

INFLUENCE OF HIGH TEMPERATURES ON THE COMPRESSIVE STRENGTH OF IRON ORE TAILINGS/CEMENT COMPOSITE

INFLUÊNCIA DAS ALTAS TEMPERATURAS NA RESISTÊNCIA À COMPRESSÃO DO COMPÓSITO REJEITO DE MINÉRIO DE FERRO/CIMENTO

INFLUENCIA DE LAS ALTAS TEMPERATURAS EN LA RESISTENCIA A LA COMPRESIÓN DEL COMPUESTO DE RESIDUOS DE MINERAL DE HIERRO/CEMENTO

EDGAR VLADIMIRO MANTILLA CARRASCO, Dr. | UFMG - Universidade Federal de Minas Gerais, Brasil

JUDY NORKA RODO MANTILLA, Dra. | FUMEC – Universidade FUMEC, Brasil

ELIENE PIRES CARVALHO, Dra. | CEFET-MG – Centro Federal de Educação Tecnológica de Minas Gerais, Brasil

MARCO ANTÔNIO PENIDO DE REZENDE, Dr. | UFMG - Universidade Federal de Minas Gerais, Brasil

REJANE COSTA ALVES, Dra. | UFES - Universidade Federal do Espírito Santo, Brasil

MARIA TERESA GOMES BARBOSA, Dra. | Universidade Federal de Juiz de Fora, Brasil

WHITE JOSÉ DOS SANTOS, Dr. | UFMG - Universidade Federal de Minas Gerais, Brasil

ABSTRACT

The various properties of mortars produced with iron ore tailings and cement (IOT/cement) at room temperature have been extensively studied, but research on high-temperature behavior is scarce. The objective of this study is to analyze the compressive strength and mass loss of these mortars when subjected to high temperatures. The test specimens (TSs) were exposed to different temperature levels (100°C to 1100°C) and subsequently subjected to axial compression tests. The results showed that with increasing temperature, there was a loss of strength. The loss of strength exhibited a linear trend and became more pronounced after 350°C. The TSs subjected to 1100°C exhibited an 80% loss of strength, while the mass loss was less than 5%. One of the contributions of this study, in line with current research, is to emphasize the importance of composites (IOT/cement) as a sustainable and economically viable alternative, given that iron ore tailings are generated in large quantities during the beneficiation process. Another contribution is to demonstrate that these composites can result in mortars with a significant reduction in compressive strength and a small mass loss when exposed to high temperatures. Furthermore, it highlights that they meet all performance and safety requirements in fire situations, making them a non-combustible product.

KEYWORDS

Iron ore tailings; cement, compressive strength; high temperatures; sustainability; thermal effects.

RESUMO

As diversas propriedades das argamassas produzidas com rejeito de minério de ferro e cimento (IOT/cimento) em temperatura ambiente, foram amplamente estudadas, já em altas temperaturas são escassas. O objetivo desse estudo é analisar a resistência à compressão e a perda de massa de estas argamassas, quando submetidas a elevadas temperaturas. Os corpos de prova (CPs) foram expostos a diferentes níveis de temperatura (100 oC até 1100 oC). Posteriormente, foram ensaios à compressão axial. Os resultados mostraram que, com o aumento da temperatura, houve perda da resistência. A perda de resistência mostrou-se linear e, mais rápida a partir dos 350 oC. Os CPs submetidos a 1100 oC



apresentaram uma perda de resistência de 80%, porém a perda de massa foi inferior a 5%. Uma das contribuições desse estudo, corroborando com as pesquisas atuais, é ressaltar a importância dos compósitos (IOT/cimento) como uma alternativa sustentável e economicamente viável, uma vez que o rejeito de minério de ferro é gerado em grande quantidade durante o processo de beneficiamento. Outra contribuição é mostrar que esses compósitos podem resultar em argamassas com considerável redução da resistência à compressão e pequena perda de massa, quando submetidas a temperaturas elevadas. Também ressaltar que eles atendem a todos os requisitos de desempenho e segurança em situações de incêndio, podendo ser considerado um produto incombustível.

PALAVRAS-CHAVE

Rejeito de minério de ferro; cimento; resistência à compressão; altas temperaturas; sustentabilidade; efeitos térmicos.

