



Microstructural analysis of interfacial transition zone (ITZ) and its impact on the compressive strength of lightweight concretes



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HIGHLIGHTS

- Microstructure in the ITZ of lightweight concretes was studied.
- Lightweight aggregates (LWA) contributed to the formation of a dense and thinner ITZ.
- Lightweight structural concretes were developed and explained.

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ABSTRACT

In this research both the microstructure and thickness of the interfacial transition zone (ITZ) in concretes of Portland cement and lightweight aggregates (LWA) are studied. It has been established that the microstructure in the ITZ strongly depends on the nature of the aggregate, specifically its porosity and water absorption. This study aims at researching the influence of physical properties such as density, porosity and morphology of lightweight aggregates such as pumice and expanded clays, on the microstructure and thickness of ITZ, and determine the effect that these factors have in turn on the mechanical properties as compressive strength of lightweight concretes (LWC). Lightweight aggregates were characterized by X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and X-ray Fluorescence (XRF), to determine their mineralogical, morphological and chemical characteristics. The characterization of ITZ by SEM-EDS, and conventional optical microscopy, was carried out on specimens of concrete manufactured with LWA and with a conventional aggregate, in order to evaluate its thickness; furthermore, to determine the porosity, digital image processing (DIP) was performed. Lightweight aggregates contributed to the formation of a dense and thinner ITZ, when compare to the ITZ of a conventional concrete. The lower porosity and greater amount of hydrated cement phases in the ITZ of lightweight aggregates are attributed to their physical, morphological properties and chemical and mineralogical composition; which contributed to the decrease of the wall effect, gestating from its surface the formation of C-S-H, achieving interlacing of hydrated phases on the surface of these aggregates with the cementitious matrix.

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1. Introduction

The ITZ is a layer formed between an aggregate and the matrix of cement paste, composed of a double layer “duplex film” of calcium hydroxide crystals ($\text{Ca}(\text{OH})_2$) oriented to the aggregate's side and hydrated calcium silicate, C-S-H gel, oriented to the side of the paste, with a thickness of about 1 μm . Farthest from the aggregates is the main interface zone of about 40–50 μm thick, containing larger crystals of calcium hydroxide [1]. In this zone the cement particles are unable to bind intimately with the relatively large

particles of the aggregate, “the wall effect”; consequently, the ITZ has a much higher porosity (2 to 3 times) than that of the hardened cement paste farthest from the aggregate particles [2]. The ITZ properties are affected by the characteristics of the aggregate and the cementitious matrix, as well. Several researchers have found that the grain size distribution of the cement, the water cement ratio, the aggregate size, and the type of aggregate have important effects on the ITZ properties [1–4].

Significant efforts have been devoted to optimizing the dosage and to the study of mechanical properties of lightweight concrete (LWC), to make them competent with normal weight concretes [5–7], which have led to the study of ITZ microstructure [3,4,8–11]. Finding that the mechanisms responsible for the ITZ formation

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are related among others with absorption and water release, which is a feature of the lightweight aggregates (LWA) themselves, due to their usual high porosity. They have higher absorption and subsequent release of water, increasing the hydration degree of the paste around the aggregate and the amount of free water, making the adjacent paste to develop a structure with greater porosity [7,8]. It is often assumed that the LWA used in the manufacture of concrete have less chemical reactivity and their interaction with the cement matrix is mainly physical [7]; therefore, the chemical influence of the LWA on the ITZ properties have received less attention.

Researches carried out by Elsharief et al. and Xiao et al. [3,4] state that both size and texture of the aggregates surface have great influence on the ITZ properties. Authors like Yu et al. [12], along with Kong et al. [13]; denote that both physical and chemical interactions that occur between LWA and the cement paste are responsible for the ITZ mechanical strength.

Some studies have shown that the water absorption and degree of LWA pre-wetting may influence the interface formation process [9]; however, further research is needed to fully understand the chemical and physical process between the LWA and cement paste, which depends on the mineralogical composition, the texture and shape of the aggregates, as well. It appears that the rougher the aggregate the larger the contact surface with the cement paste, and thus, greater adhesion [14,5,15,16].

This paper focuses on the study of the incidence of the morphology and composition (chemical and mineralogical) of the LWA in the formation of ITZ microstructure and thickness, and also its influence on the LWC compressive strength, seeking to develop structural lightweight concretes.

2. Materials and methods

2.1. Methodology

The chemical characterization of raw materials was performed through X-ray Fluorescence (XRF), using a Panalytical equipment; model Axios, through quantitative analysis over pearl per X-ray Fluorescence per dispersive wavelength.

The mineralogical analysis was performed in an equipment of X-ray Diffraction (XRD) reference Panalytical X Pert PRO MPD, in an interval 2θ between 4° and 70° , with a passage of 0.02° and an accumulation time of 56 s. A copper anode with $K\alpha = 1.5406 \text{ \AA}$ was used.

