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Durability Analysis of Concrete with the Substitution of Natural Aggregates by Copper and Pig Iron Waste

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Abstract:

Background: *The global scenario demands immediate actions to mitigate the environmental imbalances threatening the planet, reflected in natural disasters such as storms, floods, and severe droughts. This study evaluates the durability of concrete in the face of these challenges through carbonation, chloride penetration, and alkali-aggregate reaction tests, exploring the feasibility of producing sustainable concretes using copper ore waste and pig iron slag, available in the southern and southeastern regions of Pará due to the local mining activity.*

Materials and Methods: *The methodology of the study involved the collection of copper waste and blast furnace slag, which were characterized through granulometric analysis, X-ray Diffraction (XRD), and X-ray Fluorescence (XRF). These materials were used as partial substitutes for fine and coarse aggregates in the production of concretes with different dosages. Durability tests were conducted, including accelerated carbonation, chloride penetration, and alkali-aggregate reaction. The analysis of the results aimed to assess the feasibility of these wastes as sustainable alternatives for civil construction.*

Results: *The statistical results showed significance in the substitution of waste materials, highlighting copper waste and blast furnace slag as promising alternatives. The accelerated carbonation tests revealed that concretes with 75% and 100% waste exhibited lower susceptibility to* CO_2 *due to reduced porosity. In the chloride penetration test, concretes with 25% and 75% waste demonstrated lower NaCl penetration, indicating superior resistance. Furthermore, in the alkali-aggregate reaction test, the waste samples showed no reactive potential, as per NBR 15577-4, confirming their suitability for concrete production.*

Conclusion: *A comparative analysis of the properties of these concretes in relation to conventional concrete revealed significant potential for replacing natural aggregates with waste materials that would otherwise be discarded, contributing to the reduction of environmental impact.*

Key Word: *Aggregates; Waste; Durability; Concrete; Sustainability.*

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I. Introduction

The global scenario demands urgent actions to address environmental imbalances that threaten the planet's sustainability. Natural disasters such as storms, floods, and extreme droughts are clear reflections of climate change resulting from environmental degradation, wildfires, and accelerated CO_2 emissions. In this context, academic and technological efforts have gained prominence, especially in the search for alternatives that promote the reuse of industrial and mineral waste. The construction industry accounts for approximately 50% of global raw material consumption, underscoring the need for a paradigm shift in the sector, driven by social demands, environmental regulations, and the appreciation of non-renewable resources[1].

The importance of this topic is highlighted by the upcoming 30th United Nations Conference on Climate Change (COP 30) in 2025, to be held in the city of Belém, Pará, Brazil. This international event will bring together representatives from 197 countries to discuss strategies and actions aimed at mitigating climate change[2]. In this context, initiatives that promote the reduction of the ecological footprint and the development of sustainable technologies are fundamental. The replacement of natural aggregates in concrete with mineral waste aligns directly with this perspective, forming the central focus of this study: investigating the feasibility of utilizing copper and pig iron slag waste in concrete production, with an emphasis on durability and applicability in the construction industry.

Through durability tests, including carbonation, chloride penetration, and alkali-aggregate reaction assessments, this research aimed to evaluate the longevity of concrete produced with mineral waste, simulating conditions addressing potential pathological issues. These tests seek to provide viable solutions for both the construction and mining industries, promoting sustainable integration between the two sectors. Ensuring the reliability of the resulting concrete is a priority, and the selection of tests was guided to obtain robust and applicable results.

The first waste material evaluated is pig iron slag, a byproduct of iron ore production, which reached approximately 2.8 billion tons worldwide in 2022, with the state of Pará contributing 45% of Brazil's national production. Often discarded, this residue was used as a substitute for coarse aggregate, traditionally represented by crushed stone.

The second waste analyzed originates from copper production, a "fine sand" used to replace fine aggregate in concrete. In 2021, Brazil produced approximately 554,000 tons of copper, ranking tenth globally[3]. The growing demand for this metal has generated significant volumes of waste, particularly in the southern and southeastern regions of Pará, where mining is a predominant activity.

