Data Article

Experimental dataset on the residual performance of fiber-reinforced cementitious composite subjected to high temperature

Paulo Roberto Ribeiro Soares Junior\textsuperscript{a,}\textsuperscript{*}, Priscila de Souza Maciel\textsuperscript{b}, Elaine Carballo Siqueira Correa\textsuperscript{a}, Augusto Cesar da Silva Bezerra\textsuperscript{a,b,}\textsuperscript{*}

\textsuperscript{a} Department of Materials Engineering, Federal Center for Technological Education of Minas Gerais (CEFET-MG), Avenida Amazonas, 5253, Belo Horizonte, MG 30421-169, Brasil

\textsuperscript{b} Department of Transport Engineering, Federal Center for Technological Education of Minas Gerais (CEFET-MG), Avenida Amazonas, 5253, Belo Horizonte, MG 30421-169, Brasil

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Dataset link: Raw data on fiber-reinforced cementitious composite subjected to high temperature (Original data)

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\textbf{Keywords:}
Fiber-reinforced cementitious composite
High temperature
Flexural behavior
Pull-out response
Fiber-matrix analysis

\textbf{A B S T R A C T}

The present dataset refers to the research article entitled “A multiscale investigation on the performance improvement of fiber-reinforced cementitious composites after exposure to high temperatures” [1]. Supplementary data on raw materials characterization, temperature recording, mass loss, water absorption, compressive strength, flexural behavior, pull-out response, fiber-matrix interface, and surface, microstructure and hardness of fibers are presented here. The continuous matrix was produced from cementitious grout containing Portland cement, sand, silica fume, superplasticizer, and water. The heating was carried out in an electric oven up to 260 °C. The bending tests were performed for fiber-reinforced cementitious composite (FRCC) with steel fiber contents of 1\%, 3\%, and 5\% by volume, and for non-fibrous matrix. The pull-out test was performed using single fiber embedded in the matrix. The water absorption and axial compression tests was performed for non-fibrous matrix. The fiber-matrix analysis was performed from polished sections of fibers embedded in cementitious matrix. The fiber analysis was performed
Specifications Table

| Subject | Civil and Structural Engineering |
|---------|----------------------------------|
| Specific subject area | Construction materials |
| Type of data | Table |
| | Image |
| | Graph |
| | Figure |

How the data were acquired

The data presented in this article were obtained from the following laboratory tests:

- Temperature measurement: K-type thermocouples
- 4-point bending test: UTM EMIC, 23DL (300 kN load cell)
- Axial compression test: UTM EMIC, 23DL (300 kN load cell)
- Single fiber pull-out test: UTM EMIC, 23DL (20 kN load cell)
- Nanoindentation test: Microdurometer Shimadzu, HMV
- Image analysis: Optical microscopy Kontrol, IM713

*UTM – Universal Test Machine

Data format

Raw
Analysed
Filtered

Parameters for data collection

The age of 7 days was used for all tests. The high temperature submission was set at 260 ± 1°C. For FRCC, fiber contents of 1%, 3% and 5% by volume were used.

Description of data collection

The data were collected from the test of specimens, produced through the methodology of slurry infiltrated fibers for bending, cementitious grout (non-fibrous) for compression and water absorption, and single fibers for pull-out. The fiber characterization was performed from embedded fibers (microstructure and hardness) and individual fibers (surface). The fiber-matrix interface was evaluated from fibers immersed in the cement matrix. Half of the specimens were exposed to high temperature and tested in a residual condition, after slow cooling. The other half was tested without heating.

Data source location

Institution: Federal Centre for Technological Education of Minas Gerais
City/Town/Region: Belo Horizonte, Minas Gerais
Country: Brazil

Data accessibility

With this article and online Mendeley Data:

Raw data on fiber-reinforced cementitious composite subjected to high temperature
URL: https://data.mendeley.com/datasets/hc3bct2byc
doi: 10.17632/hc3bct2byc.1

Analyzed data on fiber-reinforced cementitious composite subjected to high temperature
URL: https://data.mendeley.com/datasets/tb6bkc3g5m
doi: 10.17632/tb6bkc3g5m.1

Related research article

[1] P.R.R. Soares Junior, P. de S. Maciel, E.C.S. Corrêa, A.C. da S. Bezerra, A multiscale investigation on the performance improvement of fiber-reinforced cementitious composites after exposure to high temperatures, Cem Concr Compos. 133 (2022) 104,657.
doi: https://doi.org/10.1016/j.cemconcomp.2022.104657

from steel fibers. The data refer to the residual properties after heating and slow cooling or to the reference condition without heating. The data can help in understanding residual performance of FRCC after exposure to high temperatures and may be useful for developing resilient building materials. © 2022 The Author(s). Published by Elsevier Inc. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/)
Value of the Data

- These data are important for predicting and understanding the behavior of fiber reinforced cementitious composites (FRCCs) subjected to high temperatures.
- The data was obtained from experimental tests at different levels of analysis (multiscale – macro, meso and microstructural). This approach provides a valuable dataset for developing resilient materials.
- Engineers, designers, and researchers can benefit from this data, particularly from the residual performance improvement after high temperatures.
- Researchers working on the development of materials optimized for severe temperatures would recognize the value of this data.
- This dataset can be useful in the design of fire-resistant structures and development of heat-cured precast structures.
- The data can be useful to calibrate, verify, and validate numerical and analytical models in predicting the behavior of FRCCs under fire conditions.

