental raw material. Upon the reaction between cement and water, water-resistant and chemically durable mineral hydrates are formed. Cement is classified into various grades according to its fineness, with the current study utilizing OPC 53 cement.

3.4. Zeolite

Zeolite is a naturally occurring or synthetic mineral that has been increasingly used in concrete as an alternative supplementary cementitious material. It is a group of hydrated aluminosilicate minerals that possess unique properties, making them suitable for various applications, including concrete production. The combined percentage of SiO_2, Al_2O_3, and Fe_2O_3 is about 83% according to the chemical composition of zeolite which is helpful in the pozzolanic behaviour.

3.5. Silica Fume

The addition of silica fume improves the concrete's compressive strength and aids in void filling. Silica fume helps in reducing the water absorption of the zeolite used in the concrete. Silica fume is also rich in SiO_2 which is helpful in the pozzolanic behaviour.

3.6. Class C Fly Ash

The use of Class C Fly Ash lowers the heat produced during the curing process of concrete, hence reducing the possibility of heat-induced cracking, particularly in big concrete pours. Fly ash addition with zeolite helps in reducing the water absorption by zeolite. Class C fly ash is also high in SiO_2 and Al_2O_3. The chemical composition and properties of cement, zeolite, silica fume, and fly ash are given in Table 2 and Table 3, respectively. Fig. 1 gives the supplementary cementitious materials used for the present study.

Table 2. Chemical composition of cement, zeolite, silica fume and fly ash

Oxides (Percentage)	CaO	SiO_2	Al_2O_3	Fe_2O_3	K_2O	Na_2O	MgO	SO_3	Loss on ignition
Cement	62.34	23.45	4.24	4.32	0.47	0.27	1.12	0.47	3.32
Zeolite	1.68	66.98	14.01	1.52	1.5	2.10	1.3	0.4	10.3
Silica fume	-	94.12	1.21	0.62	-	-	0.9	0.04	1.5
Class C Fly ash	1.5	62	22.5	2.3	-	1.5	3.4	4	2.1

Table 3. Properties of cement, zeolite, fine aggregate and coarse aggregate

Material	Properties	Values
Cement	Specific gravity	3.18
	Initial setting time	47 minutes
	Final setting time	480 minutes
Zeolite	Specific gravity	2.2
Silica fume		2.22
Class C Fly ash		2.3

a) Zeolite b) Silica fume c) Class C Fly ash

Fig. 1. Cementitious materials used for the present study

3.7. Mix Design

The concrete specimens are cast with the designed concrete mix to achieve the required target strength of 50 MPa as per IS 10262 (2019) [22]. The mix design as given in Table 4 is used for casting the conventional concrete without replacement for cement.

The zeolite of 10%, silica fume of 7.5 %, and class C fly ash of 10% are replaced with the cement and other materials are provided as per the conventional concrete. Concrete specimens are cast with three different mixes with varying percentages of zeolite, silica fume, and fly ash. Table 5 represents the various design mixes for the concrete incorporating zeolite, silica fume, and class C fly ash. M50Z10 indicates M50 concrete grade with 10% of cement replaced by zeolite on a weight basis. Similarly, M50SF indicates M50 concrete grade with 7.5% and 10% of silica fume and fly ash replaced for cement.

Table 4. Mix design for the conventional concrete

Description	Composition (kg/m^3)
Cement	450
Fine Aggregate	803
Coarse Aggregate	1101
Water	160
Admixture	2.82

Mix 3 designated as M50ZSF represents the concrete mix with 10% of zeolite, 7.5% of silica fume, and 10% of fly ash for cement replacement. In mix 2 and mix 3, silica fume and fly ash are combined to compare the behaviour of concrete with the existing combination of supplementary cementitious materials without and with concrete containing zeolite.

