Analysis of the influence of ISR with TiO_2 addition on the compressive strength and durability of structural self-compacting concrete associated with porosity

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Abstract: In the contemporary global landscape, efforts to reduce energy consumption and mitigate the carbon footprint associated with products and human activities have intensified, with the construction industry emerging as one of the most environmentally impactful sectors due to its substantial consumption of natural resources and significant waste generation. Among the sustainable strategies under investigation, the reuse of industrial or mining residues through their incorporation into construction materials has gained particular relevance. Within this framework, the present study evaluates the utilization of industrial solid residue (ISR) generated during TiO_2 extraction for pigment production as an additive in cementitious matrices, aiming both to ensure the environmentally responsible disposal of this waste and to harness the photocatalytic potential of the residual TiO_2. Specifically, the research investigates the incorporation of 15% ISR into structural self-compacting concrete (fck ≥ 30 MPa), assessing its influence on key physical and mechanical properties, with emphasis on apparent porosity, water absorption, and axial compressive strength. Experimental results revealed marked increases in water absorption (12.03% for ISR vs. 6.73% for REF), void index (24.23% vs. 14.56%), and capillary absorption (1.71 g/cm² vs. 0.86 g/cm²), alongside a reduction in compressive strength (31.32 MPa vs. 32.61 MPa). These findings indicate that ISR incorporation significantly modifies the concrete microstructure, promoting greater capillary pore formation and diminishing mechanical performance. While the resulting material complies with applicable technical standards, the observed microstructural changes underscore the need for further durability investigations to fully ascertain the long-term implications for service life and structural performance.
Keywords: Self-compacting concrete. Absorption. Axial compression. Residue. Porosity.
Abbreviations: ISR, Industrial Solid Residue, REF, Reference.

1. Introduction

In the current international context, strategies aimed at minimizing energy consumption and the carbon footprint generated by goods and human activities are expanding. The construction industry plays a significant role in the global environmental scenario, accounting for a considerable share of natural resource consumption and waste generation [1]. Population growth and the high demand for construction have resulted in large volumes of waste and increased exploitation of natural resources. In this scenario, sustainable solutions have gained prominence, particularly the reuse of industrial waste by incorporating it into, or converting it into, construction materials. The utilization of such environmental liabilities—materials that would otherwise be landfilled or improperly disposed of, leading to environmental pollution—is of paramount importance and is considered in determining the carbon footprint and energy consumption of products [2][3].

Based on this premise, the incorporation of Industrial Solid Residue (ISR), generated during the TiO_2 extraction process for pigment production, into a cementitious matrix aims not only to ensure proper disposal of this environmental liability but also to harness the photocatalytic potential of the residual TiO_2 that could not be recovered from the ore, thereby rendering the resulting product photocatalytic.

This approach holds significant importance for the construction sector [4][5].

In this study, the influence of adding 15% ISR to a structural self-compacting concrete will be evaluated, with the objective of analyzing how the incorporation of the residue into the cementitious matrix may affect the physical and mechanical properties of the concrete.

3. Methodology

3.1. Materials

3.1.1. ISR – Unreacted Ore

The industrial solid residue containing titanium dioxide (ISR) used in this study is presented as a pulverized solid with irregular particle size distribution, as shown in Figure 1. For concrete production, the residue was employed in its natural form, without undergoing any additional grinding process.

Figure 1. Titanium Dioxide Residue.

The particle size distribution indicates a broad distribution of particles in the sample, with an average size of 22.334 µm (micrometer). The particle volume distribution according to particle size was obtained, indicating the percentage of particle size present in the sample, where 10% of the particles are smaller than 2.687 µm, 50% are smaller than 16.381 µm, and 90% are smaller than 47.281 µm. The results show that the average diameter of the residue is less than 75 µm.

The particle size distribution of the residue was established using a PSA 1190L laser particle size analyzer from Anton Paar Brasil. To analyze particle size distribution, the residue had to be dispersed in ethyl alcohol.

Laser particle size analysis is a technique based on the sedimentation of suspended particles. This method uses light absorption to accurately measure the particle size of the material, providing a detailed understanding of particle size distribution.