1. Data Description

The data presented in this dataset refers to the supplementary data from the article entitled “A multiscale investigation on the performance improvement of fiber-reinforced cementitious composites after exposure to high temperatures”. This section has been divided into subsections related to: Raw materials characterization (1.1); Ambient temperature record (1.2); Mass loss (1.3); Water absorption (1.4); Compressive strength (1.5); Flexural behavior (1.6); Pull-out response (1.7); Fiber microstructure (1.8); Fiber hardness (1.9); Fiber surface (1.10); and Fiber-matrix interface (1.11). The no-heat condition was defined as AM and the data obtained after submission to high temperature of HT. The raw and analyzed data are available in Mendeley Data (see Ref [2,3]).

1.1. Raw materials characterization

Tables 1-5 show the detailed characterization of the raw materials used. All data were obtained from manufacturers and suppliers.

Table 1 shows the physical-chemical characteristics of Portland cement, as well as tests performed and methodology adopted, with 0.57% (<1.0) of insoluble residue, 3.75% (<4.5) of loss of ignition, 1.48% (<6.5) of magnesium oxide, 2.73% (<4.5) of sulfur oxide, 2.61 (<3.0) of carbon

| Tests                    | Methodology | Unit | Result | Requirement |
|--------------------------|-------------|------|--------|-------------|
| Insoluble residue        | ABNT NM 15/12 | %    | 0.57   | ≤ 1.0       |
| Loss of ignition         | ABNT NM 18/12 | %    | 3.75   | ≤ 4.5       |
| Magnesium oxide          | ABNT NM 21/12 | %    | 1.48   | ≤ 6.5       |
| Sulfur oxide             | ABNT NM 16/12 | %    | 2.73   | ≤ 4.5       |
| Carbon dioxide           | ABNT NM 20/12 | %    | 2.61   | ≤ 3.0       |
| Surface area (Blaine)    | ABNT NM 76/98 | cm²/g | 4507   | < 3000      |
| Sieve residue #200       | ABNT NBR 11579/91 | %    | 0.06   | ≤ 6.0       |
| Sieve residue #325       | ABNT NBR 9202/85 | %    | 0.87   | –           |
| Normal consistency water | ABNT NM 43/03 | %    | 30.4   | –           |
| Setting start            | ABNT NM 65/03 | min  | 142    | ≤ 60        |
| Setting end              | ABNT NM 65/03 | min  | 191    | ≤ 600       |
| Expandability            | ABNT NBR 11582/91 | mm  | 0.00   | ≤ 5.0       |

Source: Brennand cements.
Table 2
Characteristics of steel fiber.

| Characteristic                                      | Steel fiber                          |
|-----------------------------------------------------|--------------------------------------|
| Type / nomenclature by manufacturer                 | FF3                                  |
| Material                                            | Low carbon steel                     |
| Manufacturing method                                | Cold forming (drawing)               |
| Section shape                                       | Circular                             |
| Length (mm)                                         | 50                                   |
| Diameter (mm)                                       | 0.75                                 |
| l/d (form factor)                                   | 67                                   |
| Tensile strength (limit of resistance)              | 1200 MPa                             |

Source: Maccaferri.

Table 3
Chemical composition of silica fume.

| Chemical analysis, % | Silica fume |
|----------------------|-------------|
| Silicon dioxide (SiO₂) | 91.04       |
| Aluminum oxide (Al₂O₃) | 0.10        |
| Iron oxide (Fe₂O₃)    | 0.70        |
| Calcium oxide (CaO)   | 1.10        |
| Magnesium oxide (MgO) | 1.50        |
| Sodium oxide (Na₂O)   | 0.39        |
| Potassium oxide (K₂O) | 4.40        |
| Sulfur trioxide (SO₃) | 0.16        |
| Loss of ignition      | 0.61        |

Source: Tecnosil.

Table 4
Properties of superplasticizer.

| Property                | Result                  |
|-------------------------|-------------------------|
| Aspect                  | Viscous liquid          |
| Color                   | Sienna                  |
| Homogeneity             | Homogeneous             |
| Specific mass           | 1.09 g/cm³              |
| Solid residue content   | 47.8%                   |
| pH                      | 3.1                     |
| Chloride content        | ≤ 0.15%                 |

Source: Tecnosil.

Table 5
Properties of water.

| Property    | Result                           |
|-------------|----------------------------------|
| Color       | Colourless                       |
| Turbidity   | Clear                            |
| Temperature | Ambient, around 25 °C            |
| pH          | 7.83                             |
| Chloride    | 1.09 mg/L                        |
| Fluoride    | 0.8 mg/L                         |

Source: Copasa.
dioxide, 4.507 cm²/g (>3000) of surface area (Blaine), 0.06% (<6.0) of sieve residue #200, 0.87% of residue on the #323 sieve, 30.4% of normal consistency water, 142 min of start setting (> 60), 191 min of end setting (<600) and 0.00 mm of hot expandability (<5.0).

Table 2 shows the characteristics of steel fibers. The fiber nomenclature was designated as (FF3) according to the manufacturer, the material is low carbon steel, the manufacturing method was cold-forming (drawing), the section shape is circular with length (l) of 50 mm and diameter (d) 0.75 mm, form factor equals to 67 (l/d) and tensile strength (limit of strength) of 1200 MPa.