Table 5. Proportion of concrete ingredients for varying percentage of zeolite, silica fume and fly ash

Composition (kg/m^3)	Mix 1 M50Z10	Mix 2 M50SF	Mix 3 M50ZSF
Cement	405	371.25	326.25
Zeolite	45	0	45
Silica fume	0	33.75	33.75
Class C Fly ash	0	45	45
Fine aggregate	803	803	803
Coarse aggregate	1101	1101	1101
Water	160	160	160
Admixture	2.82	2.82	2.82

4. Methodology

The mechanical specifications of M50 concrete grade are determined by Indian standards. A 100 mm diameter and 200 mm height concrete cylinder and a 100 × 100 × 100 mm concrete cube are cast, cured, and tested to determine the strength properties of concrete. Fig. 2 gives the cast specimens, and the concrete is prepared in the same way as regular concrete but with additional ingredients in addition to cement. The optimal combination for enhanced mechanical features at room temperature is found based on the outcomes of the experimental tests.

Fig. 2. Cast specimens

Optimum mix is determined by the combination of supplementary cementitious materials by modifying the existing combination of cementitious materials in concrete containing zeolite. Further specimens are cast with optimum mix to determine the effect of varying concrete ingredients on the fire endurance of the concrete specimens. Heating of concrete cubes and cylinders is performed in the furnace at the rate of 10°C/minute to reach the desired target temperature of 200°C, 400°C, and 600°C and the specimens are heated for one hour under steady state after reaching maximum temperature. The mechanical properties including compressive strength and split tensile strength are determined after cooling of specimens to room temperature. The results are compared with conventional concrete to determine the effectiveness of utilizing zeolite, silica fume, and class C fly ash in concrete for improved fire resistance.

5. Results and Discussions

5.1. Testing at Room Temperature

The workability of concrete mixes is determined using slump cone test as a preliminary investigation. The slump values obtained for conventional concrete, mix 1, mix 2 and mix 3 are 100 mm, 90 mm, 85 mm and 84 mm respectively.

5.1.1 Compressive strength

Compressive strength is one of the important mechanical characteristics and it gauges how much axial compression that concrete can bear before cracking. Fig. 3 gives the 7-, 14- and 28-day compressive strength for four different mixes determined at room temperature.

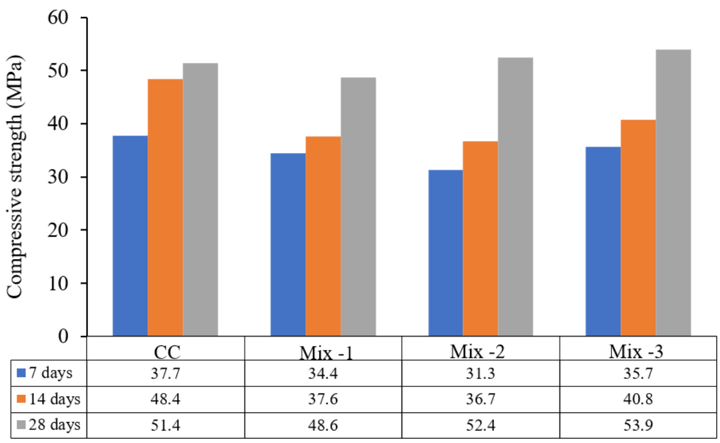
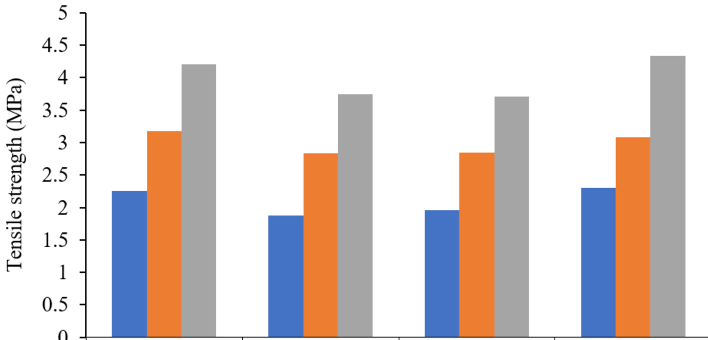


Fig. 3. Compressive strength for concrete mixes with varying percentage of zeolite, silica fume and fly ash (CC – Conventional concrete)

