



## Multiple recycled aggregate properties analysed by X-ray microtomography

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### HIGHLIGHTS

- Multiple recycling properties of recycled aggregate has been analysed.
- Computerized micro-tomography identifies phases of the recycled aggregate.
- After 3 recycling the volume of adhered mortar is 80% of the aggregate.
- Density decreases with recycling cycles and the size of the aggregate.

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### ABSTRACT

This paper presents a novel technique used to analyse the volume of adhered mortar to the recycled aggregate. A computerized microtomograph ( $\mu$ CT) device was used to evaluate the volume of the aggregate, the volume of natural aggregate and the volume of adhered mortar. To this end, a natural aggregate has been characterized, using the  $\mu$ CT, with which a source concrete has been produced. Subsequently, the source concrete has been crushed to obtain a first cycle recycled aggregate. After the characterization of the first-generation of recycled aggregate, a new source concrete has been made with it to be subsequently crushed again obtaining a second-generation recycled aggregates. In the same way a third-generation recycled aggregate has been obtained and has been equally characterized. The results show that the compaction capacity of the aggregate is reduced after successive recycling. It has been possible to quantify how much the closed porosity of the recycled aggregate decreases with the number of times it is recycled. The loss of natural aggregate and increase of the volume of adhered mortar have also been evaluated using this technique.

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### 1. Introduction

The increasing production of construction and demolition waste (C&DW) and the ever-greater consumption of natural resources is forcing society to search for alternatives in order to reduce both. Fortunately, many studies have analysed the possibility of producing recycled aggregates (RA) using old concrete from C&DW [1–6], precast industries [7–10] and industry wastes [11–13]. However, the use of RA against the use of natural aggregate (NA) for structural concrete on material performance, environmental benefits and financial viability of the studies conducted so far do not fully demonstrate the choice of production of recycled aggregate concrete (RAC) with a significant advantage [14].

RA influences the physical and mechanical properties of RAC. The direct influence of the quality of RA on the durability is analysed in [7,15] showing that RA coming from precast-structural concretes is one of the most adequate in order to produce RAC. In terms of durability, the incorporation of recycled aggregate was responsible for worse results but did not compromise their use in structural concrete [16,17]. The properties of the interfacial transition zone (ITZ) have a significant impact on the macro mechanical properties of concrete [18]. X-ray computed axial tomography (CT) provides cross-sectional views of materials, components, and assemblies for non-destructive evaluation [19]. It can be used to examine concrete [20] and the high-resolution X-ray micro-CT allows modelling the permeability of cementitious materials [21]. On the one hand, the irregular surface of the old adhered mortar of the RA contributes to the improvement of the physical bond between the old and new cement matrix [22]. On the other hand, the lower mechanical resistance due to the adhered mortar

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contributes to reduce the compressive strength [15] and significantly so in the case of dynamic rather than static loading [3,23–25] but no significant influence of the recycled aggregate content on the durability performance of concrete exposed to aggressive environments is detected after years [26].

This paper presents a novel technique used to analyse the volume of adhered mortar to the recycled aggregate. A computerized microtomograph ( $\mu$ CT) device was used to evaluate the volume of the aggregate, the volume of natural aggregate and the volume of adhered mortar. To this end, a natural aggregate has been characterized, using the  $\mu$ CT, with which a source concrete has been produced. Subsequently, the source concrete has been crushed to obtain a first cycle recycled aggregate. After the characterization of the first-generation of recycled aggregate, a new source concrete has been made with it to be subsequently crushed again obtaining a second-generation recycled aggregates. In the same way, a third-generation recycled aggregate has been obtained and characterized. The effectiveness of using X-ray microtomography to access recycled concrete pore size and spatial distribution requires small samples in order to increase the resolution, which is still a drawback to overcome [22]. In the present case, representative samples of the recycled aggregates have been obtained and analysed [27].

## 2. Experimental program

Four aggregates divided in three fractions were studied. A natural limestone aggregate (NA) was used to produce a source concrete (SC). The SC was crushed to obtain a first-generation recycled aggregate (RA1). A second-generation recycled aggregate (RA2) was obtained from crushing the first recycled aggregate concrete (RAC1), made with the first-generation of recycled aggregate. In the same way, a third-generation recycled aggregate (RA3) has been obtained crushing the second recycled aggregate concrete (RAC2). RAC1 and RAC2 were produced using 100% recycled aggregate from the previous recycling cycle. In all of the cases, the cement, aggregates grading and target concrete characteristics (strength, slump and durability) were the same: compressive strength class C30/37; slump class S3 ( $125 \pm 15$  mm); exposure class XC3, limestone sand and Portland CEM I 42.5. The mix proportions, compressive strength and slump are presented in Table 1. Cement with low content of additions was used to avoid effects due to the increasing volume of adhered mortar. NA, RA1, RA2 and RA3 were divided in three coarse aggregate fractions in order to obtain the physical and mechanical properties of each one: 4–5.6, 5.6–8 and 8–11.2 mm, using three different techniques: classic methods, computerized micro-tomography ( $\mu$ CT) and scan electron microscopy (SEM).