Table 3 shows the chemical composition of silica fume, which had 91.04% silicon dioxide, 0.10% aluminum oxide, 0.70% iron oxide, 1.10% calcium oxide, 1.50% magnesium oxide, 0.39% sodium oxide, 4.40% potassium oxide, 0.16% sulfur trioxide and 0.61% loss of ignition.

Table 4 shows the properties of the superplasticizer, which has a homogeneous viscous liquid appearance, siena color, 47.8% solid residue content, 3.1 pH and ≤0.15% chloride content.

Table 5 shows the properties of the water used in the mixture of the fluid mortar, which had a colorless and clear appearance, at room temperature around 25 °C, 1.09 mg/L of chloride and 0.8 mg/L of fluoride.

1.2. Ambient temperature record

Fig. 1 shows the record of ambient temperature during submission of specimens to high temperature. The data are in the range between 23.4 °C and 25.6 °C, over approximately 120 min of measurement.

![Fig. 1. Record of ambient temperature.](image)

1.3. Mass loss

Tables 6 and 7 show the raw data for mass loss and relative mass loss, both due to high temperature submission. The average mass loss of the six specimens was 19.17 g after drying in an oven and 7.50 g after heat treatment. The total mass loss was 26.66 g. The average relative mass loss was 4.88% after oven drying, 2.02% after heat treatment and 6.90% in total.
Table 6
Data of mass loss.

| Sample | ML - OV (g) | ML - HT (g) | ML - Total |
|--------|-------------|-------------|------------|
| 1      | 18.85       | 7.84        | 26.69      |
| 2      | 19.97       | 7.68        | 27.65      |
| 3      | 18.93       | 8.13        | 27.06      |
| 4      | 19.56       | 8.32        | 27.88      |
| 5      | 18.81       | 7.02        | 25.83      |
| 6      | 18.87       | 5.99        | 24.86      |

Average 19.17 7.50 26.66

Standard deviation 0.44 0.78 1.04
Coefficient of variation 0.023 0.10 0.039

Note: ML – mass loss; OV – oven; HT – heat treatment.

Table 7
Data of relative mass loss.

| Sample | ML - OV (%) | ML - HT (%) | ML - Total (%) |
|--------|-------------|-------------|----------------|
| 1      | 4.72        | 2.06        | 6.78           |
| 2      | 5.21        | 2.11        | 7.32           |
| 3      | 4.74        | 2.14        | 6.88           |
| 4      | 5.06        | 2.27        | 7.33           |
| 5      | 4.83        | 1.89        | 6.73           |
| 6      | 4.73        | 1.57        | 6.31           |

Average 4.88 2.01 6.90

Standard deviation 0.20 0.24 0.38
Coefficient of variation 0.04 0.12 0.05

Note: ML – mass loss; OV – oven; HT – heat treatment.

I.4. Water absorption

Tables 8, 9 and 10 show the data from the water absorption test.

Table 8
Saturated, dry, and submerged mass, before and after heat treatment.

| Sample | AM M_{ssd} (g) | M_{dry} (g) | M_{sub} (g) | HT M_{ssd} (g) | M_{dry} (g) | M_{sub} (g) |
|--------|----------------|-------------|-------------|----------------|-------------|-------------|
| 1      | 399.25         | 380.40      | 209.24      | 393.21         | 372.56      | 202.85      |
| 2      | 383.17         | 363.20      | 191.67      | 375.11         | 355.52      | 183.34      |
| 3      | 398.70         | 379.77      | 207.86      | 393.53         | 371.64      | 203.05      |
| 4      | 385.89         | 366.33      | 194.96      | 378.50         | 358.01      | 188.68      |
| 5      | 389.02         | 370.21      | 198.46      | 382.10         | 363.19      | 192.52      |
| 6      | 398.38         | 379.51      | 207.73      | 391.06         | 373.52      | 200.94      |

Note: M_{dry} – dry mass; M_{ssd} – saturated mass dry-surface; M_{sub} – submerged-saturated mass.
Table 9
Data of water absorption, dry bulk density, saturated bulk density and porosity for the unheated condition.

| Sample | WA (%) | DBD (g/cm³) | SBD (g/cm³) | PR (%) |
|--------|--------|-------------|-------------|--------|
| 1      | 4.96   | 2.00        | 2.10        | 9.25   |
| 2      | 5.50   | 1.90        | 2.00        | 9.44   |
| 3      | 4.98   | 1.99        | 2.09        | 9.02   |
| 4      | 5.34   | 1.92        | 2.02        | 9.29   |
| 5      | 5.08   | 1.94        | 2.04        | 8.98   |
| 6      | 4.97   | 1.99        | 2.09        | 9.00   |
| Average| 5.14   | 1.96        | 2.06        | 9.13   |
| Standard deviation | 0.2272 | 0.0437 | 0.0448 | 0.1915 |
| Coefficient of variation | 0.0442 | 0.0224 | 0.0203 | 0.0210 |

Note: WA - water absorption; DBD - dry bulk density; SBD - saturated bulk density; PR - porosity.