Thus, this study aims to assess the effectiveness of manufacturing concrete incorporating copper waste and pig iron slag, comparing their properties with those of conventional concrete. The reuse of these wastes contributes to reducing environmental impact and promotes sustainability in regions where these residues are abundant. By eliminating or minimizing the use of natural aggregates, the study emphasizes the potential for innovation in the construction industry and points to pathways for a more circular and responsible economy.

II. Metodologia

Initially, 250 kg of copper waste from a mine in the Sossego area, in Canaã dos Carajás, was collected under the following conditions: dry and in the open air. Simultaneously, 250 kg of pig iron slag was collected, provided by a steel company located in the industrial yard of the industrial district in Marabá, Pará.This study was based on granulometric analysis, which is part of a doctoral thesis, supported by data on specific mass and the identification of the microstructure of mining residual materials that will be the focus of this research[4]. The main objective was the identification of the physicochemical properties. The results of X-Ray Diffraction (XRD) and X-Ray Fluorescence Spectrometry (XRF) were used to analyze the parameters and tools adopted to achieve the objectives established for this investigation. In order to establish an alternative for the use of these types of waste as fine and coarse aggregates for the production of concrete artifacts, the experimental plan was divided into three stages:

Stage 1: Concrete artifacts were manufactured using different dosages (15%, 25%, 50%, 75%, and 100%) for both types of waste. Fine aggregates were replaced with copper waste, and coarse aggregates were replaced with pig iron slag in the same proportions. Based on the characterization of the waste in the referenced study, the mineral waste showed potential for increasing the consistency of the concrete as the proportion of waste used in the concrete production increased[4]. This is illustrated in Figure 1 below:

Figure 1: Production of Artifacts and Dosages

Stage 2: Durability tests were conducted, including an accelerated carbonation study, where the artifacts were placed in a CO_2 chamber for periods of up to 90 days. A 1% phenolphthalein solution was applied to verify the carbonated area. A chloride penetration test was also performed, where the artifacts were immersed in a 3% NaCl solution for up to 90 days to assess the porosity of the concrete. In this test, greater concrete strength correlates with lower porosity. The third test conducted was the alkali-aggregate reaction test, which investigates the reactive potential of the aggregate. These reactions can be prevented by using inert aggregates, making this test crucial.

Materials

Coppes Waster

The copper waste used in this experiment as a partial substitute for fine aggregates was collected according to NBR 10007 [5], from the tailings dam at the Sossego Mine, at various points along the dam. Approximately 250 kg of waste was transported to Marabá and Belém. The material was stored in plastic bags of approximately 15 kg each and then sent to laboratories for characterization and testing. For the tests, the samples were homogenized, quartered, and reduced following NBR NM 27 [6].

Pig Iron Slag

The pig iron slag used in this experiment as a partial substitute for coarse aggregates was collected from the Sinobrás steel mill in Marabá. The collection and characterization procedures were performed following NBR 10007 [5]. Approximately 200 kg of waste was collected from the water jet cooling area. The material was stored in plastic bags weighing approximately 15 kg each, and the samples were then sent to laboratories for characterization. For the tests, the samples were homogenized, quartered, and reduced according to NBR NM 27 [6].

Methods

The concrete test specimens for this study were molded and cured according to the recommendations of ABNT NBR 5738 [7]. The concrete mix ratio used was 1:2:3 by weight. Four substitution percentages were defined for replacing fine aggregates with copper waste (15%, 25%, 50%, 75%, and 100%) and pig iron slag (15%, 25%, 50%, 75%, and 100%). These percentages were selected based on parameters from similar studies described in the literature. Research conducted in 2019 produced concrete samples with copper waste additions at substitution levels of 15%, 30%, and 45% of fine aggregates, demonstrating satisfactory results in mechanical tests [8].

After analyzing the specific mass of the two materials in question, their values were found to be similar, and weight-based substitution was adopted. A total of 135 test specimens (TSs) were prepared with the following proportions: 15% copper waste (CW) and 15% pig iron slag (PIS), 25% CW and 25% PIS, 50% CW and 50% PIS, 75% CW and 75% PIS, and 100% CW and 100% PIS. The remaining proportions will complement the mix design, which will remain unchanged.

Scanning Electron Microscopy (SEM)

The experimental technique used, Scanning Electron Microscopy (SEM), is an advanced imaging method that allows for the examination of material surfaces with high resolution. When applied to concrete blocks, SEM provides detailed insights into the morphology, structure, and surface characteristics of the concrete's constituent materials [9,10].

