Mechanical and microstructural properties of recycled concretes mixed with ceramic recycled cement and secondary recycled aggregates. A viable option for future concrete

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Abstract: ~~The operations of renovation and demolition of buildings, warehouses, civil works, etc. generate vast amounts of residues known as construction and demolition wastes (CDW). Frequently, CDW contain an important quantity of ceramic materials (mainly, bricks and tiles, that are classified as Rb according to EN 12620 [1], or coded as 17 01 02 and 17 01 03 by the European List of Waste [2]), which could represent more than 50 % of the total in many countries of the European Union, especially in the Mediterranean areas such as Spain, France, Italy, Greece and Portugal.~~

~~In recent decades, c~~The concept of construction and demolition waste (CDW) ~~represents~~embodies a vast amount~~s~~ of residues, among which ~~characterized by presenting an important quantity of~~the ceramic materials (mainly, bricks and tiles) are an important part as, in many countries of the European Union, they~~, which~~ ~~could~~ represent more than 50 % of the total~~ in many countries of the European Union.~~ Therefore, the reutilization of this type of waste is one of the most important challenges faced by the construction sector within the circular economy paradigm. This research work deals with a dual reutilization of the CDW ceramic fraction~~ of CDW~~: firstly, as coarse recycled aggregates and, secondly, through the use of clay brick powder as a pozzolanic addition ~~in~~ to the cement~~ and, secondly, by means of the use of the CDW, which were comprised of more than 1/3 of ceramic materials, as coarse recycled aggregates~~. Thus, two different ~~types of~~ recycled concrete mixtures were produced: (1) with a 50 % partial substitution of the natural coarse aggregates (RC-RA) and (2) with a 50 % substitution of the natural coarse aggregates and a 25 % substitution of the ordinary Portland cement (RC-RAC). The mechanical performance (consistency, density and compressive strength) and microstructural properties such as porosity, elemental mapping analysis, hydration products and interfacial transition zones (ITZ) were assessed and compared with a control concrete (CC). It was revealed that the recycled concretes incorporating ceramic as secondary materials have a comparable performance level to the one exhibited by the conventional concrete at 28 days, ~~probably~~ in part due to ~~the lower effective w/c ratio and~~ their pozzolanic characteristics but also due to a lower effective w/c ratio~~of the resulting recycled concrete~~, which demonstrates their potential for reuse~~, and, hence, their~~and possible contribution to the~~a~~ circular economy.

Keywords: Recycled concrete; Construction and demolition waste (CDW); Recycled cement; Recycled aggregates; Ceramic

1. INTRODUCTION

Nowadays, the construction sector remains one of the main contributors to worldwide development. In the European Union (EU), the construction industry is responsible for around 9 % of the gross domestic product (GPD) and more than 18 million direct jobs in 3.~~,~~4 million companies [1].

~~By contrast~~Conversely, ~~the~~ construction ~~industry~~ is not an environmentally friendly activity. The materials employed ~~by this industry~~ provoke an intense demand for natural resources, which has given rise to an enormous environmental pressure. Among those, the so-called clay-based materials ~~(façades, roofs, partitions, floors, etc.)~~ are one of the most used (façades, roofs, partitions, floors, etc.). Albeit, ceramics are based on some of the most abundant raw materials in the Earth's crust, the pressure of their manufacture has ~~begins~~ begun to take its toll on our surroundings. According to the European Environment Agency [2], some European territories have already reported some initial shortage of natural aggregates (either sand or gravel), and the United Nations Environment Program [3] has also warned of a possible depletion ~~of these raw materials~~.

Furthermore, waste production is one of the most serious problems ~~facing~~ that the current society has to face, both in the developed world and developing countries. Contemporary models of linear economy based upon the use of goods and their subsequent disposal as waste are leading to an increasing accumulation of wastage in landfills, which is a~~n~~ concerning unsustainable attitude ~~that could lead humanity to a, at least, uncertain future~~. In this regard, the relevance of the residues generated by the construction sector should also be recognized. ~~as c~~Construction and demolition wastes (CDW) are the greatest flow of waste generated in many countries of the EU [4], with the ceramic fraction being of significant importance in the Mediterranean zones [5].