### 2.1. Properties of the aggregate by classic methods

The densities and porosities were determined by displacement of water by immersion of the saturated samples and evaluation of

**Table 1**  
Mix proportions (by  $m^3$ ) and main properties of concrete.

Concrete	SC	RAC1	RAC2
Sand (0–2) (kg)	732	732	732
Coarse aggregate (4–8) (kg)	205	183	171
Coarse aggregate (8–16) (kg)	443	396	371
Coarse aggregate (16–22) (kg)	327	292	274
Cement (CEM I 42.5) (kg)	350	350	350
Water (kg)	194	194	194
Extra water (kg)	0	40	49
Effective w/c ratio	0.55	0.55	0.55
Apparent w/c ratio:	0.55	0.67	0.69
Slump (cm)	12.4	12.6	12.1
Compressive strength (MPa)	55.9	54.1	53.3

the saturated and dry weights of the samples. The methods used are described below. The water absorption capacity of the aggregates NA, RA1, RA2 and, RA3 was determined by evaluating the open pore volume saturated with water using vacuum. The relationship between the volume of accessible pores and sample volume, obtained by evaluating the difference between saturated and dry weights, provides the porosity of the material.

The real density (without pores) was determined by crushing each fraction of aggregate and concrete to powder smaller than 100  $\mu$ m using an automatic agate mill. The closed porosity was calculated by the difference between the relative volume and the real volume. The open porosity was calculated by the difference between the apparent volume and the relative volume.

24 h absorption of the aggregates was determined following standard EN 1097-6:2014. The determination of loose bulk density and voids was performed following standard EN 1097-3:2014. The Los Angeles wear test intends to measure the resistance to fragmentation of the aggregate through the loss of mass. The tests were carried out according to standard EN 1097:2014. All the tests following the EN 1097 standard were performed before obtaining the final mix proportions (Fauy). For this reason, the commercial natural aggregate and the aggregate resulting from crushing the SC and RC, not the 4–5.6, 5.6–8 and 8–11.2 mm fractions, were considered.

### 2.2. Computerized micro tomography

In order to analyse the properties of the samples, a computerized micro-tomograph was used. The equipment can provide qualitative and quantitative information of the tested specimens.

The  $\mu$ CT analysis consists, first, of a scanning phase in which an X-ray beam impinges on the sample at the same time as it rotates, obtaining a set of X-ray images that the computer will compose. A Skyscan I172  $\mu$ CT, similar to the one used by [28] in order to analyse concrete, with 80 kV and 100  $\mu$ A X-ray source, was used. With these parameters the resolution Voxel Size is 27  $\mu$ m. In the reconstruction, the linear absorption of each material was correlated with a shade of grey between 0 [black] and 255 [white] using the same parameters for all the specimens.

The image quantitative analysis software provides the volume of the different components, because the voxel intensity is proportional to the density of the material [22]. In this case it is possible to evaluate the volume of aggregate, the volume of natural aggregate, the volume of adhered mortar and the closed porosity of the sample. Regarding the qualitative analysis, this technique allows analysing how pores and aggregate are distributed. For the identification of the aggregate and cement paste phases, previous analyses were made using only, on the one hand, cement paste and, on the other hand, natural aggregate used in the research.

Each fraction of aggregate was introduced in a cylindrical container of 40 mm diameter. A sample of 25 mm height has been compacted for 20 s using a vibration table. After that, the sample was introduced into the  $\mu$ CT with the container. Each analysis, with the indicated parameters lasts approximately 10 h.

The properties of the coarse aggregate, obtained by  $\mu$ CT, were obtained as follows. The compaction index is obtained by dividing the volume occupied by the aggregate by the total analysed volume. The volume of aggregate (solid fraction) is obtained by adding the volume of the natural aggregate, the adhered mortar and the porosity. The total volume corresponds to the volume of a cylinder inscribed in the volume occupied by the sample. Therefore, the compaction index quantifies the volume of voids between aggregates. Closed porosity in % vol. is obtained by dividing the closed porosity volume, bigger than 27  $\mu$ m, by the aggregate volume. The NA volume is obtained by dividing the volume of NA by the total analysed volume. The adhered mortar is the

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