The morphology of the particles comprising the copper waste was obtained using Scanning Electron Microscopy (SEM), performed with a ZEISS EVO MA10 microscope equipped with secondary electron and backscattered electron detectors. The analysis will be carried out at the laboratory of the Geosciences Institute (IGE) at the Federal University of Southern and Southeastern Pará (Unifesspa). Employing SEM techniques enables in-depth analysis of both specific sample surfaces and the structure of concretes containing waste materials. This approach enhances the understanding of mechanical behavior and improves the evaluation of the obtained results.

Durability Tests

Durability refers to a material's ability to resist and maintain its functionality over a predetermined time interval. NBR 6118 [11] defines durability as the ability of a structure to withstand environmental influences specified at the beginning of the project, as agreed upon by the designer and the client. Meanwhile, NBR 15575-1 [12] defines durability as a building or component's ability to maintain its performance over time.

Concrete deterioration can be classified into physical, chemical, and mechanical causes. Reinforcement corrosion may result from carbonation, exposure to chloride ions, or a combination of both. Brazilian guidelines on environmental aggressiveness mainly consider these two agents of deterioration [13].

Carbonation Tests

The accelerated carbonation test followed the methodology described by Ribeiro et al., based on the parameters established by the European standard ISO 1920-12:2015 [14], which addresses material durability. The test was conducted at the Federal University of Southern and Southeastern Pará (Unifesspa) in Marabá, at the Civil Engineering Materials Laboratory. A chamber was prepared with a controlled CO_2 level of 5%, maintained in an environment with a relative humidity of 68% ± 2% and a temperature of 21°C ± 2°C.

For this analysis, three cylindrical concrete specimens (10 x 15 cm) were prepared for each dosage (0%, 15%, 25%, 50%, 75%, and 100%), totaling 18 units, as shown in Figure 5. Once demolded, these cylinders were wrapped in plastic to prevent any interaction with the external environment and were stored in a humid chamber for curing over 30 days. After this period, they were weighed and transferred to a humidity equilibrium chamber. Once equilibrium was reached, the specimens were placed in a carbonation chamber and removed only for measurements at 30, 60, and 90 days.

The specimens were removed from the carbonation chamber and fractured using a compression press to extract a section, referred to as a "sample." After extraction, the fractured section was cleaned and treated with a phenolphthalein solution. Following a 20-minute interval, the thicknesses of the carbonated areas were measured. Measurements were taken to assess the carbonation depth in the cylinder sections using a digital caliper with a resolution of 0.01 mm. Measurements were performed at five approximately equidistant points along each side of the section, as illustrated in Figure 5. The mean depth (d) was calculated by averaging the 20 individual measurements (in millimeters) for each analyzed slice, considering the concrete and the corresponding age of each sample.

Figure 2: Measurement Scheme Extracted from Test Specimens (TSs) via Circular Saw Cutting [7].

For the purpose of standardizing the test, procedures were established for the accelerated carbonation chamber based on the recommendations of the European standard ISO 1920-12, as well as suggestions from other authors [15,7].

Chloride Penetration Tests

According to some researchers, the presence of aggressive elements during concrete preparation is common. The main agent among them is the ion Cl−Cl^-Cl−, which can be introduced through setting accelerators, aggregates, contaminated water, or cleaning procedures [7].

In the chloride penetration test, the artifacts produced in the Civil Engineering Laboratory at UNIFESSPA were transported to Belém, specifically to the Civil Engineering Laboratory (LEC) at UFPA. In this facility, the test specimens were partially submerged in a saline solution prepared with sodium chloride (NaCl) dissolved in water, reaching a concentration of 3.5%. This value was adopted to simulate the average salinity of oceans, as described by Vieira (2019), and is widely used in studies on chloride penetration in concrete concreto [16].

The analysis of chloride penetration will be conducted after 90 days of exposure, following methodologies recognized by various researchers to investigate the effects of these ions on concrete durability.

