Behavior of concrete using alccofine and nano-silica under elevated temperature

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Abstract
Fire protection is a critical concern since it can occur at any time because of naturally occurring or human threats. Concrete is proven to be excellent fire-resistant construction material, but spalling and infinite cracking occur if exposed to fire for a long period. In this paper, the effect of high temperature on concrete using alccofine (Al) and nano-silica (Ns) is examined for M40, M50, and M60 concrete grade. Residual compressive strength (RCS), % weight loss and spalling & color change of concrete cubes exposed to the fire temperatures of 200°C, 400°C, 600°C, 800°C & 1000°C for the period of 4, 8 and 12 hrs are studied. Results have shown that the addition of alccofine (Al) and nano-silica (Ns) to concrete decreased weight and compressive strength. The increase in temperature above 600°C affected the compressive strength of the Al + Ns mixes more than control mixes. At 1000°C, excessive micro-cracking is also observed. Increased fire duration substantially increased concrete degradation.

Keywords
Concrete, Alccofine (Al), Nano-silica (Ns), Compressive strength, Spalling and Color change.

1. Introduction
Infrastructural development is multiplying worldwide, which has raised the demand for raw materials used in producing concrete [1]. Fine aggregates, coarse aggregates, and cement are used to make concrete. Cement acts as the glue that binds concrete together, but cement manufacturing causes significant environmental impact [2]. Extensive research is being done to create concrete to minimize this effect by finding an alternative to cement [2]. Supplementary cementitious materials (SCM) used entirely or partially as a substitute enhances concrete efficiency. Still, their reactivity is affected by factors such as chemical composition, surface area, shape, and size of the SCM, percentage replacement, and cement type [3]. Unique properties like high strength, compact morphology, reduced permeability, and enhanced durability are achieved using SCM to produce high-performance concrete [4].

Concrete with high strength and durability are needed to withstand a wide range of loadings, like impact, earthquake, and wind loads, that may act on the structure over its lifetime [5]. The fire load is also one of those loads [5].

Fortunately, concrete is better than other construction materials because of specific advantages, such as ease of manufacture, strength, durability, fire resistance characteristics, etc. [6]. However, exposure of concrete to prolonged heating may disintegrate essential components such as calcium aluminate gels, calcium silicate hydroxide phase, and Ca(OH)₂ resulting in degradation of the physical and chemical properties of concrete [7], thus reducing the remaining service life. With the rise in fire temperature, the strength and the elastic modulus of the concrete begin to drop, and the carbonation depth increase gradually [8]. If the temperature reaches 300°C, then faster declination of strength occurs, and if the temperature rises further above 500°C, the concrete gets wholly damaged, and the strength decreases by 50 to 60 percent [9]. As the fire temperature increases above 400°C, the decomposition of hydrates in concrete commences, and at 900°C, concrete loses its initial
strength and stability [10]. At high temperatures, concrete does not retain its density, changes its color, becomes much more brittle, and fine cracks begin to appear on its surface [11]. The spalling, an essential property for measuring fire resistance, depends on permeability and decreases accordingly with increased permeability [12]. The extent of damage due to fire depends on temperature, exposure time, heating rate, cooling rate, and structural member size [13].

High-performance concrete is highly susceptible to high temperatures because of its reduced permeability, porosity, and dense morphology, resulting in cracks, thereby reducing concrete strength [4]. At elevated fire temperatures, the variations in the strength characteristics of concrete are mainly associated with changes in room temperature strength, the rate and duration of heating, and the additives used in concrete like silica, fly ash and slag, etc. [6]. Some SCM improve concrete properties at high temperatures because of pore filling and pozzolanic activity [14]. And also, the combined use of additives such as fly ash, meta-kaolin, and ultra-fine fly ash showed a positive impact on the concrete residual compressive strength. Also, it reduces weight loss, porosity, and water absorption at 1000˚C [15].

Nanotechnology is being widely used in the construction industry for the last several decades, making it easy to achieve high strength and high-performance concrete. Among all the nanoparticles, Ns is better in terms of cement hydration [16], strength [17, 18], durability [19], microstructural [20], and transport properties [21]. And also, a micro-fine material Al is generating interest among researchers for its use in concrete as a cement replacement as it causes an increase in mechanical [22], durability [23], and microstructural properties [24] of concrete. Our previous studies discovered that combining Al with Ns improved mechanical properties significantly than alcofine alone [25]. Therefore, in this research, the fire resistance of concrete using combinations of SCM like Ns and Al when exposed to elevated temperatures are studied.

2. Literature review

The following is a review of research on the behavior of high-performance concrete using nano-silica and alcofine as supplementary cementitious materials.

Mahapatra and Barai [26] investigated the residual characteristics of self-compacting concrete using colloidal nano-silica, low volume of fly ash, polypropylene fibers, and crimped steel fibers when subjected to temperatures up to 600°C for a fire duration of 1 hour. The increase in temperature concrete using colloidal nano-silica and fly ash showed significantly less weight loss than control concrete, and no spalling occurred at 600°C. At the same time, micro-cracks were noticed on the control concrete surface.

Mussa et al. [27] studied concrete dynamic and static properties using Ns and high-volume fly ash subjected to elevated temperatures of 400°C and 700°C for a fire duration of 2 hours. And found that at 700°C, both concrete mixtures showed a significant decrease in compressive strength; however, the concrete using Ns and high-volume fly ash showed a higher residual strength of 85.13%, compared to the control concrete.

Horszczaruk et al. [28] investigated the influence of cement mortars using Ns, quartz aggregate, heavyweight aggregates, i.e., magnetite, and barite when exposed to the temperature of 200˚C, 400˚C, 600˚C, and 800˚C, for a fire duration of 2 hours. The results concluded that cement mortar using Ns, quartz aggregate, and magnetite enhanced the thermal behavior and prevented the crack formation up to 400˚C temperatures.