3.1.2. Fine aggregate

The fine aggregate consists of natural quartz sand, with a fineness modulus of 1.56 mm and a maximum particle size of 2.36 mm, sourced from deposits in Barra de Pojuca, Camaçari/BA, as illustrated in Figure 2.

For concrete production, the sand was used in a dry state after oven-drying to ensure the absence of moisture. In industrial-scale production, where the sand is exposed to ambient conditions, it is recommended that moisture content be determined and controlled, and the water present in the sand be compensated for in the concrete mix.

Figure 2. Fine Sand.

3.1.3. Coarse aggregate

The coarse aggregate used is crushed stone, with a characteristic maximum particle size of 12.5 mm (commonly referred to as “brita 0”), as shown in Figure 3.

Figure 3. Brita 0.

3.1.4. Cement

The cement used in this study was CP V-ARI RS produced according to the parameters stipulated by ABNT NBR 16697:2018 (Portland Cement – Requirements) [6] (Figure 4).

CP V-ARI (High Early Strength Portland Cement) is a type of Portland cement specially formulated to provide high early strength. It is widely used in constructions requiring rapid formwork removal or when accelerating the project schedule is desired. This cement achieves high mechanical strength at early ages due to its high clinker content and low limestone filler addition.

Figure 4. CP V-ARI RS.

3.1.5. Admixture

The admixture used in this study was a superplasticizer (Figure 6), classified as a normal setting admixture for Portland cement concrete. It is a liquid substance composed of polycarboxylates, which are macromolecules employed as high-efficiency dispersants in cementitious matrices. These compounds reduce the material’s viscosity and minimize the amount of water used in the mix by up to 45%.

The superplasticizer has a specific gravity of approximately 1.070 g/cm³ and acts through surface adsorption and steric separation effects on Portland cement particles during its hydration process.

Its primary advantage in relation to self-compacting concrete, the focus of this research, is the ability to adjust its dosage to meet stringent requirements for flowability, workability retention, and the reduction of cement and water consumption in the mixture.

Figure 5. Superplasticizer Admixture.

3.1.6. Water

The mixing water used in the concrete production was sourced from the public supply network of Empresa Baiana de Água e Saneamento S.A. (Embasa).

3.2. Methods

3.2.1 Physical Properties

3.2.1.1. Determination of Absorption, Voids and Specific Gravity

The water absorption, void index, and specific gravity tests are conducted with the objective of evaluating the porosity and density of cementitious matrix materials such as concrete. These parameters are essential for understanding the durability and strength of the material.

The water absorption test in this study was performed according to the procedure described in ABNT NBR 9778:2005 (Hardened mortar and concrete – Determination of water absorption, void index, and specific gravity) [7], from which the values of Water Absorption (Equation 1), Void Index (Equation 2), and Real Specific Gravity (Equation 3) are obtained. The Bel Mark electronic scale, model K-200001, with a resolution of ± 0.1, was used to verify the mass of the test specimens.

$$A = \frac{m_{sat} - m_s}{m_s} \times 100 \ (\%) \qquad (1)$$

Where:

m_{sat} = saturated mass after soaking (g);

m_s = dry mass (g).

$$Iv = \frac{m_{sat} - m_s}{m_{sat} - m_i} \times 100 \ (\%) \qquad (2)$$

Where:

m_i = mass of the saturated sample immersed in water.

$$\rho_r = \frac{m_s}{m_s - m_i} \quad (g/cm^3) \qquad (3)$$

For the execution of the test, the specimens, at 28 days of curing, were initially placed in an oven at a temperature of 105 ± 2ºC. After 72 hours, the dry mass of the specimens was determined. Subsequently, the specimens were immersed in water at room temperature (23 ± 2ºC) for a period of 72 hours, after which their immersed mass was determined using a hydrostatic balance. Afterwards, the specimens were removed from the water, the excess surface moisture was wiped off with an absorbent cloth, and their mass was determined again, corresponding to the saturated condition. The oven used was the DL Type A.C, manufactured by Labomax.