Table 10
Data of water absorption, dry bulk density, saturated bulk density and porosity after high temperature.

| Sample | WA (%) | DBD (g/cm³) | SBD (g/cm³) | PR (%) |
|--------|--------|-------------|-------------|--------|
| 1      | 5.54   | 1.96        | 2.07        | 9.78   |
| 2      | 5.51   | 1.85        | 1.96        | 9.26   |
| 3      | 5.89   | 1.95        | 2.07        | 10.30  |
| 4      | 5.72   | 1.89        | 1.99        | 9.74   |
| 5      | 5.21   | 1.92        | 2.02        | 9.06   |
| 6      | 4.70   | 1.96        | 2.06        | 8.44   |
| Average| 5.43   | 1.92        | 2.03        | 9.44   |
| Standard deviation | 0.4256 | 0.0444 | 0.0451 | 0.6508 |
| Coefficient of variation | 0.0784 | 0.0231 | 0.0222 | 0.0690 |

Note: WA - water absorption; DBD - dry bulk density; SBD - saturated bulk density; PR - porosity.

Table 8 shows the weighing data of the six specimens used, in the following ways: dry mass in oven or after HT ($M_{dry}$), saturated mass dry-surface ($M_{ssd}$) and submerged-saturated mass ($M_{sub}$). For the AM condition, the $M_{dry}$ data are comprised between 363.20 g and 380.40 g, $M_{ssd}$ between 383.17 g and 399.25 g, and $M_{sub}$ between 191.67 g and 209.24 g. For the HT condition, the $M_{dry}$ data are comprised between 355.52 g and 373.53 g, $M_{ssd}$ 375.11 g and 393.53 g, and $M_{sub}$ between 183.34 g and 202.85 g.

Table 9 shows the water absorption (WA), dry bulk density (DBD), saturated bulk density (SBD) and porosity (PR) data for the unheated condition. The WA data are between 4.96% and 5.50%, average of 5.14%. DBD data are between 1.90 g/cm³ and 2.00 g/cm³, average of 1.96 g/cm³. The SBD data are between 2.00 g/cm³ and 2.10 g/cm³, average of 2.06 g/cm³. PR data are between 8.98% and 9.44%, average of 9.13%.

Table 10 shows the WA, DBD, SBD and PR data after high temperature submission. The WA data are between 4.70% and 5.89%, average of 5.43%. DBD data are between 1.85 g/cm³ and 1.96 g/cm³, average of 1.92 g/cm³. The SBD data are between 1.96 g/cm³ and 2.07 g/cm³, average of 2.03 g/cm³. PR data are between 8.44% and 10.30%, average of 9.44%.

1.5. Compressive strength

Table 11 shows the compressive strength data of the cementitious matrix (non-fibrous). For the AM condition, the strength was between 67.05 MPa and 83.87 MPa, average of 70.53 MPa and standard deviation of 3.4119 MPa. For HT, the strength was between 85.51 MPa and 89.66 MPa, mean of 88.07 MPa and standard deviation of 2.2385 MPa.
Table 11
Compressive strength of cementitious matrix.

| Condition | Sample | Strength | Average | Standard deviation |
|-----------|--------|----------|---------|--------------------|
| AM        | SP1    | 67.05    | 70.53   | 3.4119             |
|           | SP2    | 70.66    |         |                    |
|           | SP3    | 73.87    |         |                    |
| HT        | SP1T   | 85.51    | 88.07   | 2.2385             |
|           | SP2T   | 89.04    |         |                    |
|           | SP3T   | 89.66    |         |                    |

Fig. 2. Flexural behavior of (a) F1AM and (b) F1HT.

Fig. 3. Flexural behavior of (a) F3AM and (b) F3HT.

1.6. Flexural behavior

Figs. 2, 3 and 4 show the flexural behavior of FRCC without heating (AM) and after heat treatment (HT). The fiber content is designated as “F”, that is, 1%, 3% and 5% of fibers correspond to F1, F3 and F5. Data from two specimens is shown for each condition.
Tables 12-15 show the bending behavior parameters, in terms of load, strength, deflection and toughness, for three specimens in each condition. The respective average values and standard deviations are also shown.

Table 12 shows the load (P_max), strength (σ_P,max) and deflection (δ_P,max) corresponding to the maximum load or peak load. The maximum load remained between 1511 N (non-fibrous matrix, without heating) to 28,205 N (FRCC with 5% fiber, after heat treatment). The strength started at 3.12 MPa and went up to 58.17 MPa, while the deflection was from 0.049 mm to 1401 mm, for the same composites and conditions (matrix-AM and F5HT).

Table 13 shows the toughness before the peak, at the point corresponding to 0.9 of the maximum load (T_{0.9P,pp}). The toughness of the cementitious (non-fibrous) matrix was negligible. Toughness started at 125 N.mm (FRCC with 1% fibers, without heating) and went up to 12,984 N.mm (FRCC with 5% fibers, after heat treatment).

Table 14 shows the toughness at the point corresponding to the peak load (T_{P,pc}). The toughness of the cementitious (non-fibrous) matrix was negligible. Toughness started at 418 N.mm (FRCC with 1% fibers, without heating) and went up to 23,499 N.mm (FRCC with 5% fibers, after heat treatment).

Table 15 shows the toughness after the peak, at the points corresponding to 0.9, 0.7 and 0.5 of the maximum load (T_{0.9P,pp}, T_{0.7P,pp} and T_{0.5P,pp}). For T_{0.9P,pp} the toughness was between 637 N.mm and 37,226 N.mm; for T_{0.7P,pp} between 1086 N.mm and 49,876 N.mm; and for T_{0.5P,pp} between 1276 N.mm and 67,297 N.mm; all in relation to F1AM and F5HT, respectively.