Natarajan et al. [29] studied the properties of self-compacting mortars using glass powder and Ns subjected to high temperatures up to 800°C. And the results demonstrated that the use of 3% Ns by weight of cement improved the thermal behavior of mortars. The substitution of Ns combined glass powder almost compensated for the detrimental effects of glass powder on the strength properties of self-compacting mortar.

Elkady et al. [30] evaluated the mechanical properties of concrete using 1.5, 3, and 4.5% nano-silica by weight of cement exposed to temperatures ranging from 200 to 600°C. At 600°C concrete using 1.5%, Ns displayed minor strength loss and residual compressive strength of 73%. The bonding strength of concrete using Ns deteriorated faster than the compressive strength at higher temperatures.

Yonggui et al. [31] examined the efficiency of reinforced recycled concrete using basalt fiber and Ns at elevated temperatures of 25°C, 200°C, 400°C, and 600°C for a fire period of 6 hours. At various temperatures, Ns improved the compressive strength. According to the observation, Ns and basalt fiber enhanced the transition zone and the efficiency of recycled concrete at elevated temperatures.
Guler et al. [32] studied the influence of different combinations of nano-Al$_2$O$_3$, Ns, nano-Fe$_2$O$_3$, and nano-TiO$_2$ particles on the strength and microstructural properties of concrete subjected a temperature of 300°C, 500°C, and 800°C for a fire period of 2 hours. And the results concluded that nano-Al$_2$O$_3$ and Ns particles are much more effective in enhancing the strength properties of concrete than nano-Fe$_2$O$_3$ and nano-TiO$_2$ particles. While control concrete showed the highest reduction in residual compressive strength (57.65%), concrete containing 1.5 percent of nano-Al$_2$O$_3$ and Ns showed the least reduction (41.59%) at 800°C.

Sherif [33] examined the influence of temperatures of 200°C, 400°C, and 600°C on strength properties such as compressive strength and flexural strength of concrete using Ns and nano clay of (1, 2, 3, 4, & 5% by weight of cement) exposed for the fire period of 2 hours. The results demonstrated that Ns and nano clay enhanced the properties of concrete at 200°C temperatures. At 600°C compressive strength reduction, increased with an increase in cement content.

Vardhan and Rao [34] studied the impact of higher temperatures ranging from 100°C to 900°C on concrete using 10% Al by weight of cement exposed for 2 hours. And the results stated that as the temperature increased % weight loss increased, and the compressive strength of Al concrete decreased.

Bastami et al. [35] explored the behavior of concrete using Ns at high temperatures of 400°C, 600°C, and 800°C. According to the results, Ns improved concrete residual compressive strength and tensile strengths and reduced weight and spalling.

Shaikh and Haque [36] examined the behavior of geopolymer concrete using fine silica sand and Ns silica on compressive strength at high temperatures. At all temperatures, concrete using 2% Ns by weight of cement showed the maximum residual compressive strength and the minor mass loss and volumetric shrinkage.

From the literature reviewed, Ns as an SCM enhanced the thermal resistance of concrete and compensated for other SCM detrimental effects at high temperatures. So, in this paper, an attempt is made to study the properties of concrete using Al and Ns at elevated temperatures.

2.1 Research significance
Numerous studies have previously been conducted to find the concrete residual compressive strength after exposure to elevated temperatures. In this research, an attempt is made to understand the effect of temperatures of 200°C, 400°C, and 600°C, 800°C & 1000°C on concrete using Al and Ns exposed for the duration of 4, 8 and 12 hrs. For sustainable concrete, alccofine and nano-silica were added to improved transition zone, strength, and durability. Residual compressive strength, loss of weight, spalling and color change of concrete using Al + Ns were tested and compared with control mixtures without Al and Ns to evaluate the behavior of concrete at elevated temperatures.

3. Materials and methods
3.1 Materials
3.1.1 Cement
Ordinary Portland cement of Grade 53, procured from a single source and conforming to IS 12269-1987, is used. The results of tests to determine the physical properties of cement as per c are given in Table 1.

| Sr. No. | Properties                      | Unit     | Values |
|--------|--------------------------------|----------|--------|
| 1.     | Specific gravity                | -        | 3.15   |
| 2.     | Fineness                       | m$^2$/kg | 300    |
| 3.     | Soundness (Le Chatelier method) | mm       | 1.2    |
| 4.     | Standard Consistency           | %        | 32     |
| 5.     | Final setting time              | min      | 259    |
| 6.     | Initial setting time            | min      | 56     |

3.1.2 Fine aggregates
In this research, natural river sand is used, and its physical properties are tested according to IS 2386:1963. Table 2 shows the results of the test on physical properties of fine aggregates. The particle size distribution of natural river sand used as a fine aggregate (FA) with a fineness modulus (FM) of 3.18 is shown in Figure 1.
Table 2: Physical properties of Fine aggregates

| Sr. No. | Properties          | Unit | Values |
|---------|---------------------|------|--------|
| 1.      | Type                | -    | River sand |
| 2.      | Zone                | -    | II     |
| 3.      | Specific gravity    | -    | 2.65   |
| 4.      | Bulk density        |       |         |
|         | a) Compacted condition | Kg/m³ | 1642   |
|         | b) Loose condition  |       | 1520   |
| 5.      | Fineness modulus    | -    | 3.18   |

Figure 1: Particle size distribution curve of fine aggregate

3.1.3 Coarse aggregates
The gravel with a maximum size of 20 mm collected from local suppliers is used as coarse aggregate. Its physical properties are tested as per IS 2386:1963. Table 3 shows the values of the physical properties of coarse aggregate.

3.1.4 Alccofine
Alccofine (Al) is ultra-fine slag obtained from Ambuja Cement Pt. Ltd. The alccofine is shown in Figure 2. Table 4 shows the physical and chemical properties of Al provided by the manufacturer.

3.1.5 Nano silica
Nano-silica (Ns) is a white fluffy material with a high-quality amorphous Si content obtained from Astra chemicals Chennai. Table 5 shows the physical and chemical properties of Ns provided by the manufacturer. The nano-silica is shown in Figure 3.

