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Homenaje al Prof. Emilio Galán Huertos
Microstructure and Mineralogy of Lightweight Aggregates Produced from Washing...

# Microstructure and Mineralogy of Lightweight Aggregates Produced from Washing Aggregate Sludge and Clay-rich Sediment

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## INTRODUCTION

Lightweight aggregates (LWAs) can be used in a wide range of applications, such as: acoustic and thermal insulation, manufacturing of structural lightweight concrete, geotechnical applications, and gardening *(Bodycomb and Stokowski, 2000)*. Each application depends on the physical and chemical properties of LWAs. In turn, it has been reported *(Rice and Dekker, 1998)* that important ceramic properties, such as density and water absorption, are intrinsically correlated with the LWA microstructure. Additionally, Farran (1956) showed that the mineralogical composition of the LWAs is the main factor to consider in the study of concrete fracture, since the mineralogy of the LWAs mainly determines the adhesive behaviour between the cement paste and the aggregate.

Here, we describe the mineralogy and microstructure of various artificial LWAs produced by pelletizing and sintering washing aggregate sludge (W) and clay-rich sediment (C). The heating temperature and dwell time effects on the mineralogy and microstructure of the studied LWAs are evaluated. In turn, the influence of these characteristics on the bloating index (BI), density, water absorption ($WA_{24h}$), and compressive strength (S) are analyzed.

## MATERIALS AND METHODS

Different types of LWAs resulting from a previous work *(González-Corrochano et al., 2009)* were selected for the present study. They were manufactured with a 50:50 proportion (weigth %) of washing aggregate sludge:clay-rich sediment (W:C).

The washing aggregate sludge was collec-ted from a representative gravel pit located along the middle sector of the Tagus river. The clay-rich sediment is a lutite from the Miocene Red Clays of the La Sagra formation *(García-Romero et al., 1990)*.

The chemical composition of W and C mainly consist of silica and alumina. The $SiO_2$, $Al_2O_3$ and $FeO+Fe_2O_3$ contents reflect the presence of quartz and phyllosilicates (illite, kaolinite and smectite) in both samples. Calcium oxide was also important in W, due to the presence of carbonate minerals in the sample. $Na_2O$ and $K_2O$ contents can be mainly attributed to the feldspars and to the interlayer cations of the clay minerals *(González-Corrochano et al 2009)*.

The LWA sintering was carried out in a rotary kiln in groups of 25 pellets. The heating temperatures ($T_{heating}$) were 1150°C, 1175°C and 1200°C for dwell times of 10 and 15 minutes.

The LWAs studied in this paper were selected according to previous results *(González-Corrochano et al., 2009)*:

• LWAs heated at 1150ºC for 10 min (W50C50-1150-10): these are the LWAs with the highest water absorption and the lowest compressive strength values (8.45% and 12.55 MPa, respectively).
• LWAs heated at 1150ºC for 15 min (W50C50-1150-15) and at 1200ºC for 15 min (W50C50-1200-15): to compare with W50C50-1150-10 and determine the influence of the temperature and the time on the microstructure and mineralogy of the LWAs.

A scanning electron microscope (JSM-6400), connected to an energy-dispersive X-ray (EDX) analyzer, was used to examine the microstructure of gold-coated surfaces of the LWAs. Electron microprobe analyses (EMPA) were performed on a JEOL JXA-8900 M WD/ED equipment. The bulk mineralogy (BM) was obtained by X-ray diffraction (XRD) after grinding and homogenization of the samples (three entire pellets of each type) to <53 µm. Random-oriented powders were examined on an X´Pert Pro diffractometer, using Cu Kα radiation and operating at 45 kV/40mA.

## RESULTS AND DISCUSSION

Different relationships have been established among the parameters studied. These relationships can be classified into two groups:

### Relationship between Heating Temperature/Dwell Time - Microstructure/Mineralogy

W50C50-1150-10 is quite homogeneous and does not show a distinct external layer *(Fig. 1)*. In general, the material is highly sintered, with slight signs of expansion. Pores of variable diameter are observed. They are mostly regular in shape, spherical, discrete, and closed.
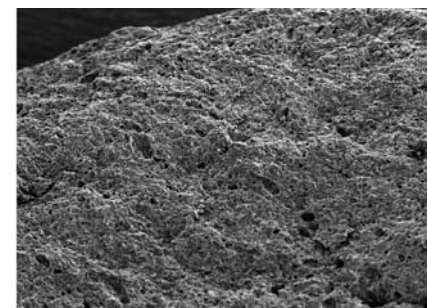


*Fig. 1.* SEM micrograph of W50C50-1150-10(x50)

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**palabras clave:** Árido Ligero, Mineralogía, Microestructura