During sample preparation and up to the test itself, after demolding, the specimens will undergo wet curing, remaining submerged for 90 days. This allows the pozzolanic action of the cement (CP IV) to intensify during hydration [17,18], as described in European standards that inspired the test methodology. After curing, the samples will be dried in an oven at a temperature of 50±5°C50 ± 5°C50±5°C for 48 hours until they reach a constant mass.

By adopting a 3% NaCl solution concentration to simulate the salinity of ocean waters, two distinct moments will be considered for test execution. This aims to understand the evolution of the phenomenon over time, although only the final result will be used to classify the concrete's resistance to chloride penetration. The minimum test duration, as recommended by standards, exceeds 35 days. After each test period, the specimens will be removed from the NaCl solution and left to air-dry for 24 hours to allow humidity to stabilize with the environment.

Subsequently, the specimens will be sectioned longitudinally using compressive force, resulting in two equal halves. A fine mist of silver nitrate ($AgNO_3$) solution (0.1%) will be sprayed on both parts using a manual sprayer, following the recommendations of previous studies [7].

After the solution dries on the cross-sections of the test specimens and clear demarcation is visible with “whitish” coloration (chloride-contaminated area) and “brown” coloration (chloride-free area), measurements will be taken. Using a digital caliper (Digimess® - 0.01mm resolution), each side of the exposed section will be measured. Based on these data, the depth of chloride ion penetration will be calculated, averaging the measurements from both halves of the same prism.

Alkali-Aggregate Reaction Based on NBR 15.577-1

The Brazilian Committee on Cement, Concrete, and Aggregates (ABNT/CB18), through the Study Commission on Requirements and Testing Methods for Aggregates (CE-18:200.01), published in 2008 a set of standards known as NBR 15577 [19].

This standard establishes the necessary measures to prevent the occurrence of deleterious expansive reactions, provides guidelines for assessing the risk of pathological manifestations related to the alkali-aggregate reaction (AAR), prescribes requirements for analyzing potential reactivity, indicates the methodology for applying tests on collected samples, and recommends preventive measures for the use of aggregates in concrete when they are potentially reactive or when there is uncertainty about their reactivity [20].

Stain Method – Qualitative Analysis of AAR

The test was carried out in the Total Mix technological control laboratory located in Belém, where the material was sieved following the ABNT NBR 15577 [19], standard. The aggregates were passed through sieves with mesh sizes of 4.74 mm, 2.36 mm, 1.18 mm, 600 μm, and 300 μm. After weighing, the materials were mixed and molded.

After 30 days immersed in the chemical reagents used in the research—comprising Na-cobaltinitrite and rhodamine B base—the alkali-aggregate reaction (AAR) in affected regions of the samples was analyzed. Accelerated laboratory samples were also tested.

The use of the Na-cobaltinitrite reagent on samples identifies reaction edges at the paste/aggregate interfaces and in pores filled with xerogel, turning them yellowish. The presence of AAR products in regions affected by the reagents was confirmed through microstructural analyses.

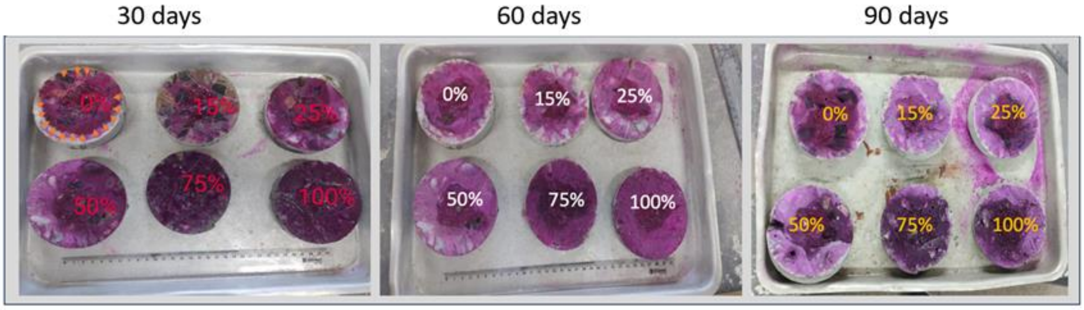
The inclusion of the stain test as a diagnostic tool for detecting AAR in accelerated samples is highly recommended for identifying areas degraded by the reaction and evidencing the development of AAR "gel."