RESUMEN

El objetivo es analizar la resistencia a la compresión y la pérdida de masa de morteros producidos con desechos de mineral de hierro (IOT) y cemento cuando se someten a altas temperaturas. Las probetas se expusieron a diferentes niveles de temperatura (100°C a 1100°C) y posteriormente se sometieron a ensayos de compresión axial. Los resultados mostraron que con el aumento de la temperatura, hubo una pérdida de resistencia. La pérdida de resistencia exhibió una tendencia lineal y se hizo más pronunciada después de 350°C. Las probetas sometidas a 1100°C exhibieron una pérdida de resistencia del 80%, mientras que la pérdida de masa fue inferior al 5%. Una de las contribuciones de este estudio, en línea con la investigación actual, es enfatizar la importancia de estos compuestos como una alternativa sostenible y económicamente viable, dado que los IOT se generan en grandes cantidades durante el proceso de beneficio. Otra contribución es demostrar que estos compuestos pueden resultar en morteros con pequeña pérdida de masa e grande reducción de resistencia, cuando se exponen a altas temperaturas. Además, destaca que cumplen con todos los requisitos de rendimiento y seguridad en situaciones de incendio, lo que los convierte en un producto no combustible.

PALABRAS CLAVE

Residuos de mineral de hierro; cemento; resistencia a la compresión; altas temperaturas; sostenibilidad; defectos térmicos.

1. INTRODUCTION

The construction industry has been increasingly seeking sustainable and efficient solutions to address contemporary challenges (ZHAO et al., 2014). In this context, the use of alternative and recycled materials has proven to be a promising approach, both for reducing environmental impact and improving the performance of construction materials (ALMADA et al., 2022). One of the alternatives that has gained prominence is the composite of iron ore tailings/cement (IOT/cement). Iron ore tailings (IOT), which are generated in large quantities by the mining industry, are a waste product that presents significant challenges in terms of management and environmental impact (BALAJI, 2022). However, recent studies have explored its potential as a component in construction materials, especially in combination with Portland cement (DUARTE et al., 2022). Portland cement is the primary binder used in the production of concrete and mortar, and the incorporation of IOT can contribute to reducing the amount of cement required, while adding value to the waste (TAURINO, 2023). The use of these composites as construction material has shown promise as an alternative for the sustainable reuse of industrial waste and the reduction of environmental impact (ALMADA et al., 2023; CAMPOLINA et al., 2023).

The IOT/cement composite is characterized by its versatility and the mechanical properties and durability it offers. Studies have shown that these composite exhibits satisfactory performance in terms of compressive strength, durability, and dimensional stability. Furthermore, the use of IOT as a component of the composite can reduce the demand for natural resources (sand and crushed stone) and contribute to the circular economy by providing a more sustainable disposal solution for industrial waste. However, it is important to highlight that the IOT/cement composite is not immune to the effects of high temperatures. Exposure to high-temperature conditions, such as fires, can compromise the structural integrity of this material, affecting its compressive strength and other mechanical properties, as is the case with most construction materials. Therefore, understanding the influence of high temperatures on this composite is crucial for assessing its viability and safety in applications subject to fire risks, such as commercial, industrial, and residential buildings (FIGUEIREDO et al., 2021).

While there is extensive literature on the influence of temperature on the compressive strength of cementitious

materials, such as concrete and mortar, specific research on the IOT/cement composite is still limited. Few studies have comprehensively addressed this topic, considering different proportions of IOT, variations in curing and thermal exposure conditions, as well as the influence of other factors, such as tailings particle size and the addition of additives (BAI et al., 2022). One of the main concerns related to the influence of high temperatures on the compressive strength of the IOT/cement composite is the effect of moisture loss during heating. Water is essential for the hydration of Portland cement and the formation of chemical bonds responsible for the material's mechanical strength. Therefore, moisture loss can compromise the hydration reaction and consequently reduce compressive strength (MD AZREE, 2023).

Compressive strength is a crucial parameter for assessing the composite's ability to resist applied loads, and its degradation under high temperatures can compromise structural stability and safety (DAHISH and ALMUTAIRI, 2023; CARRASCO et al., 2017).

Zhang and Tan (2022) investigated the compressive strength of iron ore tailings/cement (IOT/cement) composite specimens subjected to temperatures up to 800°C. The results demonstrated a gradual decrease in compressive strength with increasing temperature, highlighting the adverse influence of high temperatures on the mechanical properties of the composite. Furthermore, theoretical and numerical studies have also contributed to understanding the degradation mechanisms of compressive strength at high temperatures. Mathematical models and computational simulations have been developed to analyze the thermomechanical behavior of the composite and predict its performance in fire scenarios. In a study conducted by Jhatial et al. (2023), numerical models were used to simulate the behavior of conventional mortar specimens under fire conditions. The results showed a significant reduction in compressive strength with rising temperature, corroborating the experimental findings.