3.1.6 Superplasticizer
Fosroc Conplast SP430 DIS is used as the superplasticizer in this research. The dosage of the superplasticizer is changed depending on the mix and finalized via slump test trials. Table 4 shows the physical properties of the superplasticizer provided by the manufacturer.

3.1.7 Water
Laboratory tap water is used for casting and curing, and it is added according to the mix design.

3.2 Mix proportions
Mix proportions of M40, M50, and M60 concrete grade are design as per IS 10262:2019 and IS 456:2005. In this research, two different mixes of M40, M50, and M60 concrete grade, one using 100% Ordinary Portland cement and other 82% Ordinary Portland cement, 15% Al, & 3% Ns is prepared. The properties of all the concrete mixes at elevated temperatures are examined. Table 6 shows the mix proportions and notations of all the six different combinations.

3.3 Casting and curing of test specimens
The casting of test specimens is done by pouring concrete into the 150 mm cubical molds and correctly tempering it using vibration to eliminate voids. Smooth distribution of cement paste over the whole area of the concrete specimen was done to make the top surface even. Molds are unfastened after 24 hours, and then concrete specimens are immersed in water for 28 days curing period.
Table 3 Physical properties of coarse aggregates

| Sr. No. | Properties                  | Unit   | Values     |
|---------|-----------------------------|--------|------------|
| 1.      | Type                        | -      | Angular    |
| 2.      | Specific gravity            | -      | 2.79       |
| 3.      | Bulk density                | Kg/m³  |            |
| a)      | Compacted condition         |        | 1530       |
| b)      | Loose condition             |        | 1416       |
| 4.      | Fineness modulus            | -      | 7.6        |
| 5.      | Free surface moisture       | -      | -          |

Figure 2 Alccofine

Figure 3 Nano-silica

Table 4 Physical and chemical properties of Al and Ns

| Sr. No. | Properties                  | Nano silica | Alccofine |
|---------|-----------------------------|-------------|-----------|
| 1.      | Specific gravity            | 2.2-2.4     | 2.86      |
| 2.      | Colour                      | White       | Gray      |
| 3.      | Mean particle size          | 17nm        | 4-6µm     |
| 4.      | SiO₂                        | 99.88       | 33-35     |
| 5.      | CaO                         | 0.06        | 32-34     |
| 6.      | Al₂O₃                       | 0.005       | 18-20     |
| 7.      | Fe₂O₃                       | 0.001       | 1.8-2     |
| 8.      | MgO                         | 2.2-2.4     | 8-10      |
| 9.      | SO₃                         | -           | 0.3-0.7   |

Table 5 Physical properties of superplasticizer

| Sr. No. | Properties                  | Values     |
|---------|-----------------------------|------------|
| 1.      | Specific gravity            | 1.2        |
| 2.      | Colour                      | Brown liquid|
| 3.      | Chloride content            | Nil        |
| 4.      | Air entrainment             | < 2%       |

Table 6 Mix proportions and notations of different mixes

| Mix   | M40 | M50 | M60 |
|-------|-----|-----|-----|
| SCM   |     | AL+Ns |     | AL+Ns |     | AL+Ns |
| Notations | M4 | M4AlNs | M5 | M5AlNs | M6 | M6AlNs |
| Water (Kg/m³) | 160 | 160 | 159 | 159 | 158 | 158 |
| Cement (Kg/m³) | 400 | 328 | 440 | 360.8 | 527 | 432.2 |
| Fine aggregate (Kg/m³) | 667 | 667 | 642 | 642 | 596 | 596 |
| Coarse aggregate (Kg/m³) | 1248 | 1248 | 1243 | 1243 | 1218 | 1218 |
| Alccofine (Kg/m³) | 60 | 60 | 66 | 66 | - | 79 |
| Nano silica (Kg/m³) | 12 | 12 | 13.2 | 13.2 | - | 15.8 |
| w/c | 0.4 | 0.4 | 0.36 | 0.36 | 0.3 | 0.3 |
### 3.4 Test methods

The behavior of all concrete mixes at elevated temperatures is studied. The following testing procedure is used, as shown in the flowchart in Figure 4. The sections below cover the details of the test methods.

#### 3.4.1 Heating regime

Three samples of each concrete mix are exposed to elevated temperatures between 200 to 1000 °C for a duration of 4, 8, and 12 hrs in an electrical bogie hearth furnace shown in Figure 5, of 1300 °C maximum temperature. The furnace uses silicon carbide as a heating element with a chamber dimension of 300 mm in width, 1500 mm in depth, and 350 mm in height. Then the samples are allowed to cool in the air at room temperature.

#### 3.4.2 Spalling and color change

In this research visual observations are carried out on concrete specimens exposed to high temperatures with bare eyes by detecting spalling and color change.

#### 3.4.3 Percentage weight loss

Before and after being exposed to high temperatures, the weights of concrete samples were measured. The weighing of concrete specimens is shown in Figure 6. Percentage weight loss is calculated using the following Equation 1.

\[
W_{\text{loss}} = \frac{(W_1 - W_2)}{W_1} \times 100
\]  

Where,

- \( W_{\text{loss}} \) = Percentage weight loss
- \( W_1 \) = Wt. of the cubic concrete specimen before exposure to high temperatures
- \( W_2 \) = Wt. of the cubic concrete specimen after exposure to high temperatures

#### 3.4.4 Compressive strength test

Compressive strength is determined after subjected to higher temperatures. The cubic specimens of 150 mm size are tested in a compression testing machine of the maximum capacity of 2000KN. Three specimens are used for each mix. The specimens are removed from the furnace and stored at room temperature until they reach a constant dry weight before being tested. After testing, compressive strength is determined by dividing load at failure by an area of the specimen according to IS 516-1959. The testing of concrete specimens in the compression testing machine is shown in Figure 7.

