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Petrographic Characterization of Concretes with Replacement of Coarse Aggregates by Steel Slag

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Abstract
The replacement of aggregates in concrete formulations is a common practice for improving properties such as strength, durability, impermeability, and workability. One material used for this purpose is slag, a by-product of the steel industry. During the setting and hardening of concrete, cracks may form, which play a critical role in the penetration of corrosive agents that degrade its properties. The objective of this work was to study the Interfacial Transition Zone between the cement paste and slag aggregate, due to the significant impact of microcracking on concrete properties. The micro-structural fractographic study focused on this aggregate-paste transition zone, which is generally the weakest link and is considered the strength-limiting phase of concrete. This study provides valuable information on the material's properties, especially its defects. Polished and fractured surfaces of concrete with different proportions of steel slag were characterized by scanning electron microscopy. All concrete samples with a replacement rate of 25% to 100% showed a dense internal structure with well-bonded paste-slag interfaces. Only isolated microcracks were observed in the cement paste, within which new hydration products are expected to form over time, potentially enhancing the concrete's long-term strength and durability. Optical microscopic examination of in sections confirmed the dense and compact structure of the concrete. The resulting good compressive strength demonstrated the advantages of incorporating slag.

Keywords: Steel Slag, Concrete, Petrography, Optical Microscope, Scanning Electron Microscope.

Introduction

Concrete, similar to refractory materials used for ornamental tiles, can be used to immobilize environmentally polluting substances, simultaneously enhancing the material's properties. For instance, ornamental tiles immobilize heavy metals from electroplating waste, which are difficult to eliminate by other means. In concrete, natural aggregates can be replaced by electric furnace slag, a by-product of the steel industry. The use of such industrial waste reduces the consumption of non-renewable natural resources; a prime example is the utilization of artificial and recycled aggregates like construction and demolition waste, municipal solid waste, and steel slag (blast furnace slag, electric arc furnace slag) ☐1-4☐. Electric arc furnace slag is one of the most common waste products generated by the steel industry worldwide. According to the World Steel Association, global crude steel production increases annually, underscoring the importance of valorizing these wastes [5].

The replacement of aggregates in concrete formulations is a common practice to improve properties such as strength, durability, impermeability, and workability. The penetration of aggressive agents through the concrete matrix causes significant deterioration, ultimately destroying reinforced concrete structures. This deterioration is not an isolated phenomenon but is interrelated and depends on numerous factors. The transport of these aggressive agents into concrete occurs primarily through its macropores and fissures, as well as through the interconnected capillary pores of the hydrated cement paste and the microcracks that develop within it. These pathways are critical for the ingress of corrosive agents that degrade the properties of reinforced concrete. The objective of this work is to study the interface between the cement paste and the slag during setting, to determine the occurrence of cracks, and to evaluate their influence on the properties of Portland cement concrete.

It is well-established that the interfacial transition zone (ITZ) between the cement paste and the aggregates plays a crucial role in the properties of hydraulic concrete, including its strength, stiffness, and durability. This transition zone is the most critical phase in concrete, even more so than the aggregates or the hydrated cement paste itself. A higher-quality transition zone leads to better mechanical behavior of the concrete [6]. In many countries, concrete quality control is predominantly based on mechanical strength, with durability assessments being rarely applied. This is a significant concern for a country with high levels of environmental aggressiveness harmful to concrete [7]. Therefore, it is important to incorporate complementary analyses, such as the study of fracture surfaces by scanning electron microscopy (SEM) and transmission optical microscopy (OM), to corroborate other quality control results. The wider application of these analyses in standard testing could help prevent structural deterioration failures and the need for costly repairs.

Scanning electron microscopy (SEM) enables the characterization and interpretation of the relationships between components on material fracture surfaces [8]. Fracture facies provide valuable information about material properties, especially their defects. Poor interfaces act as initiation sites for fractal crack patterns, and there is a close relationship between fractures and fractals [9]. A micro-mechanism of bridge formation explains the increased fracture resistance of tough ceramics like concrete, where particles that do not break help to prevent crack propagation [10].