The target strength of 50 MPa is chosen for this study, and the conventional concrete has a strength of 51.4 MPa whereas mix 1 has 48.6 MPa, mix 2 has 52.4 MPa, and mix 3 has 53.9 MPa after 28 days of curing period. When compared to the conventional concrete, compressive strength for mix 1, mix 2, and mix 3 decreased by 5.4%, and increased by 2% and 4.9%, respectively. The compressive strength of all three concrete mixes with zeolite, silica fume, and fly ash decreased initially (7 and 14 days) and upon curing, the required target strength is reached. The compressive strength of Mix 1 is lower compared to other mixes, and the results are in correlation with studies performed by other researchers [23, 24, 25]. The reactivity of zeolite increases with the increase in the days of curing, and the reactivity depends upon the water-cement ratio [23]. Adding fly ash and silica fume resulted in the development of target strength comparable to conventional concrete. The compressive strength of concrete mix with silica fume, fly ash, and zeolite increases upon curing age due to secondary hydration induced by the addition of zeolite. The continuous hydration results in the development of higher strength [24] and hence the strength value is higher for Mix 3 under a constant water-cement ratio.

5.1.2 Split Tensile Strength

Split tensile strength testing offers important information on the tensile behaviour of concrete, information that is necessary for the design and assessment of different structural components, such as pavements, slabs, and beams. It also plays a crucial role in the building industry's quality assurance and control procedures. The split tensile result of the conventional concrete is shown in Fig. 4.



	CC	Mix -1	Mix -2	Mix -3
7 days	2.26	1.88	1.96	2.3
14 days	3.18	2.84	2.85	3.08
28 days	4.21	3.74	3.71	4.34

Fig. 4. Split tensile strength for concrete mixes with varying percentage of zeolite, silica fume and fly ash (CC – Conventional concrete)

The 28-day split tensile strength of the conventional concrete has reached 4.21 MPa, mix 1 has reached 3.74 MPa, mix 2 has attained 3.71 MPa and mix 3 has attained 4.34 MPa. The percentage reduction in split tensile strength for mix 1 and mix 2 is 11% and 11.8% when compared to the conventional concrete and mix 3 has gained the increase in strength value by 3.1%. From the test results, it is noted that the optimum mix is Mix 3 with compressive strength and a split tensile strength greater by 4.9% and 3.1%, compared with conventional concrete. Concrete cubes and cylinders are further cast with the arrived optimum mix and the specimens are tested after exposure to temperatures of 200°C, 400°C, and 600°C. The effect of temperature rise on concrete is examined through percentage of weight loss, compressive strength, split tensile strength, and microstructural characteristics. The effectiveness of utilizing alternative cementitious materials in improving the fire resistance of concrete is studied by comparing the performance of concrete containing zeolite, silica fume, and fly ash with conventional concrete.

5.2. Testing at Elevated Temperature

5.2.1 Weight Loss of Specimens

Fig. 5 gives the percentage of weight loss observed in both conventional concrete mixes and mixes with zeolite, silica fume, and fly ash. The percentage of weight loss for the conventional concrete after exposure to 200°C, 400°C, and 600°C is 0.4%, 4.9%, and 6.3% respectively. The percentage of weight loss observed in the concrete with optimum mix is

0.23%, 4.4%, and 5.35% respectively, after exposure to 200°C, 400°C and 600°C. The percentage of weight reduction is observed lesser in concrete with Zeolite (Z), Silica Fume (S), and Fly Ash (F) compared with conventional concrete at 400°C and 600°C. The comparison in % of weight reduction for specimens with and without ZSF is nominal at 200°C. The reduction in weight of concrete specimens during exposure to elevated temperatures is linked to the degradation that takes place in the concrete during heating.