![Fig. 4. Flexural behavior of (a) F5AM and (b) F5HT.](image)

| Sample | P_max (N) | σ_P,max (MPa) | δ_P,max (mm) |
|--------|-----------|---------------|---------------|
|        | P_max Md  | σ_P,max Md    | δ_P,max Md    | σ_P,max Sd | δ_P,max Sd |
|        | Md Sd     | Sd            | Sd            | Sd         | Sd         |
| Matrix-AM | SP1 1814 | 3.74          | 0.069         | 0.010      |
|         | SP2 1764 | 3.64          | 0.057         | 0.049      |
|         | SP3 1511 | 3.12          | 0.157         | 0.039      |
| F1AM    | SP1 4248 | 8.76          | 0.157         | 0.039      |
|         | SP2 3609 | 7.44          | 0.233         | 0.039      |
|         | SP3 4329 | 8.93          | 0.180         |           |

(continued on next page)
### Table 12 (continued)

| Sample  | $P_{\text{max}}$ (N) | $\sigma_{P_{\text{max}}}$ (MPa) | $\delta_{P_{\text{max}}}$ (mm) |
|---------|---------------------|--------------------------|--------------------------|
|         | $P_{\text{max}}$ | Md | Sd | $\sigma_{P_{\text{max}}}$ | Md | Sd | $\delta_{P_{\text{max}}}$ | Md | Sd |
| F3AM    | SP1                | 9662 | 9726 | 294 | 19.93 | 20.06 | 0.61 | 0.338 | 0.317 | 0.049 |
|         | SP2                | 9469 | 9726 | 294 | 19.53 | 20.06 | 0.61 | 0.352 | 0.260 |
|         | SP3                | 10047 | 20.72 | 0.61 | 0.338 | 0.317 | 0.049 |
| F5AM    | SP1                | 12115 | 11473 | 1421 | 24.99 | 23.66 | 2.93 | 0.643 | 0.424 | 0.190 |
|         | SP2                | 9844 | 20.30 | 0.352 |
|         | SP3                | 12460 | 25.70 | 0.310 |
| Matrix-HT | SP1            | 2312 | 2160 | 158 | 4.77 | 4.46 | 0.326 | 0.095 | 0.104 | 0.008 |
|         | SP2                | 2170 | 4.12 | 0.106 |
|         | SP3                | 1997 | 4.12 | 0.106 |
| F1HT    | SP1                | 7117 | 6113 | 887 | 14.68 | 12.61 | 1.83 | 0.640 | 0.434 | 0.191 |
|         | SP2                | 5434 | 11.21 | 0.399 |
|         | SP3                | 5789 | 11.94 | 0.262 |
| F3HT    | SP1                | 14356 | 13876 | 2509 | 29.61 | 28.62 | 5.18 | 0.610 | 0.710 | 0.176 |
|         | SP2                | 16110 | 33.23 | 0.607 |
|         | SP3                | 11162 | 23.02 | 0.607 |
| F5HT    | SP1                | 24525 | 23849 | 4730 | 50.58 | 49.19 | 9.75 | 1.401 | 1.180 | 0.217 |
|         | SP2                | 16110 | 33.23 | 0.607 |
|         | SP3                | 11162 | 23.02 | 0.607 |

Notes: $P_{\text{max}} = P$ – maximum load or peak load; $\sigma_{P_{\text{max}}}$ – strength; $\delta_{P_{\text{max}}}$ – deflection at peak load; Md – average; Sd – standard deviation.

### Table 13

| Sample  | $T_{0.9P_{\text{bp}}}$ (N.mm) |
|---------|-------------------------------|
|         | $T_{0.9P_{\text{bp}}}$ | Md | Sd |
| Matrix-AM | SP1 | – | – | – |
|         | SP2 | – | – | – |
|         | SP3 | – | – | – |
| F1AM    | SP1 | 3823 | 217 | 216 | 90 |
|         | SP2 | 3248 | 125 |
|         | SP3 | 3896 | 305 |
| F3AM    | SP1 | 8696 | 965 | 1016 | 164 |
|         | SP2 | 8522 | 1199 |
|         | SP3 | 9042 | 884 |
| F5AM    | SP1 | 10904 | 2351 | 1610 | 646 |
|         | SP2 | 8860 | 1162 |
|         | SP3 | 11214 | 1317 |
| Matrix-HT | SP1 | – | – | – |
|         | SP2 | – | – | – |
|         | SP3 | – | – | – |
| F1HT    | SP1 | 6405 | 1228 | 829 | 347 |
|         | SP2 | 4891 | 657 |
|         | SP3 | 5210 | 602 |
| F3HT    | SP1 | 12920 | 3731 | 3765 | 1098 |
|         | SP2 | 14499 | 4880 |
|         | SP3 | 10046 | 2685 |
| F5HT    | SP1 | 22073 | 11984 | 10526 | 3429 |
|         | SP2 | 25385 | 12984 |
|         | SP3 | 16935 | 6609 |

Notes: $P_{\text{max}} = P$ – maximum load or peak load; $0.9P_{\text{bp}}$ – point before the peak, which the load is 0.9 of the maximum load; $T_{0.9P_{\text{bp}}}$ – toughness at point 0.9P_{bp}; Md – average; Sd – standard deviation.
Table 14