Petrographic study of thin concrete sections by transmission optical microscopy (OM) can reveal the homogeneity of the concrete's components. Concrete has a heterogeneous and complex structure, with interrelated macro- and micro-structural features. Understanding the structure and properties of its components, as well as the relationships between them, is essential for predicting the strength, dimensional stability, and long-term durability of concrete [11].

Methodology

The natural aggregates used in the concrete mixtures were gravel and sand from Dragón Camoa [12]. The artificial aggregate used to replace the coarse natural aggregate was black slag, a waste product from the "José Martí" steel company. The binder used was Portland cement P-35, and a superplasticizer additive, Dynamon SR-356, was employed as a water reducer.

The analyzed samples consisted of concrete with coarse aggregates replaced by black slag at replacement rates of 25%, 50%, 75%, and 100%. A reference sample without any replacement (standard mixture) was also prepared for comparison. All tests were performed 28 days after the concrete was prepared.

The following equipment was used for microstructural characterization:

- A TESCAN Vega 3 high-vacuum scanning electron microscope (SEM).
- A Zeiss Primo Star optical microscope equipped with a Canon EOS 1000 D#2 camera.
- Macrographs were captured using a Samsung Galaxy A14 5G cell phone (model SM-A146U1/DS)

Results and Discussion

Microstructural analysis conducted 28 days after preparation revealed a homogeneous distribution of components within the three constituent phases of the concrete. Figure 1 shows the surface of the reference concrete without slag. Figure 1a presents a micrograph of the polished surface, while Figure 1b shows the fractured surface. Both micrographs reveal well-defined aggregate-paste interfaces. Voids between the edges, not exceeding 4 µm, are also visible. These voids in the paste may have contained traces of hydration water that were removed by the vacuum during sample preparation and SEM analysis. Hydrated calcium silicates could crystallize in these spaces as a result of slow chemical reactions between the cement paste and the aggregates. These interactions in the transition zone contribute to improved concrete strength by reducing the concentration of calcium hydroxide.

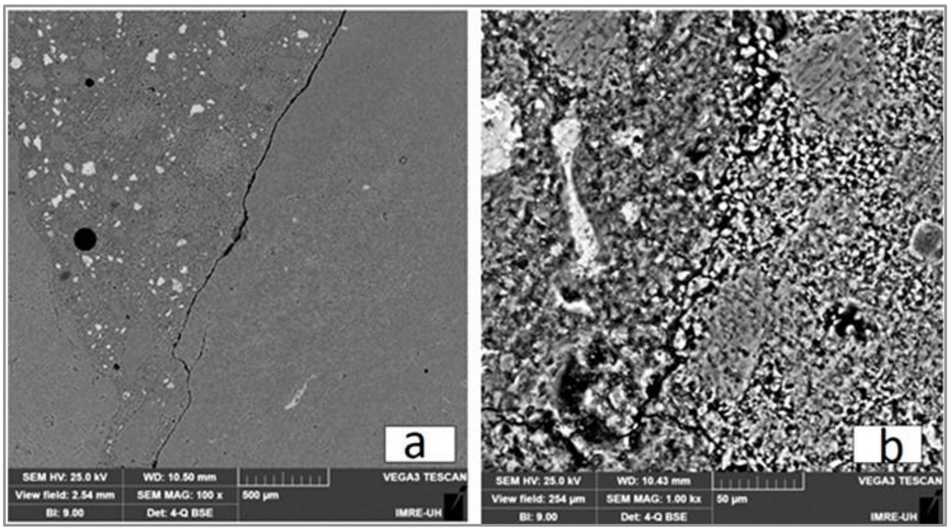

Figure 1: Surface of reference concrete. Backscattered electron (BSE) micrographs of the polished (a) at 100x and fractured (b) at 1000x.