The reuse and recycling techniques developed under the umbrella of the Circular Economy, which emphasizes the need to maintain the value of products and materials for as long as possible within the economy through their repeated use after the end of their life is reached to create further value [6], seems a promising solution for both problems. Therefore, the negative impacts ~~provoked~~triggered by the construction sector could be partially alleviated by considering the reuse of their own wastes as secondary materials through the reincorporation as inputs in the manufacture of new-use materials. ~~In this regard, construction ceramic wastes have been suggested as a possible substitute of the natural aggregates in mortar and concretes,~~

~~as well as a pozzolanic addition in cements [7–9], due to their intrinsic properties of “fired-clay” [7,10–15].~~

The emergence of the use of recycled materials in the construction industry, specifically in the production of ~~concrete~~cement-based materials, has met with varying degrees of success due to the barriers that the market for recycled products has still to overcome. Numerous studies have investigated the possibilities of using recycled aggregates from CDW in concrete mixes as total or partial replacement of the natural coarse aggregate in order to prove the feasibility of this practice. Hence, research ~~works~~reports focusing~~ed~~ on the influence that the incorporation of mixed recycled aggregates [7–13] ~~as well as~~and ceramic aggregates [5,14–21] has o~~i~~n the concrete production are somehow common in the literature. Although there is no clear consensus, when the incorporation of recycled aggregates is controlled, i.e. in terms of impurities and ~~at the~~limitation of replacement level, a moderately lower (6-27%) [9–11,22,23] or comparable [14,24] concrete strength performance to the reference mix could be reached~~, which is mainly adequate for non-structural applications~~. Nonetheless, mixed and ceramic recycled aggregates are still considered with suspicion as a reliable source of secondary aggregates for the concrete manufacture.

As the use of supplementary cementitious materials increased, ceramic wastes such as fired-clay [25–27], bricks [28–36], tiles [37–42], sanitary ware waste [43] and CDW [44] have also been suggested as a possible pozzolanic addition in cement. The influence of the ceramic waste addition on ~~the mortar and concrete~~ the performance of mortar and concrete has been reported both as beneficial [28,29,32,45] and disadvantageous [28,30,31,46]. Nonetheless, it also has been stated that the latter effect could be alleviated by establishing a substitution limit ranging between 15% and 30 % [26,32–35,47,48].

However, there are not many studies [49,50], which have considered ~~using~~the combined use of ceramic secondary materials ~~as both~~and recycled aggregates and recycled cement in the concrete manufacture.

Since the quality of the concrete mixture depends upon the properties of the particular raw materials used in its manufacture, the suitability of the recycled mixed ceramic aggregates and the recycled cement was assessed through the performance evaluation of the recycled concrete. Thus, ~~T~~this research work analyzes and compares the mechanical and microstructural characteristics of three types of concrete mixed in the laboratory to reduce the uncertainty linked to a future field application:

1. Conventional concrete ~~(CC),~~ mixed with natural siliceous aggregates and commercially-available blast furnace slag~~Portland~~ cement (CC).

2. Recycled concrete made with a ~~partial~~ 50 % replacement ~~of 50 %~~ of the coarse natural aggregates by recycled mixed ceramic aggregates, fine natural aggregates of siliceous nature and commercially-available blast furnace slag~~Portland~~ cement (RC-RA).

3. Recycled concrete made with~~h~~ a 50 %~~partial~~ replacement ~~of 50 %~~ of the coarse natural aggregates with recycled mixed ceramic aggregates, fine natural aggregates of siliceous nature and recycled cement comprised of 75 % ~~of~~ commercially-available Portland cement and 25 % ceramic powder addition (RC-RAC).

2. MATERIALS

The main characteristics and properties of the materials used in this study are presented in this section. Special attention was paid to the recycled ceramic cement and recycled mixed ceramic aggregates. Moreover, the three different mix designs employed are also described in this section.~~: conventional concrete (CC), recycled concrete with coarse recycled mixed ceramic~~

~~aggregates (RC-CA) and recycled concrete with coarse recycled mixed ceramic aggregates and ceramic cement (RC-RAC)~~

2.1. BLAST FURNACE SLAG CEMENT

Blast furnace slag cement, namely CEM III/A 42.5 N/SR, was used for this study. The table 1 shows its chemical composition and the compliance limits as stated in the European standard EN 197-1 [51]

Table 1. Blast furnace slag cement chemical composition

Chemical composition	Value (wt%)	Limit (wt%)
Clinker (SiO_2, Fe_2O_3, Al_2O_3, CaO, MgO and SO_3)	54	35-64
Blast-furnace slag	41	36-65
Minor components	5	≤5
Loss on ignition	1.5	≤5

This type of conventional cement was selected according to the recommendations of Mas et al. [13]. Their research showed that sulfate-resistant CEM III/A was ~~specially appropriate~~especially appropriate for recycled concrete mixtures since lower strength declines were obtained compared to the control concrete. Moreover, ~~it was~~the authors also praised this cement type due to its sulphate resistance ~~to sulfate~~ as recycled aggregates ~~can~~ could contain a significant quantity of gypsum.