When analyzing the effects of high temperatures, these studies have investigated various aspects related to the behavior of the composite (ZHANG and TAN, 2022). This includes the influence of temperature on the cement matrix and constituent materials of the composite, such as IOT, as well as the interactions between these components (GOA et al., 2023). Studies like the one conducted by Chen et al. (2022) analyzed the influence of porosity and water/cement ratio on the compressive strength of IOT/cement composite at high temperatures. The results indicated that porosity and water content directly influence the material's mechanical strengths,

especially under extreme thermal conditions. Water loss during exposure to high temperatures can lead to increased porosity and compromise compressive strength. Another relevant study is that of Li et al. (2018), which investigated the effects of temperature on the hydration process of cement in IOT composites. The results indicated that exposure to high temperatures during the curing process negatively affected the formation of chemical bonds between the composite particles, resulting in reduced compressive strength. This research underscores the importance of controlling curing conditions to prevent significant strength losses.

Another interesting approach is presented by Tang et al. (2019), who investigated the effects of additive additions on the compressive strength of IOT/cement composite after exposure to high temperatures. The results showed that the addition of additives, such as silica fume and metakaolin, can enhance the compressive strength of the composite even under high-temperature conditions. This research emphasizes the importance of exploring new formulations and additives to optimize the composite's performance under extreme conditions.

These studies contribute to advancing scientific knowledge in the field, providing experimental data and theoretical analyses that aid in understanding the involved phenomena and making informed decisions regarding the safe and efficient use of this material in high-temperature exposure environments (DAHISH and ALMUTAIRI, 2023; LI et al., 2023; RAMZI et al., 2023).

In summary, the influence of high temperatures on the compressive strength of IOT/cement composite is a highly relevant topic for civil engineering and the construction materials industry. Experimental, theoretical, and numerical studies have contributed to understanding degradation mechanisms and developing strategies to enhance the mechanical properties of this material under elevated temperatures. The pursuit of more efficient and sustainable solutions for the construction industry necessitates ongoing investigation into these aspects, aiming to ensure the safety and durability of structures in fire situations.

In this context, the objective of this work is to determine, through rigorous experimental tests with temperature control, the variation in compressive strength and mass loss of the IOT/cement composite as a function of temperature.

2. MATERIALS AND METHODS

IOT was provided by Samarco Mining Company, and

the tests were conducted at the laboratories of the School of Engineering at UFMG.

2.1. Materials

For the preparation of test specimens (TS), high early strength Portland cement (CP V-ARI) and IOTs (as fine (F) and granular (G) aggregates) were used. The chemical composition is presented in Table 01, and the granulometric characteristics, permeability, and specific mass of the IOTs are shown in Table 02.

| | Fe ₂ O ₃ | SiO ₂ | Al ₂ O ₃ | P | PPC | MnO |
|---|--------------------------------|------------------|--------------------------------|-------|------|-------|
| F | 34,26 | 45,72 | 2,06 | 0,051 | 0,09 | 3,030 |
| G | 21,99 | 67,97 | 0,15 | 0,140 | 0,4 | 0,010 |

Where F = fine and G = granular.

Table 01: Chemical composition of IOT.

Source: Authors.

| | Sand % | Silt % | Clay % | k (cm/s) | ps (g/cm ³) |
|---|--------|--------|--------|----------------------|-------------------------|
| F | 14,0 | 79,2 | 7,1 | 2,3x10 ⁻⁵ | 3,47 |
| G | 44,1 | 53,8 | 2,1 | 1,3x10 ⁻³ | 3,14 |

Where F = fine and G = granular.

Table 02: Granulometry, permeability, and specific mass.

Source: Authors.

The mortar mix proportion for the study was 1:1:4:3.04 (cement: fine tailings: granular tailings: water), by weight. The mortar consistency was 300 ± 15 mm, measured on a flow Table. The typical consistency used in mortars is 260 ± 10 mm (SANTOS et al., 2018). However, due to the increased cohesion of the mortar under study and the fineness of the aggregates, a higher value was necessary on the flow Table to achieve proper workability. For the preparation of TS, a pressing load of 400 kN was applied, considering the use of CP V – ARI cement. The mixing sequence of the mortar components and their homogenization for TS preparation is crucial. Thus, the following sequence was adopted: first, the tailings; then, the cement; and finally, water was gradually added in a mortar mixer. Subsequently, the mixture was placed into molds and pressed using a hydraulic press. At the end of the process, the TS was extracted for curing. The curing followed the Brazilian standard NBR 13279 (ABNT, 2005). The TS fabricated with this mortar proportion were subjected to wet curing and tested after 28 days. The dimensions of the TS cylinders were 5 cm in diameter and 10 cm in length.

A total of 63 TS were prepared, with 7 TS for each temperature (100°C, 200°C, 350°C, 500°C, 600°C, 750°C, 850°C, 950°C, and 1100°C). After curing, each TS was divided using a diamond circular saw, resulting in a twin TS for each specimen. Figure 01 shows a portion of the TS after curing.



Figure 01: Test specimens ready for testing.
Source: Authors' collection.