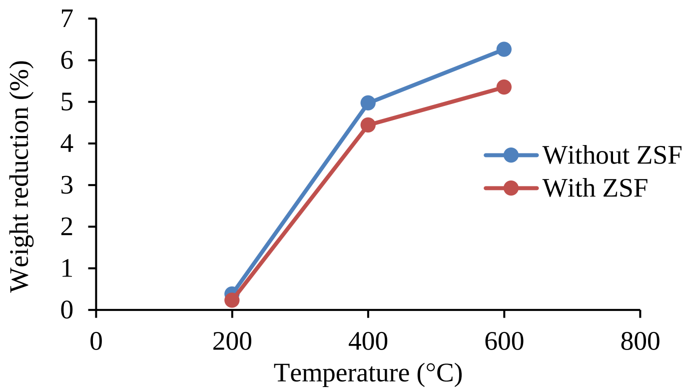


Fig. 5. Percentage of weight reduction after exposure to elevated temperatures

5.2.2 Compressive Strength

Concrete after 28 days of curing are exposed to temperatures of 200°C, 400°C, and 600°C for 1 hour after reaching the target temperature. The specimens are tested after cooling naturally in the furnace and the compressive strength test results are given in Fig. 6. It is inferred that compressive strength values of the mix 3 concrete with ZSF under elevated temperatures are higher at 200°C, 400°C, and 600°C when compared to the conventional concrete. This can be due to the fact of the denser interfacial transition zone with the filling of voids by finer zeolite, silica fumes, and class C fly ash. Mix 3 has 53.1 MPa, 51.3 MPa, and 42.7 MPa after exposure to 200°C, 400°C, and 600°C respectively, whereas conventional has the strength of 51.3 MPa, 48.5 MPa, and 40.2 MPa.

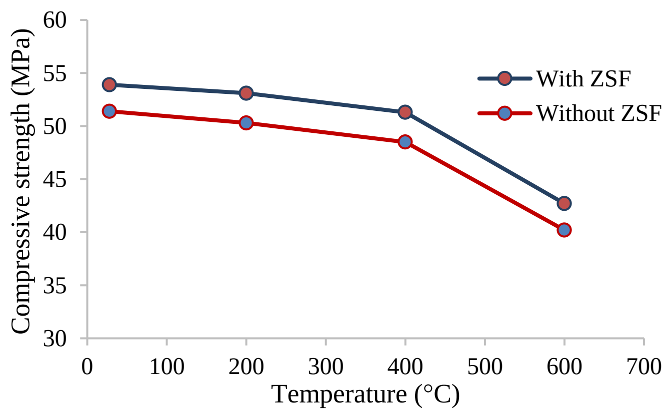


Fig. 6. Compressive strength for concrete mixes with and without ZSF

Table 6 gives the percentage difference in observed values of compressive strength obtained for concrete mixes with and without ZSF. Also, the percentage reduction in compressive strength upon temperature rise is obtained by comparing the values of strength results obtained for both concrete mixes with and without ZSF. The percentage reduction is observed lesser for concrete mixes with ZSF and this can be due to the formation of highly dense structures in the form of additional strength by CSH formation. The strength retention at a higher rate for concrete mix containing ZSF can be attributed to the fact of greater quantity of additional hydration products developed in concrete containing zeolite upon heating [14].

Table 6. Percentage reduction in compressive strength of concrete mixes with and without ZSF

Concrete mix	Compressive strength (MPa)				% Reduction in compressive strength with respect to room temperature			
	Room temperature	200°C	400°C	600°C	Room temperature	200°C	400°C	600°C
With ZSF	53.9	53.1	51.3	42.7	-	1.5	4.8	20.8
Without ZSF	51.4	50.3	48.5	40.2	-	2.1	5.6	21.8

5.2.3 Split Tensile Strength

Split tensile strength is observed to reduce at a higher rate compared to compressive strength upon temperature rise. Fig. 7 gives the split tensile strength obtained for concrete mixes with and without ZSF. Also, a percentage reduction in split tensile strength is observed higher for mixes without ZSF.

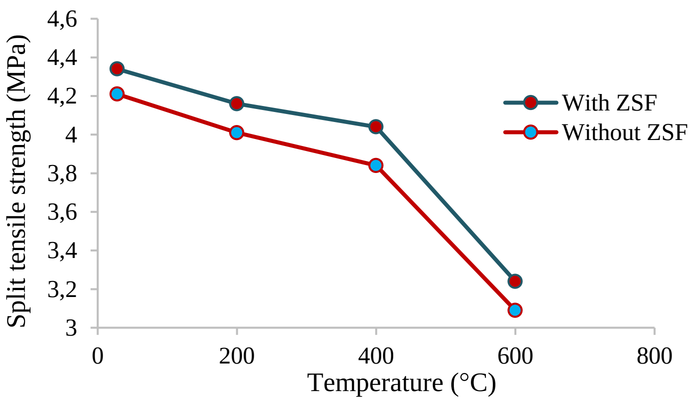


Fig. 7. Variation of split tensile strength for concrete mixes with and without ZSF