2.2. CERAMIC PORTLAND CEMENT

The binder employed in this research work was an eco-efficient cement manufactured by researchers form the Eduardo Torroja Institute for Construction Sciences~~[1–4]~~. Encouraged by the pozzolanic nature of clay-based materials, the authors [43,44,52,53] assessed CDW as an alternative addition in blended cements. ~~and~~ They ~~demonstrated the feasibility of using clay brick powder from CDW as a pozzolan addition, providing with~~ carried out a complete ~~suitable~~ characterization of the ~~ceramic-based~~clay brick po~~d~~wder from CDW ~~for its use as Pozzolan in~~

~~Cements~~as a pozzolanic addition [44,52], as well as an evaluation on~~testing~~ its sulfate resistance [54] and ~~analyzing its~~ calorimetric behavior during cement hydration [53,55].

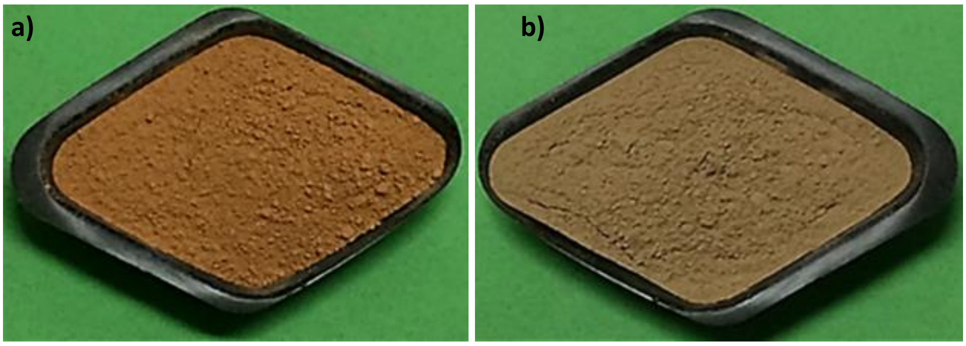


Figure 1: a) Clay brick powder from CDW. b) Eco-efficient~~e~~ blended cement containing~~-~~75% OPC and 25% clay brick powder from CDW. [52].

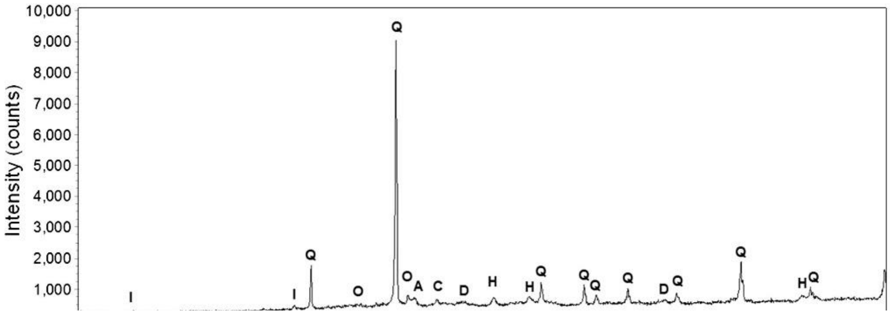
The CDW, which was collected from a Spanish recycling plant situated in Castile and Leon, was 100% comprised of fired clay materials. Prior to its use, the residue was pre-conditioned (Figure 1.a) ~~by~~ through drying at 105 ºC to constant mass, grinding ~~by means of~~ with a jaw-crusher and a ring-mill and, finally, sieving (< 63 µm).~~,~~ ~~t~~Then~~,~~ the clay brick powder and the OPC where mixed together by means of a ~~turbula~~tubular dry-powder mixer machine. Table 2 shows the characterization of the treated clay brick powder used as a pozzolanic addition in the eco-efficient cement. It is worth mentioning the high reactive silica content and the capacity of lime fixation, which corresponds with its chemical composition as shown by the X-ray fluorescence (XRF) results (Table 3). As expected, silicon oxide (SiO_2) was the major component followed by aluminum oxide (Al_2O_3) and iron oxide (Fe_2O_3). Moreover, the X-ray diffraction (XRD) results showed that the clay brick powder mostly consisted of quartz, feldspars (orthoclase and anorthite), illite, calcite, dolomite and hematite (Figure 2). As presented in Figure ~~(~~3~~)~~, the scanning electron microscope (SEM) image ~~shows~~ showed large quartz crystals surrounded ~~in~~ by a matrix with smaller particle size crystals.~~,~~ In addition, ~~whereas~~ the analysis carried out by means of X-ray dispersive energy (EDX) detected the presence of elements such as aluminum and potassium, which is in line with the feldspars detected by XRD. Iron, calcium, magnesium and sodium were also present in the matrix.