**key words:** Lightweight Aggregate, Mineralogy, Microstructure.

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Sample W50C50-1200-15 neither has the external layer, although it presents highly vitrified material with a thin external glassy film. As observed in *Fig. 2*, there are clear signs of bloating, with abundant spherical pores within a wide size range, being generally small and isolated. Their distribution throughout the cross-section is more uniform in this LWA, treated at the highest temperature and dwell time, than in W50C59-1150-10 *(Fig. 1)*. The porous structure is due to the formation of gases in the molten material with a pressure slightly higher than required to resist the viscosity of the melted raw materials *(Riley, 1951)*.
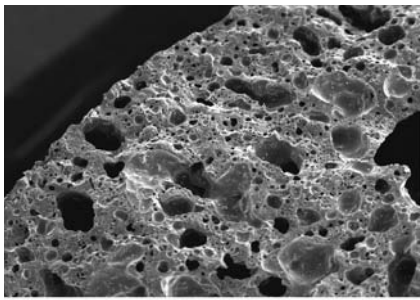


*Fig. 2. SEM micrograph of W50C50-1200-15(x75)*

The mineralogy of the studied LWAs undergoes significant qualitative and quantitative changes with increasing temperature and dwell time. Quartz, plagioclase and pyroxenes are present in both LWAs. Augite is more abundant in samples heated for a dwell time of 10 min, whereas, in those heated for 15 min, diopside is present in a greater proportion. This means that a 5 min increase in the dwell time causes changes among members of the same solid solution. The proportion of pyroxene group minerals decreases with increasing heating temperature, even disappearing, as reported by Romero et al. (2001)

### *Relationship between Microstructure/ Mineralogy - Physical Properties*

BI is influenced by the microstructure of the LWAs. This fact can be clearly seen comparing *Fig. 1 and Fig. 2*: BI of W50C50-1200-15 is higher than BI of W50C50-1150-10 *(González-Corrochano et al., 2009)*, being the microstructure of those LWA less porous than the microstructure of W50C50-1200-15.

The decrease in density in sample W50C50-1200-15 *(González-Corrochano et al., 2009)* is attributed to bloating; this hypothesis is supported by the presence of a large number of pores as shown in *Fig. 2*. Another factor that could contribute to this decrease is the mineralogical change that takes place with an increase in temperature, namely, the disappearance of pyroxenes, mainly augite (density of 3.19-3.56 $g/cm^3$), and the neo-formation of a greater proportion of plagioclase, such as anorthite (average density of 2.73 $g/cm^3$), which entails a change in the density of the solid material in the LWAs.

*Zhang and Gjørv (1991)* have demonstrated the effect of a dense external shell on the reduction of water absorption values. Even though W50C50-1150-10 and W50C50-1200-15 do not show an external shell *(Figs. 1 and 2)*, they have very low water absorption values because they present closed porosity *(Cheeseman and Virdi, 2005)*. In addition, sample W50C50-1200-15 has a vitrified material with an external glassy film, which makes the outer surface impervious to water *(de´Gennaro et al., 2001)*, although new porosity has formed due to bloating.

Although W50C50-1200-15 *(Fig. 2)* presents more and larger pores than W50C50-1150-10 *(Fig. 1)*, the former has a higher compressive strength *(González-Corrochano et al., 2009)*, primarily because the new pores are closed, not fused, regular in shape and size, relatively small *(Cheeseman and Virdi, 2005)* and uniformly distributed, and also to the presence of an external glassy film *(de´Gennaro et al., 2001)*. In addition, the presence of a greater proportion of glassy phase could also influence the higher S value of W50C50-1200-15 compared to W50C50-1150-10, since larger amounts of silicate glass and calcium silicate, especially anorthite, improve the strength of clay-based ceramics *(Sglavo et al., 2000)*.

### CONCLUSIONS

We may conclude that:

• Heating temperature and dwell time affect the degree of vitrification of the LWA material and the formation of pores inside it.
• In the studied LWAs, the main crystalline phases are quartz and neo-formed anorthite, augite, and diopside. With increasing heating temperature, the proportion of quartz decreases, whereas that of plagioclase increases.
• The most important relationships between microstructure/mineralogy and the properties of the LWAs have been:
- higher BI values can be correlated with a greater proportion and larger size of closed inner pores;
- the disappearance of pyroxene and the neo-formation of a greater proportion of plagioclase could produce a decrease in the density of the solid material in the LWAs;
- the presence of isolated pores is a factor as important as the presence of an external shell in the permeability of the LWAs;
- the type and distribution of pores in the LWAs affects the compressive strength values rather than the number of pores;
- the presence of calcium silicates, especially anorthite, and the consumption of quartz during the formation of the glassy phase improve the compressive strength.

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