2.2. Equipment and Instrumentation

The equipment used was the same as recommended for the non-combustibility tests using a muffle furnace. For weight (mass) determination, a “Marte Balanças” brand balance, model AL 500, with a resolution of 0.001 g, certified by RBC with calibration certificate number 119015, was used. A muffle furnace (test adapted following the criteria of ISO 1182 (2020), model NA1221, temperature limit 1100°C, controlled by 10 PT 100 thermoresistances calibrated by Analógica company, Figure 02).



Figure 02: Balance and muffle furnace.
Source: Authors' collection.

Type K thermocouples were used, which have a stainless-steel sheath at the tip. One thermocouple was placed inside the TS (in a hole made in the middle of the TS to the halfway point of its height) to measure internal temperature, and another was placed on the surface of the TS to measure its surface temperature. All thermocouples were connected to a Data Acquisition System (DAS), which consists of a signal conditioning board that amplifies and conditions the signal, an analog-to-digital signal conversion board (A/D), and a control board containing a multiplexer



Figure 03: Thermocouples in the muffle furnace and the Data Acquisition System.
Source: Authors' collection.

and 16 channels with their respective circuit conditioners. This entire system is connected to a notebook, Figure 03.

For the simple compression tests, a universal testing machine (UTM) from Emic/Instron brand, model DL 30000, with a capacity of 300 kN, as shown in Figure 04, was used. This machine has automatic load and displacement control, servo-controlled. A 50 kN load cell was attached for the tests, Figure 04.



Figure 04: Universal testing machine, capacity of 300 kN.
Source: Authors' collection.

2.3. Test Methodology

The test methodology will be divided into two stages: the non-combustibility test and the simple compression test. For both tests, the age of the TSs was 56 days. The preparation of the TSs was carried out using a grinder, ensuring the parallelism of the surfaces and a 90-degree

angle between the load application surface and the length of the concrete specimen.

For the non-combustibility test, the testing method was adapted from the recommendations of the standards: ISO 1182 (2020); NBR 5628 (ABNT, 2022); NBR 9442 (ABNT, 2019); and NBR 14432 (ABNT, 2001). The adopted test procedure followed the ISO 1182 (2020) standard, with slight adaptations regarding the furnace and TS dimensions. Initially, one of the twin TSs underwent the non-combustibility test. The main premise was to vary the temperature range. Two TSs were used for different temperature ranges (100°C, 200°C, 350°C, 500°C, 600°C, 750°C, 850°C, 950°C, and 1100°C).

The preparation of the TSs involved conditioning them in an oven at a temperature of $60^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ for a period of 21 hours. After removal from the oven, they were placed in a desiccator to cool, with cooling monitored using a thermometer. Once cooling was confirmed, the TSs were weighed on a balance to obtain the initial mass (m_i). The following steps were carried out for each test temperature. The muffle furnace was turned on with its temperature sensor set to the test temperature, for example, $100^{\circ}\text{C} \pm 5^{\circ}\text{C}$, and allowed to stabilize for a period of 10 minutes or until there was no variation above 2°C . Next, the entire test system was verified: the muffle furnace stabilized, the Data Acquisition System (DAS), the surface thermocouple of the TS (Ts), and the thermocouple to be inserted into the TS (Tc). The thermocouple (Tc) was then placed inside the TS and positioned inside the furnace. This operation lasted a maximum of 5 seconds. Immediate monitoring began, capturing the temperatures inside and on the surface of the TS. The test concluded when the temperatures of the two thermocouples stabilized for a period of 10 minutes. The test lasted approximately 30 minutes. Subsequently, the TS was removed from the muffle furnace, and no fragments or ash were observed. Once cooled, the TS was weighed. This procedure was repeated for the remaining 67 TS.

The simple compression test was initiated immediately thereafter. NBR 7215 (ABNT, 2019), which addresses the testing methodology for determining the compressive strength of cylindrical mortar specimens, was employed. The TSs were placed in the Universal Testing Machine (UTM), and the test was initiated. The machine utilized a script that allowed for test automation. The application of load was monotonous, with a loading rate of 1 mm/min (ABNT NBR 7215, 2019). Figure 05 shows: a detail of the TS during the test and the twin TS after the test.



Figure 05: Detail of TS during the test and TS after the test.

Source: Authors' collection.

3. RESULTS AND DISCUSSIONS

During the non-combustibility tests, no emission of fumes was observed. The furnace thermocouple temperature (TPf) did not have a significant variation, i.e., less than 50°C . No flaming of the TSs was observed during the test. Figure 06 presents a Boxplot graph of mass loss, indicating very small variation in results at each temperature. Table 03 presents the average mass loss, standard deviation, and coefficient of variation for each temperature. The coefficient of variation values were low, indicating little dispersion in the experimental results. The fracture patterns of the TSs were conical in shape.