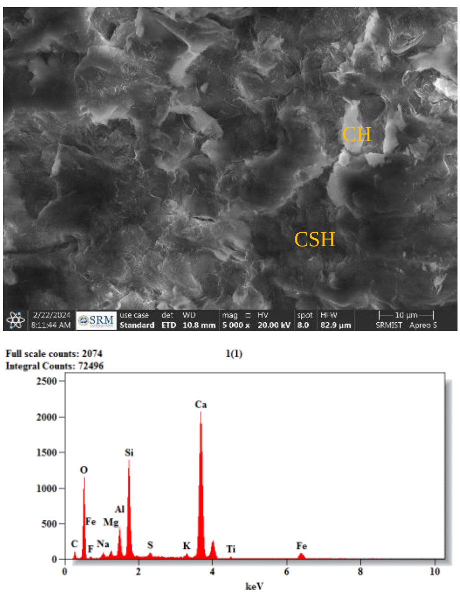
The values of split tensile strength for concrete with ZSF retained a higher percentage of tensile strength by 96%, 93.1%, and 75% after exposure to 200°C, 400°C and 600°C whereas conventional concrete without ZSF retained 95.3%, 91.2% and 73% at 200°C, 400°C and 600°C. Table 7 gives the percentage of reduction obtained for split tensile strength after exposure to elevated temperatures for concrete mixes with and without ZSF. The retainment of a higher percentage of tensile strength can be due to the dense transition zone between cement and aggregate particles due to the addition of supplementary cementitious materials.

Table 7. Percentage reduction in tensile strength for concrete mixes with and without ZSF

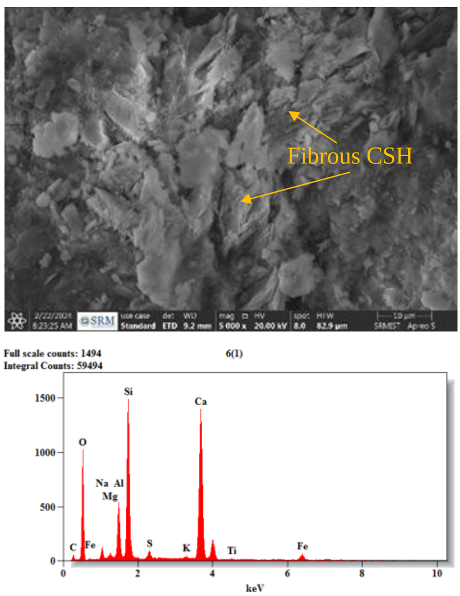
Concrete mix	Split tensile strength (MPa)				% Reduction in tensile strength with respect to room temperature			
	Room temperature	200°C	400°C	600°C	Room temperature	200°C	400°C	600°C
With ZSF	4.34	4.16	4.04	3.24	-	4	6.9	25
Without ZSF	4.21	4.01	3.84	3.09	-	4.7	8.8	27