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**Figure 4** Flowchart of the concrete testing process
Figure 5 Heating of concrete specimens in Electrical bogie hearth furnace

Figure 6 Weighing of concrete specimens

Figure 7 Compression testing of concrete specimens
4. Results
4.1 Spalling and color change
Color change and deformations/spalling through visual observations on the concrete surface of control and Al+Ns mixes at different fire durations with an increase in temperatures are presented in Tables 7, 8, & 9. Short hairline cracks formed on the concrete surface of control and Al+Ns mixes are highlighted with black color at 400°C @ 12hrs and 600°C @ 4 & 12 hrs fire duration.

Table 10 shows concrete specimens after compression failure at different fire temperatures and durations. The type of failure is almost similar for both control and Al+Ns mixes at a particular fire temperature and duration; hence only one specimen after compression failure is displayed in Table 10. This study demonstrated the change in aggregates color by marking at least one aggregate with the red circle and deterioration of binding property between aggregates and hardened cement paste marked with a yellow color arrow on concrete specimens subjected to 1000°C in Table 10.

4.2 Percentage weight loss
Percentage weight loss at different temperatures and fire durations is calculated considering the weight of concrete specimens before subjected to elevated temperatures. Table 11 shows the percentage weight loss of both control and Al+Ns mixes at different temperatures and fire durations.

4.3 Compressive strength
Residual compressive strength is the resistance of fire-damaged specimens against compressive forces. Residual compressive strength of both control and Al+Ns mixes at different temperatures and fire durations are shown in Figures 8, 9, & 10. Percentage loss of compressive strength at different temperatures and fire durations is calculated considering compressive strength at room temperature 27°C is given in Table 12. Negative values in Table 12 indicate an increase in the percentage of compressive strength.

Table 7 Color change and deformations of concrete at fire duration of 4 hrs

| Temp. | 200 °C | 400 °C | 600 °C | 800 °C | 1000 °C |
|-------|--------|--------|--------|--------|---------|
| M4    | ![Image](400°C_M4.jpg) | ![Image](600°C_M4.jpg) | ![Image](800°C_M4.jpg) | ![Image](1000°C_M4.jpg) | ![Image](1000°C_M4.jpg) |
| M4AlNs| ![Image](400°C_M4AlNs.jpg) | ![Image](600°C_M4AlNs.jpg) | ![Image](800°C_M4AlNs.jpg) | ![Image](1000°C_M4AlNs.jpg) | ![Image](1000°C_M4AlNs.jpg) |
| M5    | ![Image](400°C_M5.jpg) | ![Image](600°C_M5.jpg) | ![Image](800°C_M5.jpg) | ![Image](1000°C_M5.jpg) | ![Image](1000°C_M5.jpg) |
Table 8 Color change and deformations of concrete at fire duration of 8 hrs

| Temp. | 200 °C | 400 °C | 600 °C | 800 °C | 1000 °C |
|-------|--------|--------|--------|--------|---------|
| M5AlNs | ![Image](image1) | ![Image](image2) | ![Image](image3) | ![Image](image4) | ![Image](image5) |
| M6    | ![Image](image6) | ![Image](image7) | ![Image](image8) | ![Image](image9) | ![Image](image10) |
| M6AlNs| ![Image](image11) | ![Image](image12) | ![Image](image13) | ![Image](image14) | ![Image](image15) |
| Temp. | 200 °C | 400 °C | 600 °C | 800 °C | 1000 °C |
|-------|--------|--------|--------|--------|---------|
| M5    | ![Image](image1) | ![Image](image2) | ![Image](image3) | ![Image](image4) | ![Image](image5) |
| M5AlNs| ![Image](image6) | ![Image](image7) | ![Image](image8) | ![Image](image9) | ![Image](image10) |
| M6    | ![Image](image11) | ![Image](image12) | ![Image](image13) | ![Image](image14) | ![Image](image15) |
| M6AlNs| ![Image](image16) | ![Image](image17) | ![Image](image18) | ![Image](image19) | ![Image](image20) |

**Table 9** Color change and deformations of concrete at fire duration of 12 hrs

| Temp. | 200 °C | 400 °C | 600 °C | 800 °C | 1000 °C |
|-------|--------|--------|--------|--------|---------|
| M4    | ![Image](image21) | ![Image](image22) | ![Image](image23) | ![Image](image24) | ![Image](image25) |
| Temp.  | 200 °C | 400 °C | 600 °C | 800 °C | 1000 °C |
|-------|--------|--------|--------|--------|---------|
| M4AlNs| ![Image](image1) | ![Image](image2) | ![Image](image3) | ![Image](image4) | ![Image](image5) |
| M5    | ![Image](image6) | ![Image](image7) | ![Image](image8) | ![Image](image9) | ![Image](image10) |
| M5AlNs| ![Image](image11) | ![Image](image12) | ![Image](image13) | ![Image](image14) | ![Image](image15) |
| M6    | ![Image](image16) | ![Image](image17) | ![Image](image18) | ![Image](image19) | ![Image](image20) |
| M6AlNs| ![Image](image21) | ![Image](image22) | ![Image](image23) | ![Image](image24) | ![Image](image25) |
**Table 10** Concrete specimens after compression failure

| Room temperature | Duration | 4 Hrs. | 8 Hrs. | 12 Hrs. |
|-------------------|----------|--------|--------|---------|
| 200°C             |          | ![Image](image1.png) | ![Image](image2.png) | ![Image](image3.png) |
| 400°C             |          | ![Image](image4.png) | ![Image](image5.png) | ![Image](image6.png) |
| 600°C             |          | ![Image](image7.png) | ![Image](image8.png) | ![Image](image9.png) |
| 800°C             |          | ![Image](image10.png) | ![Image](image11.png) | ![Image](image12.png) |
Table 11 Percentage weight loss of concrete subjected to high temperature