Figure 07 displays the average mass loss values as a function of temperature. It can be observed that the average mass loss at a temperature of 1100°C was 4.99%, significantly lower than the allowed 50% (ISO 1182, 2020), and that the mass loss variation is linear with increasing temperature, as shown by equation (1).

$$M_{\text{loss}} = 0,0051 \times T \quad (R^2=0,99) \quad (1)$$

Where: M_{loss} = mass loss (%), T = temperature ($^{\circ}\text{C}$)

Al-Shwaiter and Awang (2023) and Ali and Lublóy (2022) comment that one of the properties contributing to the non-combustibility of composites is the presence of cement in the matrix. Cement in its composition contains components such as calcium oxide (CaO), which, when exposed to high temperatures, undergoes a calcination process, releasing water and other volatile compounds. This process acts as a protective layer, retarding fire propagation and safeguarding the underlying material. Furthermore, the incorporation of IOT into the cement matrix can contribute to improved fire resistance. IOT is rich in metallic oxides, which have flame-retardant properties. These metallic oxides act as physical barriers, impeding heat propagation and reducing material flammability.

Regarding the non-combustibility of the reject-cement TSs, it can be affirmed that the TSs meet all performance requirements, and can be considered a non-combustible product contributing to fire prevention

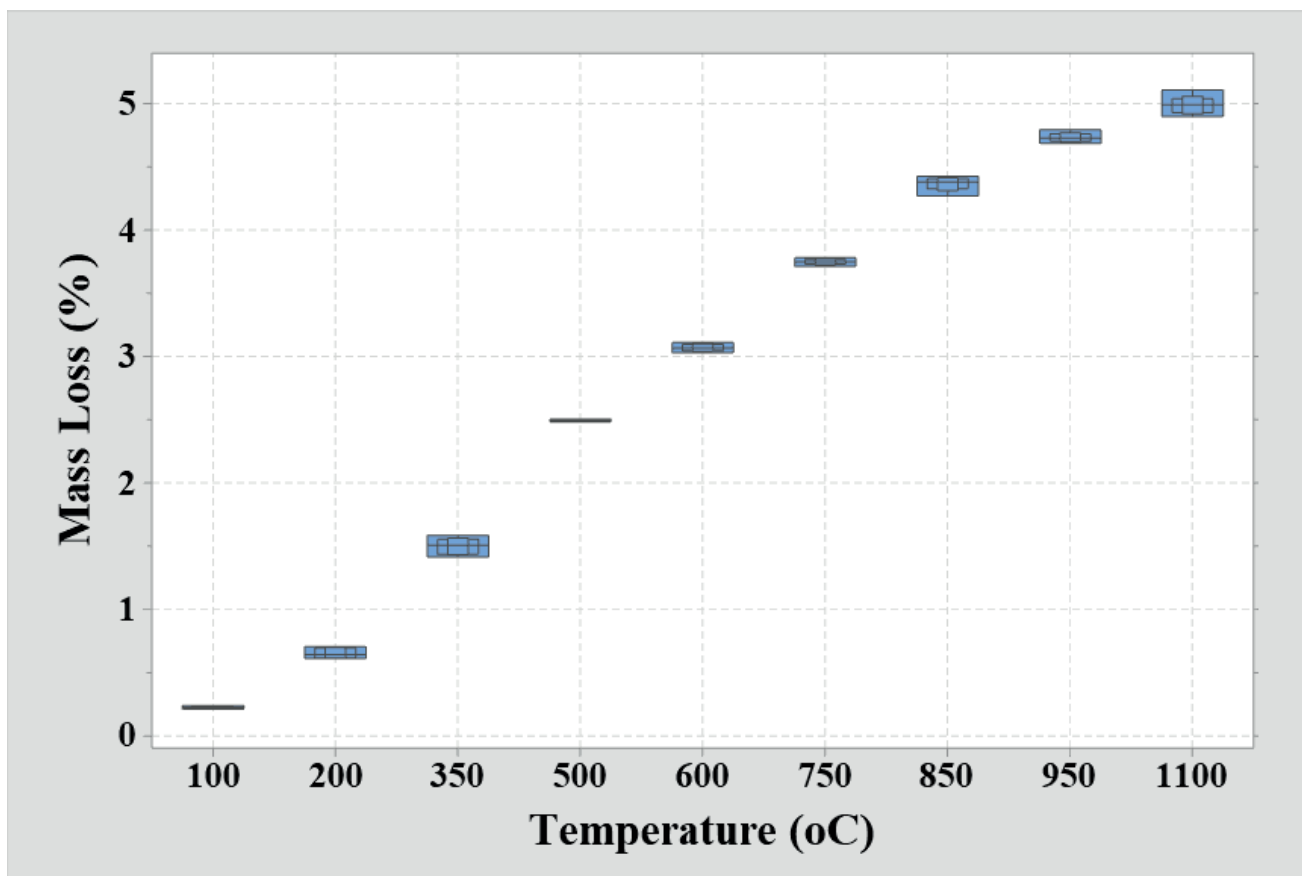


Figure 06: Mass Loss as a Function of Temperature.