Toughness at peak load.

| Sample   | Ppc          | Tpc (N.mm) | Tpc | Md | Sd |
|----------|--------------|------------|-----|----|----|
| Matrix-AM|              |            |     |    |    |
| SP1      | –            | –          | –   | –  | –  |
| SP2      | –            | –          | –   | –  | –  |
| SP3      | –            | –          | –   | –  | –  |
| F1AM     | 4248         | 418        | 534 | 131|
| F3AM     | 9662         | 2157       | 1955| 324|
| F5AM     | 12115        | 5324       | 3180| 1870|
| Matrix-HT|              |            |     |    |    |
| SP1      | –            | –          | –   | –  | –  |
| SP2      | –            | –          | –   | –  | –  |
| SP3      | –            | –          | –   | –  | –  |
| F1HT     | 7117         | 3386       | 1892| 1307|
| F3HT     | 14356        | 5205       | 6633| 2986|
| F5HT     | 24525        | 23499      | 18670| 5880|

Notes: \( P_{\text{max}} = P \) – maximum load or peak load; \( P_{\text{bp}} \) – point at peak load; \( T_{0.9P,\text{pp}} \) – toughness at point \( P_{\text{bp}} \); Md – average; Sd – standard deviation.

Table 15

Toughness post-peak at 0.9P, 0.7P and 0.5P

| Sample   | 0.9P,pp   | 0.9P,pp (N.mm) | 0.7P,pp | 0.7P,pp (N.mm) | 0.5P,pp | 0.5P,pp (N.mm) |
|----------|-----------|----------------|----------|----------------|----------|----------------|
| Matrix-AM|           |                |          |                |          |                |
| SP1      | –         | –              | –        | –              | –        | –              |
| SP2      | –         | –              | –        | –              | –        | –              |
| SP3      | –         | –              | –        | –              | –        | –              |
| F1AM     | 3823      | 637            | 1022     | 450            | 2974     | 1174           |
| F3AM     | 8696      | 3452           | 3771     | 1673           | 6763     | 14893          |
| F5AM     | 10904     | 10002          | 6339     | 3434           | 8481     | 18486          |
| Matrix-HT|           |                |          |                |          |                |
| SP1      | –         | –              | –        | –              | –        | –              |
| SP2      | –         | –              | –        | –              | –        | –              |
| SP3      | –         | –              | –        | –              | –        | –              |
| F1HT     | 6405      | 3889           | 2674     | 1327           | 4982     | 5203           |
| F3HT     | 12920     | 7617           | 10572    | 5653           | 10049    | 11010          |
| F5HT     | 232073    | 30989          | 29119    | 9185           | 37168    | 42073          |

Notes: \( P_{\text{max}} = P \) – maximum load or peak load; 0.9P, 0.7P, 0.5P – points after the peak load, where the load is 0.9, 0.7 and 0.5 of the maximum load; \( T_{0.9P,\text{pp}} \), \( T_{0.7P,\text{pp}} \) and \( T_{0.5P,\text{pp}} \) – toughness at points 0.9P, 0.7P and 0.5P; Md – average; Sd – standard deviation.
1.7. Pull-out response

Fig. 5 shows the single fiber pull-out response. Two load-slip (P-s) curves are shown for each condition, each curve from one specimen. The blue data represents the AM condition and the red data the HT condition. PL means pull-out.

Table 16 shows the pull-out test parameters. The maximum load was between 412.75 N and 395.40 N without heating, and between 450.75 N and 479.01 N after heat treatment. The displacement in peak load was on average 1.2364 mm for AM and 1.3415 mm for HT. The pull-out energy was between 3140.53 N.mm and 4044.96 N.mm for AM, and between 3490.56 N.mm and 4293.77 N.mm after heating.

![Fig. 5. Load-slip curves of pull-out response (a) without heating and (b) after thermal treatment.](image-url)
### Table 16
Parameters of pull-out response.

| Sample | $P_{\text{max}}$ (N) | $S_{\text{p, max}}$ (mm) | $E_p$ (N·mm) | $P_{\text{max}}$ (N) | Sd (N) | $S_{\text{p, max}}$ (mm) | Sd (mm) | $E_p$ (N·mm) | Sd (N·mm) |
|--------|-----------------|-----------------|-------------|-----------------|------|-----------------|------|-------------|------|
| PL_AM  | 405.59          | 1.5494          | 3140.53     | 404.58          | 8.718| 1.2364          | 0.3390| 3504.30     | 477.45|
| CP1    | 412.75          | 1.2836          | 4044.96     |                 |      |                 |      |             |      |
| CP2    | 395.40          | 0.8763          | 3327.43     |                 |      |                 |      |             |      |
| PL_HT  | 460.74          | 1.7399          | 4059.21     | 463.5           | 14.330| 1.3415         | 0.5735| 3947.84     | 413.02|
| CP1    | 479.01          | 1.6005          | 4293.77     |                 |      |                 |      |             |      |
| CP2    | 450.75          | 0.6842          | 3490.56     |                 |      |                 |      |             |      |

Notes: $P_{\text{max}}$ - maximum load; $S_{\text{p, max}}$ - displacement at the point of maximum load; $E_p$ - pull-out energy; Sd - standard deviation.