The physical properties of aggregates, such as surface area were performed through BET test by chemisorption, the water absorption in compliance with ACI 211.2, [18] and the density is appropriate to ASTM C127, [19]. The shape index (I_a) was calculated by the procedure established in the ASTM D3398 standard [20].

The concrete mixture design was performed in compliance with ACI 213R-03 [23]. Aggregate concrete specimens and Portland cement type I were prepared. The dosage of cement was 500 kg/m^3 , the aggregate was 400 kg/m^3 . The water cement ratio was varied in 0.35 and 0.45. To achieve as the response variable the concrete density and afterwards the compressive strength at 28 days.

The encoding used for the name of the mixtures corresponds to the abbreviation of the aggregate used: pumice (PO), aliven (AL) and conventional (CO), followed by the water/cement ratio of the mixture, 0.35 or 0.45. Once the concrete specimens were fabricated they were taken to 28 days curing as suggested by ASTM C511 [24], in immersion in water saturated with lime ($\text{Ca}(\text{OH})_2$). After reaching the age of curing, the compressive strength test to the corresponding samples was conducted in compliance with ASTM C109 standard [25], at ages 3 and 28 days of curing.

Taking micrographs was performed using both optical microscopy and Scanning Electron Microscopy (SEM), the hydration

process stopped, drying the samples in an oven at 60°C and afterwards they were taken for cutting, the sub samples of 2 cm^2 for ITZ morphological analysis. The SEM micrographs were performed in a SEM JEOL JSM 5910LV with detectors of backscattering electrons and applying 15 kV to image generation and a working distance of 10 mm. The samples were subjected to high vacuum and were then coated with an 8 nm layer of gold-palladium to enhance their electrical conductivity. Micrographs were taken in a Nikon stereoscope reference Eclipse LV100, objectives 2x, up to 11.5x, the stereoscopy micrographs allowed digital image processing (DIP) for the porosimetry analysis; which describes the pores in a section of the aggregate, when adding all images it is sought to recreate the pores area of a complete aggregate.

2.2. Materials and characterization

In the development of this study a normal weight conventional aggregate (CO) and two LWA, pumice (PO), thermally expanded clay, aliven (AL) and Portland cement type I (OPC) were used. In Table 1 is shown the chemical composition of each one obtained by XRF.

The chemical composition of aggregates corresponds to silicon oxide, aluminium oxide, iron oxide, and in lesser proportion magnesium, sodium, calcium, and potassium oxides.

The mineralogical composition for the aggregates is given in the diffractograms of Figs. 1–3. In Fig. 1, can be seen that for the pumice a broad peak is formed just between positions 2θ in 20° and 30° , where the characteristic peak of quartz is around 26.5° ,

Table 1
Chemical composition of aggregates and cement.

Chemical composition	Weight%			
	PO	AL	CO	OPC
Silicon Oxide (SiO_2)	72.1	59.67	58.43	20.9
Aluminum oxide (Al_2O_3)	13.35	16.95	13.46	4.72
Iron oxide (Fe_2O_3)	1.30	9.79	8.33	3.20
Magnesium oxide (MgO)	0.05	4.13	6.00	1.80
Calcium oxide (CaO)	1.22	3.57	7.17	60.69
Sodium oxide (Na_2O)	3.35	2.07	1.89	0.37
Potassium oxide (K_2O)	4.63	1.28	1.02	0.61
Sulfur oxide (SO_3)	0.08	0.04	0.09	0.13
Ignition losses at 1000°C	2.93	0.75	2.40	3.68

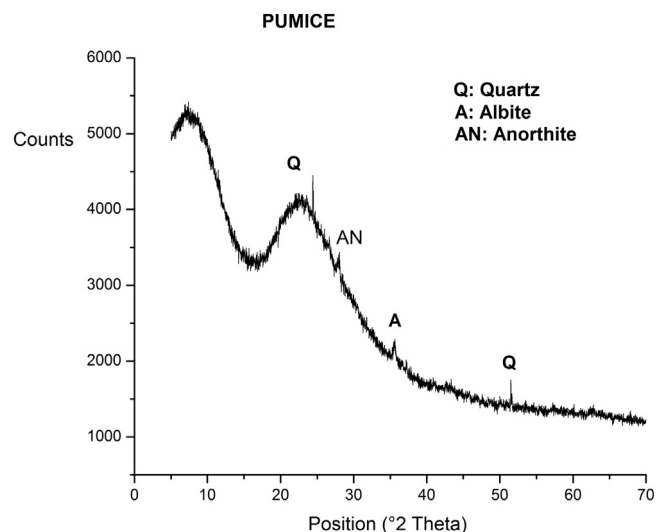


Fig. 1. LWA pumice diffractogram.

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