Source: Authors' collection.

| Temperature (°C) | Mass Loss (%) | | | Strength Loss (%) | | |
|------------------|---------------|--------------------|--------------------------|-------------------|--------------------|--------------------------|
| | Mean | Standard Deviation | Coefficient of Variation | Mean | Standard Deviation | Coefficient of Variation |
| 100 | 0,22 | 0,010 | 4,464 | 1,57 | 0,023 | 1,485 |
| 200 | 0,65 | 0,041 | 6,296 | 13,64 | 0,591 | 4,336 |
| 350 | 1,49 | 0,061 | 4,109 | 30,18 | 1,593 | 5,276 |
| 500 | 2,49 | 0,008 | 0,335 | 42,78 | 0,698 | 1,632 |
| 600 | 3,07 | 0,028 | 0,913 | 54,42 | 0,296 | 0,544 |
| 750 | 3,75 | 0,023 | 0,626 | 67,69 | 1,421 | 2,099 |
| 850 | 4,36 | 0,054 | 1,242 | 71,81 | 0,330 | 0,460 |
| 950 | 4,73 | 0,036 | 0,759 | 77,31 | 0,240 | 0,311 |
| 1100 | 4,99 | 0,071 | 1,413 | 80,65 | 0,644 | 0,798 |

Table 03: Mass and Strength Loss with Descriptive Statistics.

Source: Authors' collection.

and firefighter safety (passive protection).

The results of compressive strength for the 7 TSs at different temperature ranges (100°C, 200°C, 350°C, 500°C, 600°C, 750°C, 850°C, 950°C, and 1100°C) and at room temperature (RT) are shown in the Boxplot graph of Figure 08. It is noticeable that as the temperature increases, the compressive strength decreases rapidly, with the loss becoming more pronounced after 350°C. Table 03 presents the average loss of strength, standard deviation, and coefficient of

variation for each temperature, demonstrating that the coefficients of variation for all temperatures are low, indicating good attainment of experimental results.

From Figure 07, it is evident that the average loss of strength at a temperature of 1100°C (T) was 80%, even with this loss, the TSs did not disintegrate, and its average compressive strength was 3.50 MPa, compared to an average strength of 18.17 MPa at room temperature (RT) (Figure 08), indicating a significant compromise in its strength.