III. Result And Discussion

Accelerated Carbonation Test

After the period established in this study, it was possible to diagnose the area affected by carbonation. The colorless, carbonated area (pH < 8.5) and the non-carbonated area (crimson pink; pH > 8.5) are shown in Figure 3. The depth of carbonation on the faces of the concrete specimens (CPs) was measured using a digital caliper (resolution 0.01 mm), with measurements taken at approximately 5 points:

Figure 3: Carbonation and measurement by different dosages of CPs produced in Marabá and Belém.

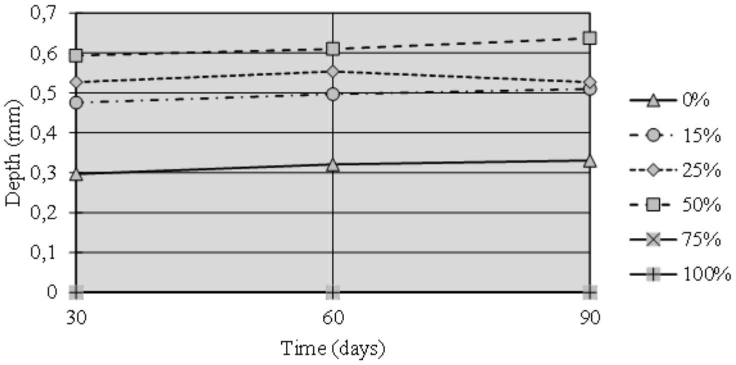


The result of the measurements is represented by the average of the 20 individual measurements (in millimeters) from each analyzed section of the same concrete, at their respective ages[7].

Measurements were taken for all 6 dosages, following the guidelines of the ISO 1920-12 standard[14] and the recommendations of Ribeiro [7].

The concretes were then classified according to the methodology described in these studies and standards, providing a consistent evaluation of the progression of carbonation over time. As shown in Figure 4.

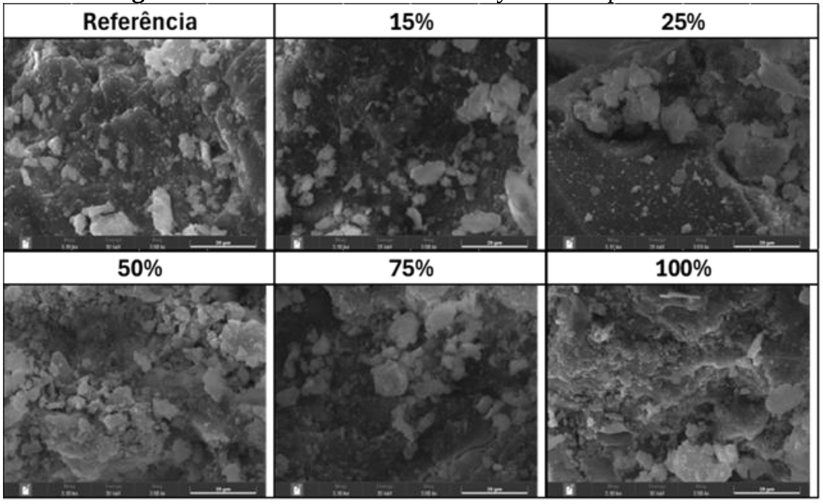
Figure 4: Carbonation depth over time.



The results indicated that the greatest carbonation depth occurred in the mixture with 50% substitution of mineral aggregates (0.59 mm to 0.63 mm), while the lowest attack was observed in the reference concrete (0% substitution), with a depth of 0.29 mm to 0.33 mm. The mixtures with mineral additions, especially in the 15%, 25%, and 50% dosages, showed higher CO_2 penetration compared to the reference concrete at all evaluated ages. This is attributed to the reduction in the portlandite content due to the mineral additions, resulting in lower pH and greater susceptibility to carbonation.

However, the mixtures with 75% and 100% substitution showed different behavior, with a lower carbonation attack depth. Scanning electron microscopy (SEM) analysis, as shown in Figure 5, revealed a denser microstructure with lower porosity due to the increased density of crystals promoted by the hydration of the mineral residues. This enhanced microstructure justifies the greater resistance to the penetration of aggressive agents observed in these dosages, suggesting a potential increase in the material's durability.