Table 2: Characterization of the clay brick powder from CDW [52,53]

	Clay brick powder from CDW
Ceramic content (%)	100
Density (g/cm^3)	2.540
Specific surface (cm^2/g)	5737
Particle size range (µm)	0.9-100
Average particle size (µm)	61-73
Reactive SiO_2 (%)	35.10
$CaCO_3$ (%)	1.66
1-day lime fixed (%)	15
28-days lime fixed (%)	81
360-days lime fixed (%)	97

Table 3: Chemical composition of the clay brick powder employed to manufacture the eco-efficient cement - LOI: Loss on ignition- [52,54].

Oxides (wt%)	SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	SO_3	K_2O	TiO_2	P_2O_5	LOI
Clay brick powder	59.63	18.51	5.92	0.09	3.12	4.78	0.73	0.42	3.59	0.84	0.15	2.15



Compound	Chemical formula	ICSD code
Illite	$K(Al_4\ Si_2O_9(OH)_3)$	090144
Quartz	SiO_2	067121
Orthoclase	KSi_3AlO_8	010270
Anorthite	$CaAl_2Si_2O_8$	202710
Calcite	$CaCO_3$	040112
Dolomite	$CaMg(CO_3)_2$	202162
Hematite	Fe_2O_3	082904

Figure 2: XRD patterns for the clay brick powder from CDW - I: illite, Q: quartz, O: orthoclase, A: anorthite, C: calcite, D: dolomite, H: hematite - [54]

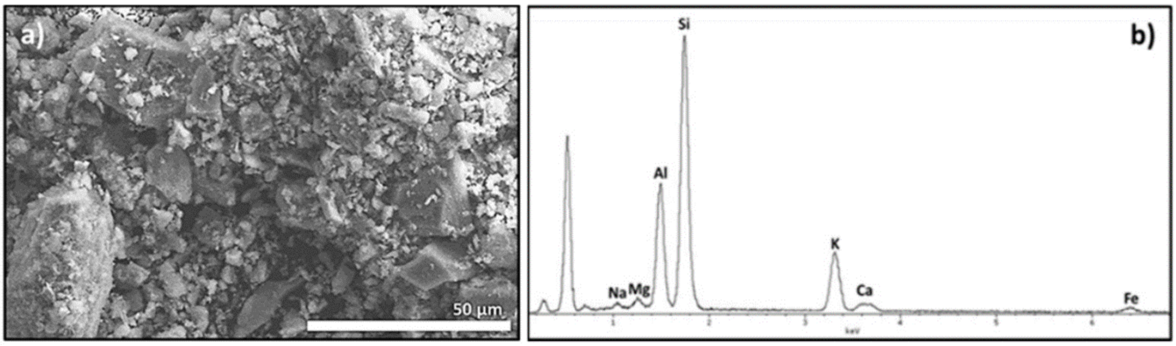


Figure 3: a) SEM image of clay brick powder ~~brick~~ from CDW. b) EDX compositional analysis. [52].

The eco-efficient cement used was factory-made as a mixture of 75 % ~~ordinary Portland cement (~~OPC) and 25 % clay brick powder from CDW (Figure 1.b). The chemical composition of both the OPC (CEM I 42.5R) and the resulting eco-efficient cement are shown in Table 4. Moreover, Figure 4 illustrates the mineralogy of the recycled cement through the XRD patterns detected ~~for the eco-efficient cement~~. As expected by the chemical and mineralogical composition of the clay brick powder incorporated, the eco-efficient cement presented greater values of silicon oxide (SiO_2), aluminum oxide (Al_2O_3), iron oxide (Fe_2O_3), sodium oxide (NaO_2) and potassium oxide (K_2O) than the reference cement.

Albeit the use of certain materials as additions to cement is allowed by ~~the~~ EN 197-1 [51], the European standard also establishes the need to fulfil some mechanical, chemical and physical requirements. The values of the compressive and flexural strength, SO_3 and Cl^- content, initial and final setting time and soundness for both the OPC and the eco-efficient blended cement are shown in Table 5. The characterization proved the compliance of the eco-efficient cement with the European standard. ~~and~~and the results stated the minor impact that the use of clay brick powder from CDW had on the recycled cement performance compared to the OPC. Among the differences detected, ~~both~~ the 28 days values of the flexural and compressive strength exhibited by the eco-efficient cement were~~ a~~ 11% and 10% lower than the CEM I 42.5R, respectively. However, a beneficial decrease in the SO_3 and Cl^- content (22% and 20%, respectively) were also noticed. Finally, no alteration was observed in the initial setting time;~~,~~ whereas, the final setting time of eco-efficient cement was recorded 12 minutes ahead of the OPC.