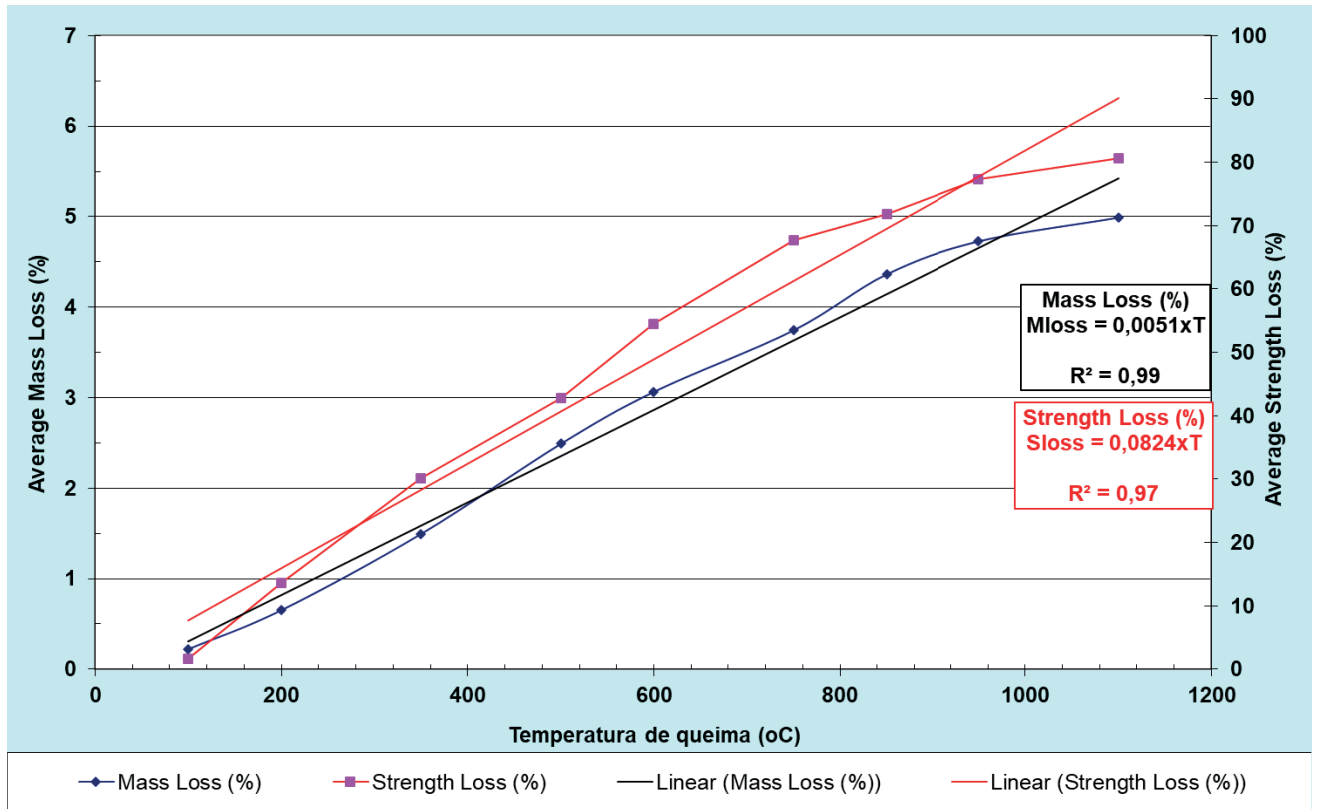


Figure 07: Mass Loss and Compressive Strength as a Function of Temperature (average values).
 Source: Authors' collection.