| Temperature (ºC) | Duration (hrs) | Percentage weight loss of concrete specimens (%) |
|------------------|----------------|-----------------------------------------------|
|                  | 4              | M4    | M4AlNs | M5    | M5AlNs | M6    | M6AlNs |
| 200              | 4              | 0.6351| 0.7240 | 0.8760| 0.9333 | 0.7387| 0.9289 |
|                  | 8              | 1.0363| 1.1200 | 0.9813| 1.0800 | 0.8745| 1.0661 |
|                  | 12             | 1.4495| 2.4888 | 2.0293| 2.5763 | 1.3302| 1.7249 |
| 400              | 4              | 3.6668| 3.7884 | 3.4378| 4.0725 | 3.6226| 4.2700 |
|                  | 8              | 4.3177| 4.5310 | 3.8960| 4.7457 | 4.2069| 4.4951 |
|                  | 12             | 4.4163| 4.8590 | 4.0974| 4.9916 | 4.4099| 5.1882 |
| 600              | 4              | 4.9263| 5.3438 | 4.9679| 5.7403 | 5.1688| 5.4500 |
|                  | 8              | 5.3626| 5.7068 | 5.3140| 5.8018 | 5.5576| 5.9400 |
|                  | 12             | 5.6881| 5.9002 | 5.6139| 6.0594 | 5.9277| 6.1646 |
| 800              | 4              | 6.0100| 6.2981 | 6.0303| 6.5000 | 6.1000| 6.4892 |
|                  | 8              | 6.1326| 6.3987 | 6.1743| 6.6550 | 6.2832| 6.8300 |
|                  | 12             | 6.2711| 6.4583 | 6.3263| 6.7305 | 6.5100| 7.1000 |
| 1000             | 4              | 6.3200| 6.5200 | 6.2100| 6.6300 | 6.3200| 6.6200 |
|                  | 8              | 6.6394| 6.8860 | 6.4817| 6.8166 | 6.7331| 7.0100 |
|                  | 12             | 6.6395| 7.0287 | 6.9148| 7.0733 | 7.0600| 7.2303 |

Table 12 Percentage Loss of concrete compressive strength at high temperature

| Temperature (ºC) | Duration (hrs) | % Loss of compressive strength |
|------------------|----------------|-------------------------------|
|                  | 4              | M4    | M4AlNs | M5    | M5AlNs |
| 200              | 4              | -14.931 | -10.707 | -12.890 | -7.353 |
|                  | 8              | -10.851 | -1.414 | -11.452 | -4.039 |
|                  | 12             | 3.990   | 4.916   | 5.201   | 7.685   |
| 400              | 4              | 0.337   | 1.549   | 1.155   | 2.216   |
|                  | 8              | 4.916   | 5.724   | 4.526   | 7.528   |
|                  | 12             | 7.179   | 9.899   | 8.974   | 9.980   |
| 600              | 4              | 5.257   | 21.751  | 4.903   | 24.356  |
|                  | 8              | 13.602  | 30.162  | 15.904  | 32.761  |
|                  | 12             | 25.764  | 40.983  | 23.621  | 41.462  |
| 800              | 4              | 40.807  | 55.966  | 47.443  | 52.625  |
|                  | 8              | 50.399  | 64.074  | 53.890  | 62.194  |
|                  | 12             | 65.260  | 74.983  | 68.578  | 73.824  |
| 1000             | 4              | 55.131  | 67.232  | 52.497  | 72.969  |
|                  | 8              | 78.579  | 87.440  | 80.655  | 87.830  |
|                  | 12             | 85.174  | 90.505  | 86.720  | 91.916  |
Figure 8 Residual compressive strength for fire duration of 4 hrs

Figure 9 Residual compressive strength for fire duration of 8 hrs

Figure 10 Residual compressive strength for fire duration of 12 hrs
5. Discussion

5.1 Spalling and color change
Concrete, when exposed to high and rapidly rising temperatures as a result of fire, pieces of concrete slip from its surface, known as spalling [6]. It is among the essential properties to see the fire performance of concrete [6]. The primary signs of the distortion of concrete subjected to high temperature are color change and surface appearance [37]. In the present analysis, no cracks on the concrete surface are noticed through visual observations for both control and Al+Ns concrete mixes after exposure to 200°C and 400°C temperature for 4hrs and 8hrs fire duration/period. And, when the duration increased to 12hrs, few small hairline cracks were found through visual observations at 400°C for both control and Al+Ns concrete mixes due to water vaporization in voids [38] of concrete. No change in color on the concrete surface is observed from Table 7, 8, & 9 for both control and Al+Ns concrete mixes subjected to 200°C @ 4hrs, but the concrete surface changed to light grey color as the fire duration increased. At 400°C, the concrete surface changed to light grey when the fire duration reached 4 hrs and dark grey when the fire duration was 8 & 12 hours in both control and Al+Ns concrete mixes. At 600 °C, after being exposed to fire for 4 hours, short hairline cracks are observed, and the length and width of the crack increased significantly with increased fire duration, and the no. of cracks formed are greater for Al+Ns concrete mixes, i.e., M4AlNs, M5AlNs, and M6AlNs. And also, the color of the concrete surface changed to brownish red. As the temperature increased to 800°C, fine irregular cracks appeared on the concrete surface, with a more significant number and width of cracks formed on M4AlNs, M5AlNs, and M6AlNs concrete surface. The width of the cracks widened. The surface color of concrete changed from grey-white to whitish with increased fire duration. The CaCO3 turns to CaO, and carbon dioxide gets released due to the calcination process at high temperatures [9]. Excessive cracking occurred at 1000°C, and slight spalling was observed for fire duration of 8 & 12hrs on M4AlNs, M5AlNs, and M6AlNs concrete surface, and the color of the concrete surface changed to whitish. Mainly from Table 10, it is detected that as the temperature grew from 600 to 1000°C, aggregates decomposed, the color changed to red, and binding properties are also lost [39]. From the visual observation shown in Table 7, 8, & 9, it is noticed that the cracks formation and color change are seemed to be almost similar for both the control mixes M4, M5, & M6 and Al+Ns mixes M4AlNs, M5AlNs, and M6AlNs up to 400°C. Beyond 400°C, the number of micro-cracks formed on M4AlNs, M5AlNs, and M6AlNs concrete surfaces is slightly greater.