Backscattered electron (BSE) micrographs of the concrete with 75% slag replacement show good contact between the cement paste and slag on both the polished (Fig. 2a) and fractured (Fig. 2b) surfaces. Subsequent shrinkage of the cement paste was less than 15 µm.

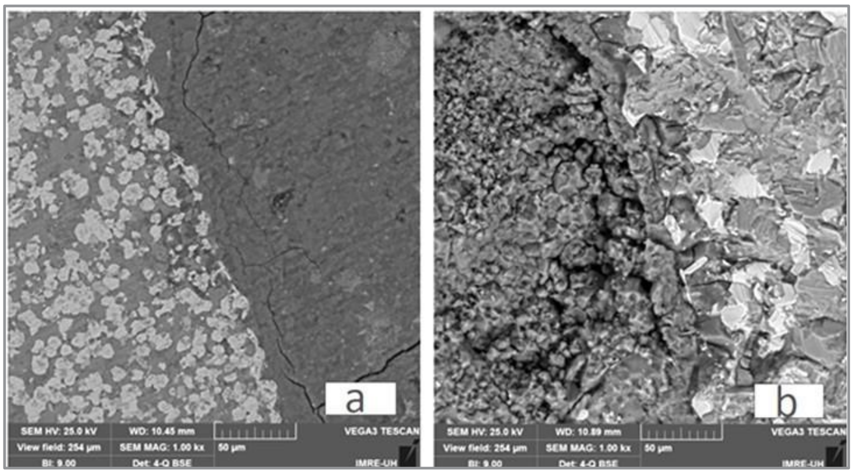

Figure 2: Concrete with 75% replacement. Backscattered electron (BSE) micrographs at 1000x of the polished (a) and fractured (b) sample surfaces.

In the concrete with 100% slag replacement, a coherent interface between the cement paste and the slag particles was observed on both the polished (Fig. 3a) and fractured (Fig. 3b) surfaces.

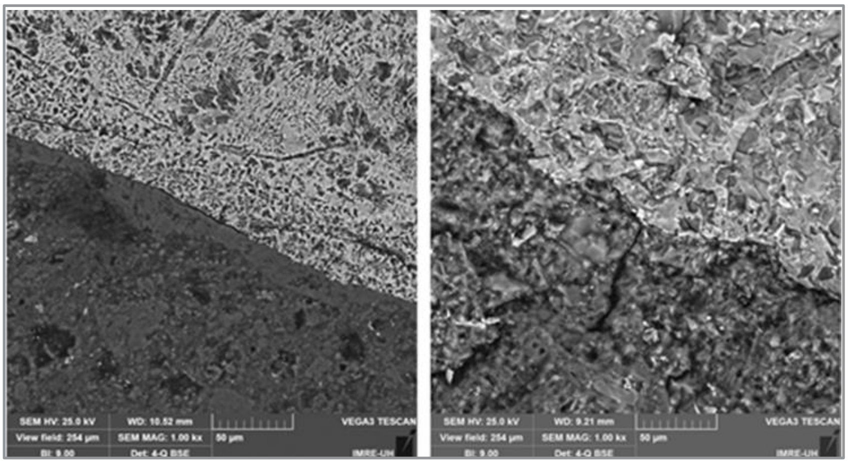

Figure 3: Concrete with 100% replacement. Backscattered electron (BSE) micrographs at 1000x of the (a) polished and (b) fractured sample surfaces.

The petrographic study by Optical Microscopy (OM) of the reference and slag-containing samples reinforces the conclusions derived from the SEM analysis. The OM micrographs show a uniform paste coloration and a good distribution of fine aggregates of varying sizes. The coarse aggregates, appearing in yellow, orange, brown, and gray with angular, subangular, and sub-rounded shapes, show no significant porosity but exhibit some internal fissures. Their size ranges from 10 µm to 10 mm. The black slag particles have a more pointed and irregular shape, with a dark gray or black color indicative of their chemical composition. Good adhesion between the aggregates and the paste was observed, with no apparent adverse reactions. No significant level of pores or cracks was detected.