### 1.8. Fiber microstructure

Fig. 6 shows the specimens for metallography and nanoindentation tests. Figs. 7, 8 and 9 show optical microscopy (OM) images of the steel fiber microstructure as received, after bending test and after high temperature submission, respectively.

![Fig. 6. Samples for metallography and nanoindentation.](image-url)
Fig. 7. OM images of fibers “as received”.

Fig. 8. OM images of fibers after bending test.
Fig. 9. OM images of fibers after heat treatment.

1.9. Fiber hardness

Fig. 10 shows an image obtained by OM during the nanoindentation test, in which the typical microstructure of steel fibers and the impressions left by the indenter can be seen. Parallel bars were used as a reference.

Table 17 shows the hardness data obtained from six samples tested by nanoindentation. The fibers as received (AR) was an average hardness of 275 MPa. After flexural testing without heating, the average of hardness was 323 MPa. After submission to high temperature, the average of hardness was 277 MPa.

| Measurements | Hardness Vickers (HV 0.2/15) |
|--------------|-----------------------------|
|              | AR     | AM     | HT     |
| 1            | 286    | 327    | 267    |
| 2            | 294    | 342    | 285    |
| 3            | 264    | 337    | 283    |
| 4            | 272    | 313    | 279    |
| 5            | 266    | 302    | 271    |
| 6            | 284    | 318    | 279    |
| Average      | 275    | 323    | 277    |
| Standard deviation | 11.04 | 13.78 | 6.37 |
| Coefficient of variation | 0.04 | 0.04 | 0.02 |

Notes: AR – as received; AM – reference condition; HT – high temperature.
Fig. 10. OM image during nanoindentation test.

Fig. 11. Data of hardness for AR, AM and HT conditions.

Fig. 11 graphically illustrates the hardness data for each condition mentioned.

1.10. Fiber surface

Figs. 12, 13 and 14 show images obtained by OM of the fibers surface, as received, after bending test and after heat treatment, respectively. The x100 and x400 magnifications were used.
Fig. 12. Fiber surface “as received” at (a) x100 and (b) x400 magnification. (Note: modified from [1]).

Fig. 13. Fiber surface after bending test at (a) x100 and (b) x400 magnification. (Note: modified from [1]).

Fig. 14. Fiber surface after heat treatment at (a) x100 and (b) x400 magnification. (Note: modified from [1]).
1.11. Fiber-matrix interface

Fig. 15 shows a specimen prepared for fiber-matrix interface analysis by OM, after sanding and polishing. The fibers sections are the bright spots indicated.

Figs. 16, 17 and 18 show images obtained by OM of the fiber-matrix interface, as received, after heat treatment, and after heat treatment with previous preparation of the samples, respectively. The x100 and x400 magnifications were used.

![Fig. 15. Sample for fiber-matrix interface analysis.](image1)

![Fig. 16. Fiber-matrix interface without heating at x100 magnification. (Note: modified from [1]).](image2)
2. Experimental Design, Materials and Methods

The experimental phase was divided into three major stages. The first consisted in the selection and characterization of materials, as well as in the development of the cementitious composite. In the second stage, the specimens were produced. In the third stage, the specimens were tested to investigate the bending and compression behaviors, water absorption, porosity, density, fiber-matrix interface and surface, microstructure and hardness of the fibers. All tests were performed at the 7 days, before and after heat treatment. For additional information see related article in Ref. [1].

2.1. Molding of specimens

For molding the specimens, the slurry infiltrated fiber method was used, which consists of placing the fibers in the mold and then casting a fluid mortar, which fills the empty spaces between the fibers [4]. Prismatic mold with dimensions of 40 × 40 × 160 mm was used for bending. The fibers were placed in the molds according to the percentages of 1%, 3% and 5% by
volume; finally casting a fluid mortar. The pull-out specimens were produced using cylindrical molds with dimensions of 50 × 100 mm. One of the hook-end of the fibers was sectioned to facilitate fitting into the test equipment. The other end of the fiber was embedded in the matrix. The length of the fibers embedded in the matrix was equal to 15 mm. The same cylindrical molds were used for compression and water absorption. The specimens for microstructural investigation and fiber characterization are described below.

After demolding within 24 h of mixing the materials, the specimens were placed in submerged curing in water saturated with calcium hydroxide, where they remained for seven days. After curing, the specimens were placed in an oven for a period of 24 h, at an average temperature of 65 °C, to remove excess water and carry out the tests. After the oven-drying, the specimens were removed and left at room temperature. Finally, half of the specimens were tested without heating, while the other half was subjected to high temperature and subsequent testing.

To produce cementitious grout, the literature suggests a water-cementitious materials ratio of 0.30; silica fume-cement ratio of 0.20; and sand-cementitious materials ratio of 1.23 \[5\]. Thus, the dosage was adjusted for the raw materials. The sand was dried in an oven for 24 h, at an approximate temperature of 105 °C, to completely remove the moisture. Then, the sieving process was adopted to obtain grains with a maximum dimension of 1.2 mm.

The variables proposed in this study were fibers content (1%, 3% and 5%) and thermal condition (with or without heat treatment). Thus, to differentiate the composites, a nomenclature was set up as follows: fiber percentage (F0, F1, F3 and F5 for 0%, 1%, 3% and 5% of fibers, respectively) and presence (HT) or absence (AM) of heat treatment.