3.2.1.2. Determination of Water Absorption by Capilarity

The capillary water absorption test was conducted to determine whether the porosity values obtained are directly related to the presence of capillary pores within the concrete structure, a crucial factor in assessing how material porosity affects durability and strength [8].

In this study, the test was performed according to the procedure described in ABNT NBR 9779:2012 (Hardened mortar and concrete – Determination of capillary water absorption) [9]. The results obtained from the test include capillary water absorption (Equation 6) and the height of internal capillary rise, which was measured following the diametral compression failure of specimens, in accordance with ABNT NBR 7222:2011 [10], after completing the partial immersion phase of the samples in water. The tests were conducted using an HD-150T servo-controlled press, manufactured by CONTENCO, with a maximum capacity of 150 tons-force and a resolution of 10 kgf.

$$C = \frac{m_{sat} - m_s}{S} \text{ (g/cm}^2\text{)} \qquad (6)$$

Where:

C = water absorption by capillarity;

S = cross-sectional area.

For the capillary water absorption test, the specimens, after 28 days of curing, were dried in an oven at a temperature of 105 ± 5ºC until a constant mass was achieved. Subsequently, the samples were cooled in a desiccator until reaching room temperature, and the initial dry mass was determined.

In the following step, the specimens were placed in trays containing a water layer of 5 ± 1 mm, ensuring that only the lower surface remained in contact with the water. During the test, the specimens were removed at predetermined time intervals (3 min, 6 min, 15 min, 30 min, 1 h, 2 h, 3 h, 4 h, 6 h, 24 h and 48 h), the excess surface water was wiped off with an absorbent cloth, and the mass corresponding to each interval was measured.

3.2.2 Compressive Strength

The axial compressive strength of the concrete was evaluated in accordance with the standard ABNT NBR 5739:2018 – Concrete – Compression test of cylindrical specimens [11]. This procedure is essential for verifying the concrete's compliance with design specifications, ensuring that the material possesses the required load-bearing capacity.

The compressive strength, represented by the strength limit (fc), is quantified as the ratio between the maximum applied load (Pmax) and the cross-sectional area (A) of the specimen, as expressed in Equation 7.

$$f_c = \frac{P_{máx}}{A} \text{ (MPa)} \qquad (7)$$

Prior to testing, the specimens underwent capping on their upper and lower surfaces. This treatment was necessary to ensure the parallelism and flatness of the loading faces, promoting uniform stress distribution during the test.

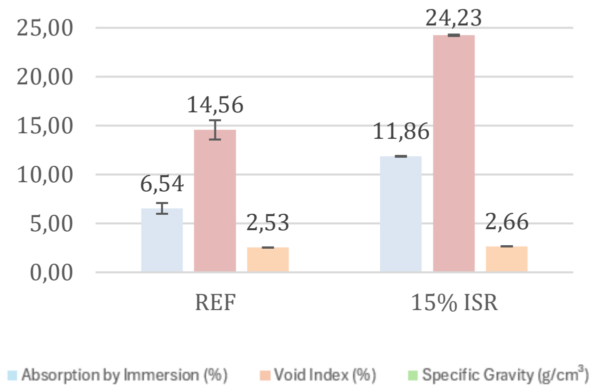
4. Results and Discussion

4.1. Physical Properties

4.1.1. Absorption, Voids and Specific Gravity

After testing, the concrete containing the TiO_2 residue absorbed nearly twice the amount of water, in percentage terms, compared to the reference concrete, as illustrated in the graph in Figure 7. It is also notable that this factor influences the void index percentage, which is considerably higher in the 15% ISR mix than in the REF. These results are consistent with the literature [12][13]. However, the mix with residue addition exhibited a specific gravity 5.14% higher than that of the reference. The specific gravity values obtained through this test are not absolute, as they do not account for the impermeable pores present in the cementitious matrix product under evaluation [14][15].

Figure 7. Water absorption by immersion, void index, and specific gravity.