Figure 5: Microstructure of the analyzed test specimens.



Chloride Penetration Test

The chloride ion penetration test was conducted at the civil engineering laboratory of UFPA in Belém, and the concrete specimens produced in Marabá were transported there for the test. After the specified exposure period to the solution, longitudinal sections were made, leaving them exposed for the application of a fine mist of silver nitrate solution ($AgNO_3$ at 0.1%). A manual sprayer was used for this, and a mixture of silver nitrate and water was prepared for dissolution, as recommended by [7].

Figure 6: Test specimens after chloride ion penetration test at UFPA/Belém

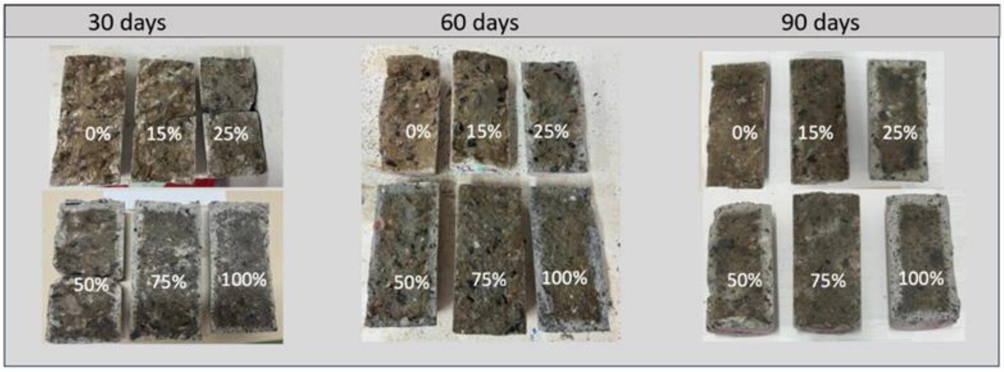
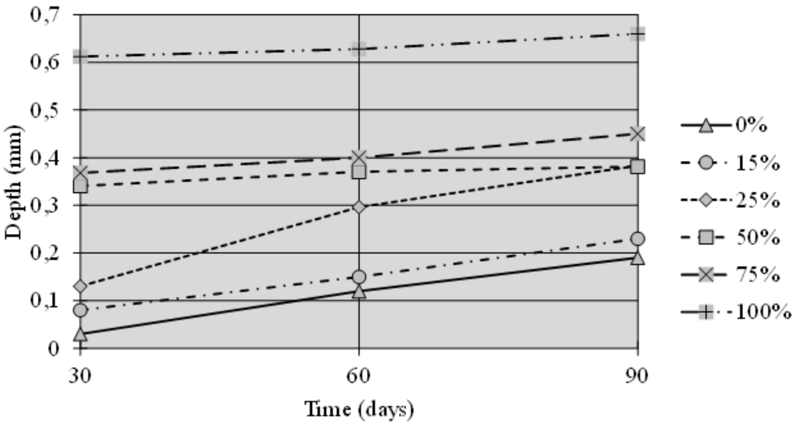


Figure 7: Chloride penetration depth over time.



According to Figure 7, it is possible to observe that the higher the replacement of natural aggregates by copper and pig iron waste, the greater the chloride penetration. However, even with higher replacements, such as 75% and 100%, it is still possible to use the waste.

Alkali-Aggregate Reaction Test

The alkali-aggregate reaction test was conducted at the Total Mix control laboratory, located in Belém, Pará. This test is of great importance as it allows the evaluation of the alkali-aggregate reactivity potential of copper ore waste and pig iron slag. The objective was to determine the expansion in mortar bars by the accelerated method, analyzing the variation in the length of the bars and assessing the susceptibility of the aggregates to the alkali-silica expansive reaction in the presence of hydroxyl ions associated with alkalis (sodium and potassium).

For the test, standard CP II-F 32 cement was used, sourced from the Nassau factory located in Primavera, Pará. The tests for studying the alkali-aggregate reaction (AAR) potential of the two samples were conducted from October 17 to November 21, 2024, as presented in Figures 8 and 9.