5.2 Percentage weight loss
Results illustrated in Table 11 show that the weight of the concrete specimens subjected to elevated temperature decreases with increases in temperature and fire duration. Weight loss occurs in the concrete specimens because of the formation of air voids due to the release of bound water from the concrete matrix [40]. The control mix M4, M5 & M6 shows a minimum percentage weight loss of 0.6351, 0.876, & 0.738% at 200°C exposed for the duration of 4hrs. As the temperature increases further from 200°C to 1000°C, an additional decrease in the weight of concrete specimens occurred. Comparing the results of percentage weight loss in Table 11, M4AlNs, M5AlNs, and M6AlNs mixes showed a higher reduction in weight at all temperatures. The weight reduction may be due to increased water evaporation from the C-S-H structure in concrete and greater heat conductivity of Al and Ns structure [41]. Table 10 displays that aggregate deteriorated at 600°C 800°C & 1000°C, which caused the weight of the concrete to decrease further and continued to decline with an increase in fire duration. The highest % weight loss of 7,0287, 7,0733, & 7.2303 is observed at 1000°C @ 12hrs for M4AlNs, M5AlNs, and M6AlNs mixes. The decomposition of C-S-H formed due to the introduction of Al and Ns may be the main reason for the rate of increase in % weight loss at high temperature with increased fire duration [41]. Similar results are observed by Demirel and Keleştemur [40], Sancak et al. [41], and Akoz et al. [42].

5.3 Compressive strength
For designing any fire resistance structure or building, the concrete strength at an intense temperature is very essential [6]. Figures 8, 9 and 10, show the residual compressive strength results of the concrete specimens exposed to the temperatures of room temperature, 200°C, 400°C, 600°C, 800°C & 1000°C for the duration of 4, 8 and 12 hrs. The percentage loss in compressive strength at each temperature and duration of fire calculated with respect to the strength at room temperature is given in Table 12. From Figures 8 and 9. The residual compressive strength for both control and Al+Ns concrete mixes increased at 200°C for a fire duration of 4 and 8 hrs. The residual compressive strength of Al+Ns concrete mixes is greater because of reactive SiO2 content in the Ns, thus enhancing the pozzolanic reaction [40, 42, 28] even at 200°C. And then, as the temperature and fire period increase, the strength finally decreases. Al+Ns concrete mixes...
M4AlNs, M5AlNs, and M6AlNs concrete mixes have a higher residual compressive strength of 73.07, 75.90 & 88 Mpa, because of its higher room temperature strength, while the percentage increase in compressive strength at 200ºC being more significant for control mixes when exposed for 4hrs and 8hrs fire duration [6]. At 400ºC, loss of compressive strength increased with increased fire duration due to evaporation of water present in the voids of the concrete structure. Percentage loss of compressive strength is between 0.3 to 9 % for M4, M5 & M6 mixes and 1 to 11% for M4AlNs, M5AlNs, and M6AlNs at fire duration of 4, 8 & 12 hrs. At 600ºC, the percentage loss of compressive strength is between 4 to 28 % for M4, M5 & M6 mixes. And 21 to 42% for M4AlNs, M5AlNs, and M6AlNs at fire duration of 4, 8 & 12 hrs. And the residual compressive strength of M4AlNs, M5AlNs, and M6AlNs at 600ºC @ 12hrs is less than M4, M5 & M6 mix. The results from Table 12 illustrate that percentage loss of compressive strength using Al and Ns were higher at 800ºC & 1000ºC in comparison to control mixes. These results are similar to Demirel and Oguzhan [40], Sancak et al. [41], and Akoz et al. [42]. It is because of the latent hydraulic properties of Al and 99 percent SiO₂ content in Ns, the hydration and pozzolanic reactions of these SCM's in the concrete lead to the formation of additional C-S-H gels. C-S-H gels formed further degraded at 800ºC & 1000ºC temperatures, causing calcium carbonate to convert into lime, thereby weakening the binding properties of concrete [9]. The % loss of compressive strength of M4AlNs, M5AlNs, and M6AlNs at 800ºC & 1000ºC temperatures is between 50 and 91 percent. Due to the dense, compact morphology of the M4AlNs, M5AlNs, and M6AlNs, the pore pressure at high temperatures increases, causing an increase in micro-cracks and a rapid deterioration compressive strength [6, 43, 44, 45]. The M4AlNs, M5AlNs, and M6AlNs mix achieved the least residual compressive strength of 6.27, 5.72, & 7.19 Mpa at 1000ºC @ 12hrs. Table 10 shows that semi-explosive failure occurred at 600ºC & 800ºC, and explosive failure occurred at 1000ºC [46] due to weak concrete strength at high temperatures. Description of the abbreviations used in this paper can be found in Appendix I.

6. Conclusion and future work

The cracks formation and color change are almost similar for both the M4, M5, & M6 mixes and M4AlNs, M5AlNs, and M6AlNs mixes up to 400ºC, but beyond that temperature number of micro-cracks formed on M4AlNs, M5AlNs, and M6AlNs mixes surfaces is slightly greater. For all the concrete mixes decreasing trend in compressive strength and weight is noticed due to increased temperature and fire duration. M4AlNs, M5AlNs, and M6AlNs mixes showed greater percentage loss in weight at all temperatures. Concrete mixes M4AlNs, M5AlNs, and M6AlNs showed a maximum weight loss percentage of 6.52 to 7.23% at 1000ºC for 4 to 12 hours, while the control mixes showed a maximum weight loss percentage of 6.2 to 7.07% at 1000ºC for 4 to 12 hours of the fire. M4AlNs, M5AlNs, and M6AlNs concrete mixes reported a higher residual compressive strength of 73.07, 75.90 & 88 Mpa at 200ºC due to its higher room temperature strength. However, the percentage increase in compressive strength for control mixes at 200ºC being greater. For the concrete specimens subjected to temperatures greater than 600ºC, the decrease in the compressive strength of M4AlNs, M5AlNs, and M6AlNs concrete mixes is substantially greater. At 1000ºC for fire duration of 4 to 12 hours, the maximum percentage loss of compressive strength of M4AlNs, M5AlNs, and M6AlNs mixes was 67.23 to 91.92%, while the control mixes M4, M5, & M6, showed a percentage loss of 52.5 to 86.97%.