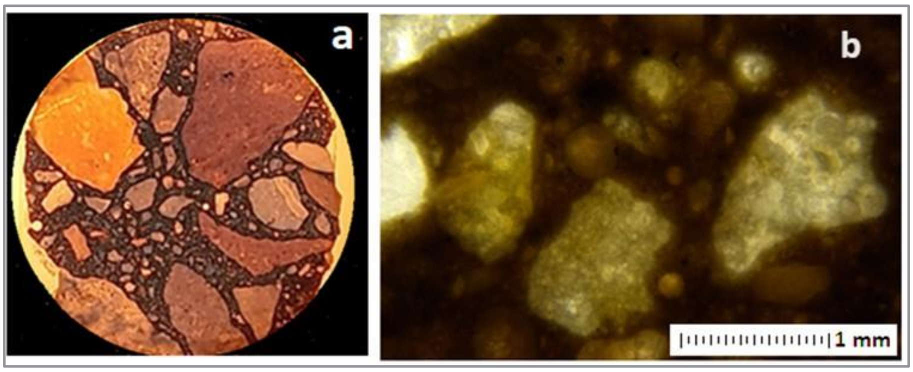

Figure 4: Transmission images of the thin reference sample. a) Macrograph; b) OM micrograph.

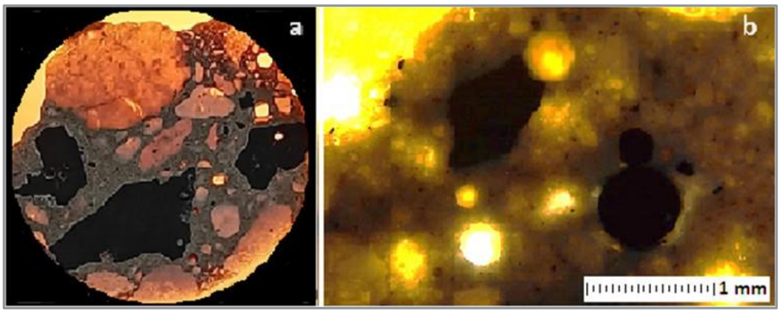

Figure 5: Transmission images of the thin sample with 25% slag replacement. a) Macrograph; b) OM micrograph

The compressive strength of the specimens tested between 28 and 90 days after preparation (Table 1) shows that all values substantially exceeded the design value of 30 MPa. The strength increase over this period was approximately 15%. Although the standard concrete (0% slag) exhibited slightly higher strength, the concretes with slag replacement are expected to show a greater increase in compressive strength over time. This anticipated gain is due to the superior cohesion achieved between the slag aggregates and the cement paste, attributed to the slag's high intrinsic mechanical strength and greater surface roughness.

Table 1: Compressive strength as a function of curing time.

Concrete Mixture	28 days (MPa)	90 days (MPa)	Strength Increase (%)
Reference (0%)	44	50	14
25 %	39	47	21
50 %	38	43	14
75 %	39	45	15
100 %	38	44	16

Conclusions

Petrographic analysis successfully characterized the morphology of the interfacial transition zones (ITZs) in concrete samples where natural aggregates were replaced with steel industry slag. The optical microscopy (OM) study confirmed the homogeneity of the concrete's components, while scanning electron microscopy (SEM) at high magnification provided high-quality images of the polished and fractured surfaces, revealing critical microstructural details.

The reference concrete without slag replacement exhibited a higher density and more uniform distribution of hydration products compared to the slag-containing concretes. Nevertheless, all concrete formulations, including those with up to 100% slag replacement, demonstrated a dense internal structure in the cement matrix. It is expected that this microstructure will continue to improve over time with the formation of new hydration products during concrete aging. This ongoing process is anticipated to enhance the compressive strength and long-term durability of the slag-based concretes, confirming the viability of slag as a sustainable alternative aggregate.

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