Figure 8: Alkali-aggregate reaction for copper waste

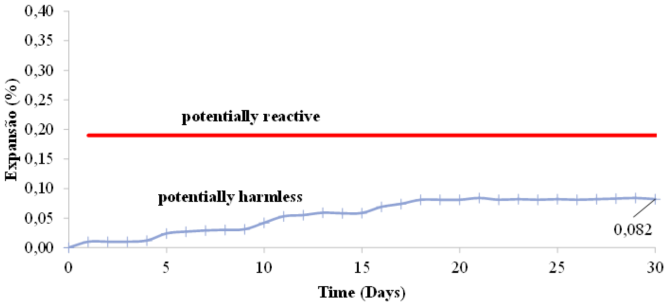
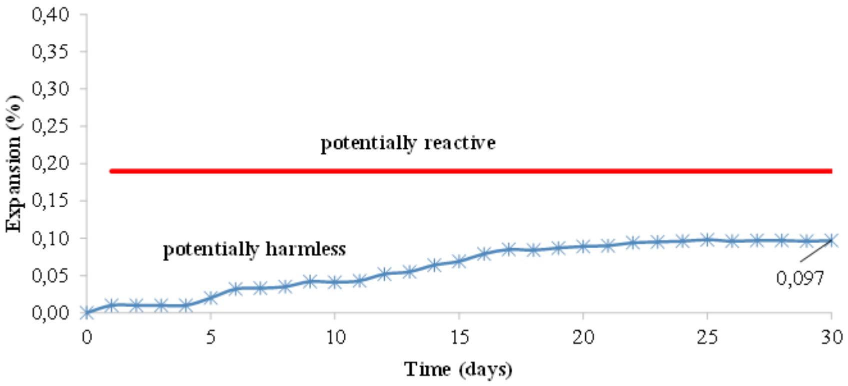


Figure 9: Alkali-aggregate reaction for pig iron slag waste



In the alkali-aggregate reaction test conducted with cement, it was confirmed that the samples of copper ore waste and pig iron slag did not show reactive potential, as per the test described in NBR 15577-4 [19]. This indicates that these materials do not possess alkali-aggregate reactivity potential, and therefore, they are suitable for use in concrete production.

IV. Conclusion

This study aimed to evaluate the durability of concrete at different mix proportions, in which coarse aggregates were replaced by pig iron slag and fine aggregates by copper waste, both sourced from mining companies in the southern and southeastern regions of Pará. The dosages analyzed were 15%, 25%, 50%, 75%, and 100%, along with the reference concrete (0%) for comparison with conventional concrete.

The tests conducted, including carbonation, chloride penetration, and alkali-aggregate reaction (AAR), allowed the determination of the performance of these new concretes concerning durability and the feasibility of replacing natural aggregates, with the aim of reducing environmental impact.

Based on the experimental results and discussions, the following conclusions were drawn:
Carbonation: The mixtures with 75% and 100% replacement showed better performance, demonstrating high resistance to carbonation. These concretes were classified as exceptionally durable according to the criteria of Ribeiro and collaborators and ISO 1920-12, surpassing the performance of conventional concrete.

Chloride Penetration: The concretes with 75% and 100% replacement demonstrated higher resistance to chloride penetration. These results, classified as resistant according to the documented methodology in the literature, indicate that these formulations are suitable for structures exposed to aggressive environments.

Alkali-Aggregate Reaction (AAR): The tests confirmed that the pig iron slag and copper waste do not have reactive potential to alkalis, according to the criteria established by NBR 15577-4. This demonstrates the suitability of these materials for use in concrete without the risk of pathological expansion associated with AAR.

In summary, the concretes with partial or total replacement of natural aggregates by mineral waste exhibited excellent durability in the conducted tests, showing comparable or superior properties to conventional concrete. The pozzolanic effect associated with the waste, although requiring higher water consumption to achieve the ideal slump, contributed to the increase in microstructure density and, consequently, the durability of the structures.

This approach stands out as a viable and sustainable alternative for the construction industry, offering significant benefits to both sectors involved. The mining industry finds an efficient solution for the disposal of its waste, reducing environmental liabilities, while the construction industry incorporates these materials, promoting more sustainable practices and minimizing future environmental impacts. Thus, this study reinforces the technical and environmental feasibility of using mineral waste in concrete production, encouraging the adoption of innovative and responsible practices.

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