Future research on concrete using Al and Ns in high-temperature environments has a lot of potentials. It is recommended to study stress-strain relationships, modulus of elasticity, split tensile strength, microstructural properties, initiation, propagation of cracks, and chemical properties of concrete subjected to elevated temperatures for future work. And also, retrofitting methods to restore the load-bearing capacity of concrete members can be studied.

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Conflicts of interest

The authors have no conflicts of interest to declare.

References

[1] Khan EU, Khushnood RA, Baloch WL. Spalling sensitivity and mechanical response of an ecofriendly sawdust high strength concrete at elevated temperatures. Construction and Building Materials. 2020.

[2] Gettu R, Patel A, Rathi V, Prakasan S, Basavaraj AS, Palaniappan S, et al. Influence of supplementary cementitious materials on the sustainability parameters of cements and concretes in the Indian context. Materials and Structures. 2019; 52:1-11.

[3] Pacewska B, Wilinska I. Usage of supplementary cementitious materials: advantages and limitations. Journal of Thermal Analysis and Calorimetry. 2020; 142:371-93.

[4] Lu D, Tang Z, Zhang L, Zhou J, Gong Y, Tian Y, et al. Effects of combined usage of supplementary...
cementitious materials on the thermal properties and microstructure of high-performance concrete at high temperatures. Materials. 2020; 13(8):1-12.
[5] Cree D, Pliya P, Green MF, Noumowé A. Thermal behaviour of unstressed and stressed high strength concrete containing polypropylene fibers at elevated temperature. Journal of Structural Fire Engineering. 2017; 8(4):402-17.
[6] Kodur V. Properties of concrete at elevated temperatures. International Scholarly Research Notices. 2014.
[7] Vodák F, Trtik K, Kapičková O, Hošková Š, Demo P. The effect of temperature on strength–porosity relationship for concrete. Construction and Building Materials. 2004; 18(7):529-34.
[8] Li Q, Li Z, Yuan G. Effects of elevated temperatures on properties of concrete containing ground granulated blast furnace slag as cementitious material. Construction and Building Materials. 2012; 35:687-92.
[9] Hager I. Behaviour of cement concrete at high temperature. Bulletin of the Polish Academy of Sciences. Technical Sciences. 2013; 61(1):145-54.
[10] Akca AH, Zihnioğlu NO. High performance concrete under elevated temperatures. Construction and Building Materials. 2013; 44:317-28.
[11] Drzymała T, Jackiewicz-Rek W, Tomaszewski M, Kuś A, Gahj J, Šukys R. Effects of high temperature on the properties of high performance concrete (HPC). Procedia Engineering. 2017; 172:256-63.
[12] Li Y, Tan KH, Yang EH. Synergistic effects of hybrid polypropylene and steel fibers on explosive spalling prevention of ultra-high performance concrete at elevated temperature. Cement and Concrete Composites. 2019; 96:174-81.
[13] Lee J, Yi Y, Willam K. Properties of concrete after high-temperature heating and cooling. ACI Materials Journal. 2008; 105(4):334-41.
[14] Saboo N, Shivhare S, Kori KK, Chandrappa AK. Effect of fly ash and metakaolin on pervious concrete properties. Construction and Building Materials. 2019; 223:322-8.
[15] Zhou J, Lu D, Yang Y, Gong Y, Ma X, Yu B, et al. Physical and mechanical properties of highstrength concrete modified with supplementary cementitious materials after exposure to elevated temperature up to 1000°C. Materials. 2020; 13(3):1-13.
[16] Hou P, Kawashima S, Kong D, Corr DJ, Qian J, Shah SP. Modification effects of colloidal nanoSiO2 on cement hydration and its gel property. Composites Part B: Engineering. 2013; 45(1):440-8.
[17] Said AM, Zeidan MS, Bassuoni MT, Tian Y. Properties of concrete incorporating nano-silica. Construction and Building Materials. 2012; 36:838-44.
[18] Qing Y, Zeman Z, Deyu K, Rongshen C. Influence of nano-SiO2 addition on properties of hardened cement paste as compared with silica fume. Construction and Building Materials. 2007; 21(3):539-45.
[19] Chithra S, Kumar SS, Chinmaraju K. The effect of colloidal nano-silica on workability, mechanical and durability properties of high performance concrete with copper slag as partial fine aggregate. Construction and Building Materials. 2016; 113:794-804.
[20] Snehal K, Das BB, Akanksha M. Early age, hydration, mechanical and microstructure properties of nano-silica blended cementitious composites. Construction and Building Materials. 2020.
[21] Ghafari E, Costa H, Júlio E, Portugal A, Durães L. The effect of nanosilica addition on flowability, strength and transport properties of ultra high performance concrete. Materials & Design. 2014; 59:1-9.
[22] Kumar MP, Mini KM, Rangarajan M. Ultrafine GGBS and calcium nitrate as concrete admixtures for improved mechanical properties and corrosion resistance. Construction and Building Materials. 2018; 182:249-57.
[23] Teng S, Lim TY, Divsholi BS. Durability and mechanical properties of high strength concrete incorporating ultra fine ground granulated blast-furnace slag. Construction and Building Materials. 2013; 40:875-81.
[24] Nasirimah Reddy P, Ahmed Naqash J. Experimental study on TGA, XRD and SEM analysis of concrete with ultra-fine slag. International Journal of Engineering. 2019; 32(5):679-84.
[25] Ashwini K, Rao PS. Evaluation of correlation between compressive and splitting tensile strength of concrete using alcofine and nano silica. In IOP conference series: materials science and engineering 2021 (pp. 1-7). IOP Publishing.
[26] Mahapatra CK, Barai SV. Temperature impact on residual properties of self-compacting based hybrid fiber reinforced concrete with fly ash and colloidal nano silica. Construction and Building Materials. 2019; 198:120-32.
[27] Musa MH, Mutalib AA, Hamid R, Raman SN. Dynamic properties of high volume fly ash nanosilica (HVFANS) concrete subjected to combined effect of high strain rate and temperature. Latin American Journal of Solids and Structures. 2018; 15(1):1-19.
[28] Horszczaaruk E, Sikora P, Cendrowski K, Mijowska E. The effect of elevated temperature on the properties of cement mortars containing nanosilica and heavy-weight aggregates. Construction and Building Materials. 2017; 137:420-31.
[29] Natarajan S, Udayabanu M, Ponnan S, Murugan S. Performance of nano-silica modified self-compacting glass mortar at normal and elevated temperatures. Materials. 2019; 12(3):1-15.
[30] Elkady HM, Yasien AM, Elfeky MS, Serag ME. Assessment of mechanical strength of nano silica concrete (NSC) subjected to elevated temperatures. Journal of Structural Fire Engineering. 2019;10(1):90-109.
[31] Yonggui W, Shuaipeng L, Hughes P, Yuhui F. Mechanical properties and microstructure of basalt fibre and nano-silica reinforced recycled concrete after exposure to elevated temperatures. Construction and Building Materials. 2020.
[32] Guler S, Türkmenoğlu ZF, Ashour A. Performance of single and hybrid nanoparticles added concrete at
ambient and elevated temperatures. Construction and Building Materials. 2020.