5.2.4 Microstructural Characteristics

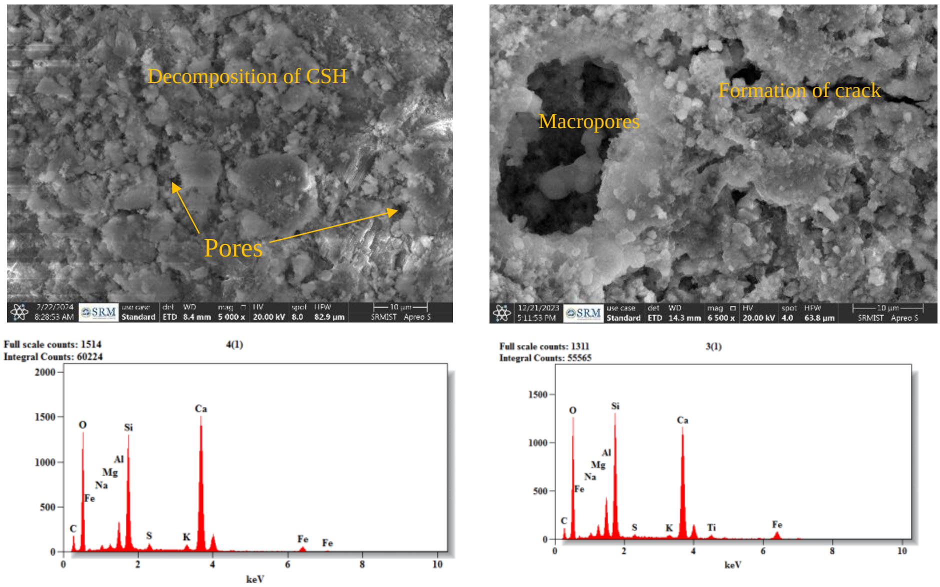
Fig. 8 gives the images obtained from Scanning Electron Microscope for concrete with ZSF at room temperature and after exposure to 200°C, 400°C and 600°C. From the images, a dense transition zone with the formation of CSH and CH is observed at room temperature. At temperatures higher than 200°C, the microstructure undergoes significant modifications, and at 400°C, the formation of a few voids with the decomposition of a dense matrix is noticed. The increase in temperature results in a decrease in the volume of CH and CSH compared to the room temperature, and temperature rise causes a change in the appearance of CSH crystals. At 200°C, partial decomposition of CH and CSH is observed with the fibrous CSH, and few pores are observed. At 400°C, the breakdown of the dense matrix is observed along with the decomposition of CSH gel. At 600°C, macropores are observed with the formation of cracks. As noticed from the SEM analysis, the EDX analysis performed on the specimens reveals a significant amount of calcium, silicon, and oxygen, in addition to other elements that suggest the formation of hydrated CSH gel.



a) Room temperature



b) After exposure to 200°C



c) After exposure to 400°C d) After exposure to 600°C

Fig. 8. Images of *scanning electron microscope* analysis and EDX analysis obtained for concrete with ZSF

Different peaks of calcium, silicon, and oxygen counts are observed with the temperature rise, and the count decreases with the temperature rise. The results are in correlation with SEM analysis for the observation of the decomposition of CSH and CH crystals at higher temperatures.

5. Conclusions

The objective of the present study is to investigate the concrete behaviour when the cement is replaced with Zeolite, Silica fumes, and class C fly ash, and the performance of concrete mix with ZSF is arrived at by testing the specimens for compressive strength test and split tensile strength. The effect of temperature rise on concrete with and without supplementary cementitious materials is identified and compared. The concluding remarks are as follows:

- In comparison to the standard concrete mix, mix 1 with 10% of zeolite has a reduction in 28 days strength by 5.4%, mix 2 with 7.5% silica fume and 10% fly ash has an increase in strength by 2%, and mix 3 which consists of 10% zeolite, 7.5% silica fume, and 10% class C fly ash has increase in strength by 4.9%. All three types of concrete had originally had lower compressive strengths at the end of 7 and 14 days of curing before eventually reaching the required target strength.
- On comparison of split tensile strength with the conventional mix, mixes 1 and 2 have a reduction in strength by 11% of their value while concrete mix 3 gains 3.1%. The addition of 10% zeolite, 7.5% silica fume and 10% fly ash resulted in the development of concrete mix with the desired compressive and tensile strength comparable with the conventional concrete mix.
- Compressive strength and split tensile strength of concrete mixes with and without ZSF is compared after exposure to elevated temperatures of 200°C, 400°C, and

600°C, and the percentage retention of strength values for concrete mix with ZSF are found higher compared with mixes without ZSF. The percentage retention of compressive strength and split tensile strength for concrete mixes with ZSF is 79.2% and 75%, and for mixes without ZSF is 78.2% and 73% after exposure to 600°C. Additional hydration products developed due to the addition of zeolite, silica fume, and fly ash resulted in the retention of strength values at a higher rate compared with the conventional concrete specimens at elevated temperatures.

- The strength value decreases with the temperature rise, and the percentage reduction in strength values is between the range of 20.8-27% at 600°C in both conventional mix and mix with ZSF. It is possible to ascribe the degradation of the microstructural components to the fact that a considerable loss of mechanical properties occurs at temperatures higher than 400°C. The degradation in mechanical properties is confirmed using EDX-SEM analysis. Microstructural study on concrete specimens presents the decomposition of CSH and CH matrix when the temperature exceeds 400°C.

The present study examined to develop sustainable concrete with the desired characteristics and performance at room and elevated temperatures by incorporating supplementary cementitious materials such as zeolite, silica fume, and class C fly ash.

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