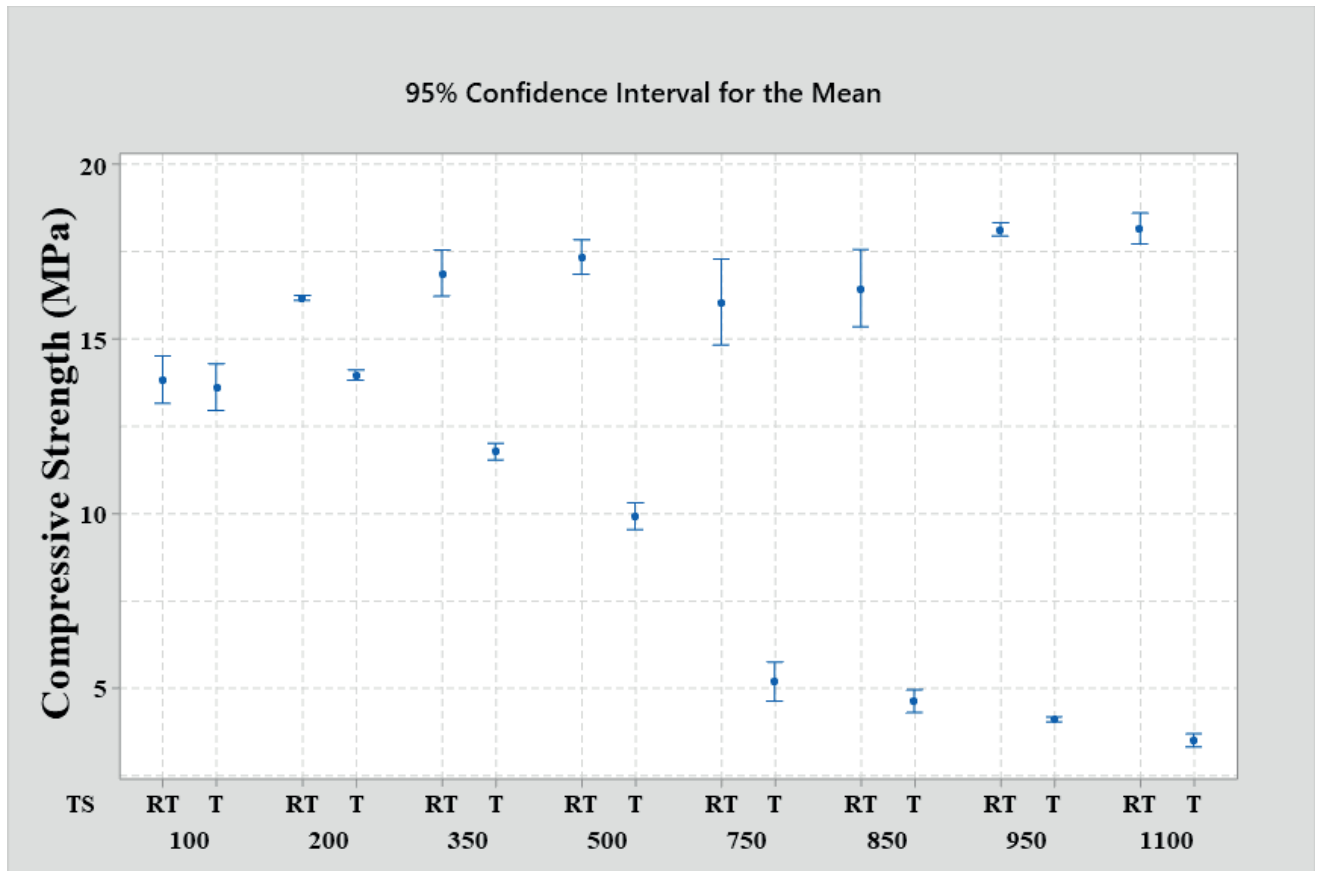


Figure 08: Compressive Strength as a Function of Temperature.
 Source: Authors' collection.

This decrease can be attributed to various factors, such as cement dehydration, loss of water associated with materials, and possible alterations in the composite's microstructure due to high temperatures. In general, there is a loss of free water, or capillary-stored water, in the temperature range of 30°C to 105°C, and this water is completely depleted by 120°C. Gypsum decomposition occurs in the temperature range of 110°C-180°C, and in the range of 180°C-240°C, the dehydration of calcium aluminate and hydrated aluminosilicates occurs. The combined loss of water from hydrated calcium silicate occurs between 50°C and 300°C; the dehydration of this silicate with respect to intercalated water occurs at 350°C; the release of adsorbed water from hydrated calcium silicate occurs around 400°C; the dehydration of calcium hydroxide occurs between 410°C and 580°C, and from 520°C to 900°C, the decomposition of calcium carbonate occurs (BACARJI, 2013; ALARCON-RUIZ et al., 2005; PAJÁ et al., 2003).

The variation of strength loss is linear with increasing temperature, as expressed in equation (2).

$$\text{Sloss}=0,0824 \times T \quad (R^2=0,97) \quad (2)$$

Where: Sloss = strength loss (%), T = temperature (°C)

4. CONCLUSIONS

The IOT/cement composites exhibit favorable non-combustibility properties due to the presence of cement in the matrix and the metallic oxides present in the IOT. However, it is important to consider the specific conditions of fire exposure and adopt appropriate fire protection measures to ensure the safety of constructions. The mass loss at a temperature of 1100°C was 4.99%, significantly lower than the permitted 50%, indicating that this composite is non-combustible.

The compressive strength of the IOT/cement composite is adversely affected by high temperatures. This reduction in strength can compromise the structural capacity of the material in fire situations and intense heat exposure. Therefore, it is essential to consider the effects of high temperatures during the design and use of this composite, developing specific guidelines to ensure its proper application in high-heat conditions. The loss of strength at a temperature of 1100°C was 80%."

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AUTHORS

ORCID: 0000-0001-7870-0283

EDGAR VLADIMIRO MANTILLA CARRASCO, Dr. | Professor Adjunto Departamento de Tecnologia da Arquitetura e do Urbanismo- Escola de Arquitetura | Universidade Federal de Minas Gerais - email: mantilla.carrasco@gmail.com

ORCID: <https://orcid.org/0000-0001-7426-0970>

JUDY NORKA RODO MANTILLA, Dra. | Professora assistente da Universidade FUMEC | judynorka@gmail.com

ORCID: <https://orcid.org/0009-0006-2664-0569>

ELIENE PIRES CARVALHO, Dra. | Professora Titular do Centro Federal de Educação Tecnológica de Minas Gerais (CEFET-MG) | email: eliene@cefetmg.br

ORCID: <https://orcid.org/0000-0001-7896-8669>

MARCO ANTÔNIO PENIDO DE REZENDE, Dr. | Prof. Dr. do Departamento de Tecnologia do Design, da Arquitetura e do Urbanismo, Escola de Arquitetura, UFMG | email: <http://lattes.cnpq.br/8413549938151614>

ORCID: <https://orcid.org/0000-0003-4059-3974>

REJANE COSTA ALVES, Dra. | Engenharia de Estruturas, Departamento de Ciências Florestais e da Madeira/ CCAE/ UFES email: rejanealves.ufes@gmail.com

ORCID: <https://orcid.org/0000-0002-3839-5728>

Maria Teresa Gomes Barbosa, Dra. | Faculdade de Engenharia - UFJF | email: teresa.barbosa@ufjf.br

ORCID: <https://orcid.org/0000-0002-7451-3365>

WHITE JOSÉ DOS SANTOS, Dr. | Engenharia de estruturas.
Departamento de materiais de construção civil, escola de engenharia UFMG. | email: white.santos@demc.ufmg.br

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