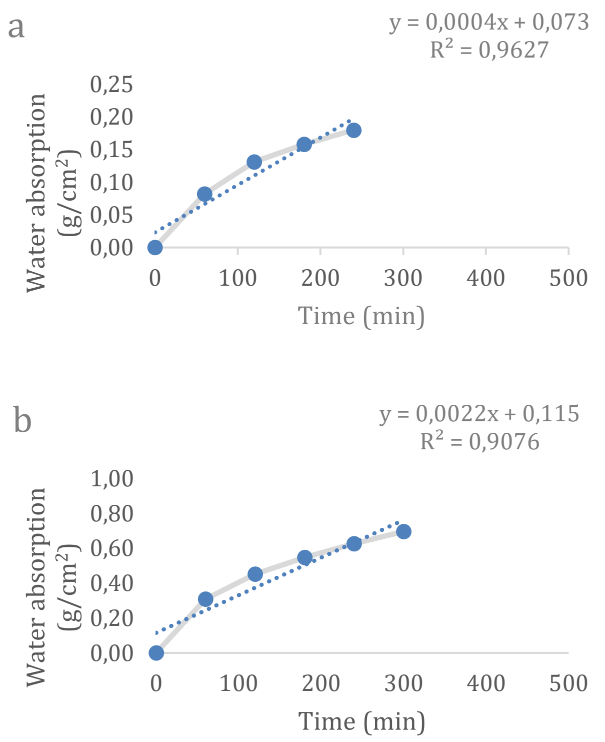


4.1.2. Water Absorption by Capillarity

The capillary absorption test demonstrated that the addition of 15% ISR results in a greater presence of capillary pores compared to the reference concrete, with an absorption value of 1.71 g/cm² after testing, whereas the REF exhibited 0.86 g/cm².

To confirm this result, graphs were generated from the absorption values, as shown in Figure 8, which illustrate the absorption behavior of each concrete and the trend line indicating the likely material response. It is notable from the generated graphs that the coefficient of the equation describing the behavior of the 15% ISR is significantly higher than that of the REF, which is consistent with the literature [16].

Figure 8. Absorption by Capillarity – a) REF; b) 15% ISR

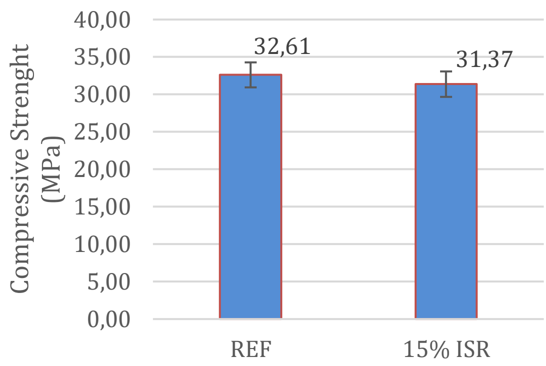


4.2. Compressive Strength

In the comparative analysis of the axial compressive strength test results of the mixes (Figure 10), it is observed that the incorporation of the residue leads to a reduction in the compressive strength of the concrete. This finding is consistent with the previously presented physical characterization test results, as typically, the greater the amount of interconnected pores in concrete, the lower the quantity of cementitious matrix, which consequently decreases its compressive strength [17][18].

Another factor contributing to the notable reduction in compressive strength of the 15% ISR mix is likely associated with the higher content of solid particles in the system, which reduces the space available for the formation of hydration compounds, thereby decreasing the hydration reaction rate of the cementitious matrix [19].

Figure 10. Axial Compressive Strength of Concretes.



The concrete with 15% residue addition, although exhibiting the lowest strength among the tested samples (31.37 MPa), still meets the minimum requirement established by ABNT NBR 6118:2023 [20], which specifies 30 MPa for applications in reinforced and prestressed concrete structural elements.

5. Conclusion

The present study evaluated the performance of self-compacting concretes incorporating titanium dioxide industrial solid residue (ISR), considering their properties in both fresh and hardened states. The results demonstrate that the addition of this residue significantly alters the physical and mechanical characteristics of the concrete, leading to an increase in capillary porosity and a consequent reduction in axial compressive strength. Based on the conducted analyses, there is a clear need to perform additional durability tests to estimate the material's service life. Although the concrete meets the normative requirements for compressive strength despite the reduction, evaluating its durability is essential to determine its long-term performance.

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