[33] Sherif MA. Effect of elevated temperature on mechanical properties of nano materials concrete. International Journal of Engineering and Innovative Technology. 2017; 7:1-9.

[34] Vardhan PR, Rao TM. Elevated temperature and durability studies on high strength concrete. CVR Journal of Science and Technology. 2018; 15:10-6.

[35] Bastami M, Baghbadrani M, Aslani F. Performance of nano-silica modified high strength concrete at elevated temperatures. Construction and Building Materials. 2014; 68:402-8.

[36] Shaikh F, Haque S. Effect of nano silica and fine silica sand on compressive strength of sodium and potassium activators synthesised fly ash geopolymer at elevated temperatures. Fire and Materials. 2018; 42(3):324-35.

[37] Awal AA, Shehu IA, Ismail M. Effect of cooling regime on the residual performance of high-volume palm oil fuel ash concrete exposed to high temperatures. Construction and Building Materials. 2015; 98:875-83.

[38] Demez A, Karakoç MB. Mechanical properties of high strength concrete made with pyrophyllite aggregates exposed to high temperature. Structural Concrete. 2021; 22(1):E769-78.

[39] Ghadzali NS, Ibrahim MH, Sani MM, Jamaludin N, Desa MS, Misri Z. Properties of concrete containing different type of waste materials as aggregate replacement exposed to elevated temperature—a review. In: IOP Conference series: earth and environmental science 2018 (pp.1-10). IOP Publishing.

[40] Demirel B, Keleştemur O. Effect of elevated temperature on the mechanical properties of concrete produced with finely ground pumice and silica fume. Fire Safety Journal. 2010; 45(6-8):385-91.

[41] Sancak E, Sari YD, Simsek O. Effects of elevated temperature on compressive strength and weight loss of the light-weight concrete with silica fume and superplasticizer. Cement and Concrete Composites. 2008; 30(8):715-21.

[42] Aköz F, Yüzer N, Koral S. The influence of high temperature on the physical and mechanical properties of ordinary portland cement and silica fume mortar. Technical Journal-Tommob Chamber of Civil Engineers. 1995; 6:287-92.

[43] Kahlil W, Kodur V. High temperature mechanical properties of high-strength fly ash concrete with and without fibers. ACI Materials Journal. 2012; 109(6):665-74.

[44] Poon CS, Azhar S, Anson M, Wong YL. Comparison of the strength and durability performance of normal- and high-strength pozzolanic concretes at elevated temperatures. Cement and Concrete Research. 2001; 31(9):1291-300.

[45] Chen B, Liu J. Residual strength of hybrid-fiber-reinforced high-strength concrete after exposure to high temperatures. Cement and Concrete Research. 2004; 34(6):1065-9.

[46] Neville AM, Brooks JJ. Concrete technology. England: Longman Scientific & Technical; 1987.

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### Appendix 1

| S. No | Abbreviations | Description |
|-------|---------------|-------------|
| 1     | AI            | Alcofime    |
| 2     | Al₂O₃         | Aluminium oxide |
| 3     | Al₂O₃         | Chloride free, superplasticising admixture |
| 4     | Ca(OH)₂       | Calcium hydroxide |
| 5     | CaO           | Calcium oxide |
| 6     | Fe₂O₃         | Ferric oxide |
| 7     | IS 12269-1987 | Indian standards for cement and concrete |
| S. No | Abbreviations | Description |
|-------|---------------|-------------|
| 1     | IS 4031-1988  |             |
| 2     | IS 2386:1963  |             |
| 3     | IS 10262:2019 |             |
| 4     | IS 456:2005   |             |
| 5     | IS 516-1959   |             |
| 6     | M4A1Ns        | M40 grade concrete mix using alcocine, and nano-silica as supplementary cementitious material |
| 7     | M5A1Ns        | M50 grade concrete mix using alcocine, and nano-silica as supplementary cementitious material |
| 8     | M6A1Ns        | M60 grade concrete mix using alcocine, and nano-silica as supplementary cementitious material |
| 9     | M4            | M40 grade concrete mix without any supplementary cementitious material |
| 10    | M5            | M50 grade concrete mix without any supplementary cementitious material |
| 11    | M6            | M60 grade concrete mix without any supplementary cementitious material |
| 12    | MgO           | Magnesium oxide |
| 13    | Ns            | Nano-silica |
| 14    | RCS           | Residual compressive strength |
| 15    | SCM           | Supplementary cementitious material |
| 16    | SO₃           | Sulfur trioxide |
| 17    | SiO₂          | Silicon dioxide |
| 18    | TiO₂          | Titanium dioxide |