2.2. Heat treatment

The heat treatment (HT) of the specimens was performed in an electric oven, to simulate a controlled condition for temperature variation with time. The temperature measurement was made using thermocouples attached to the specimens and the use of a glass wool thermal blanket. The other end of the thermocouple was connected to the digital meter, which converts the electrical signal into temperature values. To perform the heat treatment, the specimens were inserted in the oven along with sensors to measure the temperature, so that the heat flow occurred equally over the entire surface. Heating in the range of 200 °C to 300 °C promotes maintenance of properties. However, at higher temperatures the properties are deteriorated \[6\]. Around 400 °C, the propagation of micro-cracks in the matrix occurs, compromising the microstructure. At 560 °C, the decomposition of the structures of calcium silicate hydrate (C-S-H) occurs, porosity increases even more, and the microstructure is very cracked, which causes a rapid drop in strength. Thus, a temperature close to 260 °C was chosen to study the properties of FRCC in this specific condition and to evaluate the increment of residual strength.

2.3. Mechanical characterization of cementitious composite

The 4-point bending test was adopted to evaluate the mechanical behavior of FRCC, using a universal testing machine (UTM) from the manufacturer EMiC, located in the Department of Transports Engineering of CEFET/MG. The midpoint deflection was accurately measured by a deflectometer while the loading was measured by a load cell with a capacity of 300 kN. The resulting load and deflection values were captured and analyzed by TESC software, also from EMiC, generating a complete database. These data were collected and analyzed, resulting in the load-deflection (P-d) diagrams for each specimen. The bending stress were calculated using Eq. (1), considering the precepts of the bending test and considering the acting efforts, as follows:

\[
\sigma_{flex}^{4p} = \frac{Pd}{bh^2}
\]  

(1)
Where $\sigma_{\text{Flex}}^{P}$ is the 4-point bending stress, $P$ is the load, $d$ is the span ($d = 132$ mm), $b$ is the width and $h$ is the height ($b = h = 40$ mm).

2.4. Toughness

Toughness was calculated considering the area under the load-deflection curve (P-d). The ASTM C1609/C1609M [7] standard suggests the adoption of notable points to calculate the toughness when the deflections are equal to L/600 and L/150, where L is the distance between the supports. Kim, Naaman and El-Tawil [8] indicate the adoption of the other point (L/100) to fully characterize the behavior of FRCC using different fibers. In view of the conditions, the characteristics of the composite, the different fiber content and the extension of the P-d curves, it was necessary to adopt another notable point corresponding to the loads of 0.9$P$ (before and after peak load), $P$ (load of peak), 0.7$P$ (post-peak) and 0.5$P$ (post-peak). Therefore, the toughness points were $T_{0.9P Ka}$, $T_{P Ka}$, $T_{0.9P Kp}$, $T_{0.7P Kp}$ and $T_{0.5P Kp}$.

2.5. Single fiber pull-out test

The present study adopted a specific setup for single fiber pull-out test, coupled to a universal testing equipment (See related article in Ref. [1]). A load cell with a capacity of 20 kN and accuracy of 0.001 N was used to measure the load. The specimen was attached by an adapter. A clamp was used to pull-out the fiber. To measure the fiber displacement accurately, an extensometer with accuracy of 0.001 mm was used. The adopted displacement rate was 10 $\mu$m/s, in agreement with Abdallah, Fan and Cashell [9].

2.6. Compressive strength of cementitious matrix

The mechanical strength of the cementitious matrix was evaluated by the axial compression test. Six cylindrical specimens were cast, half of which underwent heat treatment. All specimens were tested in compression using the universal testing equipment.

2.7. Fiber-matrix interface analysis

The fiber-matrix interface analysis was performed using a Kontrol optical microscope (MO), IM713. The fiber-matrix interface was studied using polished sections by MO to reveal the contour of the interface.

2.8. Characterization of steel fibers

The steel fibers were characterized under three different conditions. The first refers to the fiber as received, collected in the plastic container for conditioning materials. The second condition represents the fiber after the bending test, collected from the fractured specimen. The third condition represents the fiber subjected to heat treatment and after bending test. All fibers were chosen randomly, to obtain a reliable sample. Then, the fibers were divided in half with cutting pliers and inserted in silicone molds for cold mounting, which was done with transparent acrylic resin. The samples were prepared by sanding and polishing, then chemically attacked (Nital 3% reagent) to reveal the microstructure of the steel. The samples were taken under the Kontrol optical microscope, IM713, to obtain images of the microstructure of the steel. Finally, the samples were placed in the Shimadzu microdurometer, HMV, to evaluate the Vickers microhardness of
the steel fibers. The microhardness test was performed with a load application of 1961 N (HV 0.2) and a holding time of 15 s.

Ethics Statements

This work adheres to ethical publishing standards and does not include human studies, animal experiments or data collected from social media platforms.

CRediT Author Statement

Paulo Roberto Ribeiro Soares Junior: Investigation; Data Curation; Writing – original draft preparation, Writing – review & editing; Priscila de Souza Maciel: Methodology, Writing – original draft preparation; Elaine Carballo Siqueira Correa: Methodology, Resources; Augusto Cesar da Silva Bezerra: Conceptualization, Supervision, Project administration, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Raw data on fiber-reinforced cementitious composite subjected to high temperature (Original Data) (Mendeley Data)

Analyzed data on fiber-reinforced cementitious composite subjected to high temperature (Original Data) (Mendeley Data).

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