Table 4: XFR Chemical composition of the cement employed -LOI: Loss on ignition

(wt%)	CEM I 42.5 R	Eco-efficient cement
CaO	63.21	47.04
SiO_2	18.83	29.35
Al_2O_3	4.36	7.32
SO_3	3.13	2.45
Fe_2O_3	2.55	3.06
MgO	1.85	2.24
K_2O	0.82	1.7
TiO_2	0.22	0.31
P_2O_5	0.18	0.17
Na_2O	0.16	0.41
SrO	0.1	0.08
Mn_2O_3	0.09	0.09
Cl^-	0.05	0.04
ZnO	0.04	0.03
Cr_2O_3	0.01	0.01
LOI	3.11	2.66

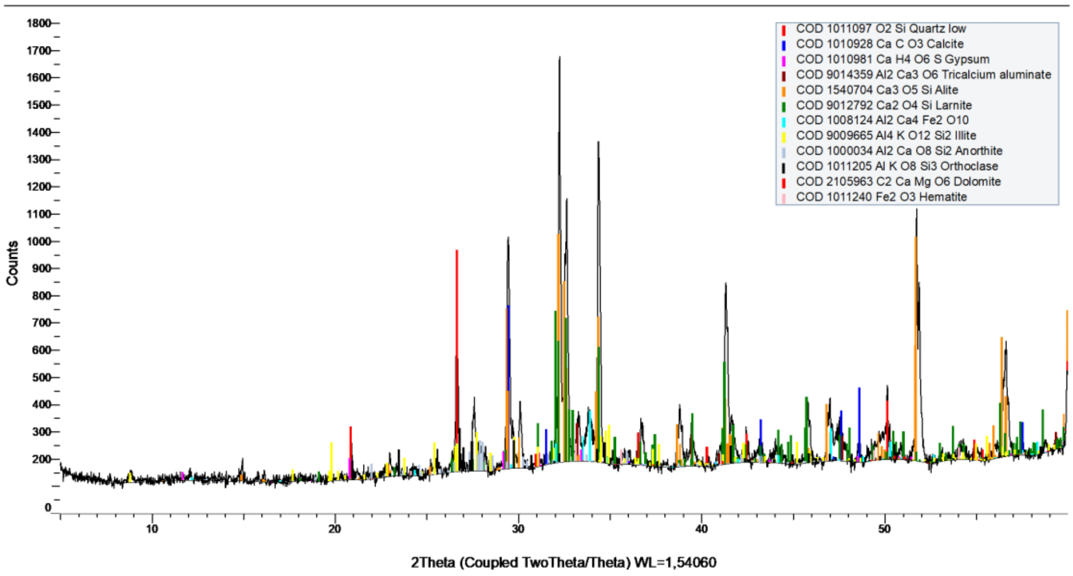


Figure 4: XRD patterns for the eco-efficient cement

Table 5: Mechanical, chemical and physical characterization of cements

	CEM I 42.5R	Eco-efficient blended cement	EN 197-1 [51] requirements	
2-days compressive strength (MPa)	30	22	≥ 20	EN 196-1 [56]
7-days compressive strength (MPa)	56.8	46	-	
28-days compressive strength (MPa)	63	56	42.5-62.5	
2-days flexural strength (MPa)	7.87	7	-	
7-days flexural strength (MPa)	10	8	-	
28-days flexural strength (MPa)	10	9	-	
LOI (%)	3.11	2.66	≤ 5	EN 196-2 [57]
SO_3 (%)	3.13	2.45	≤ 4.0	
Cl^- (%)	0.05	0.04	≤ 0.1	
Initial setting time (minutes)	165	165	≥ 60	EN 196-3 [58]
Final setting time (minutes)	263	251	-	
Soundness (mm)	1	1	≤ 10	

2.3. NATURAL AGGREGATES

Natural aggregates, both fine and coarse, had a siliceous nature and complied with the standard EN 12620:2003+A1 [59], which states the properties of the different aggregates to be used for the concrete manufacture. More specifically, the natural aggregates employed in this research work were crushed sand (0-4 mm), rounded sand (0-5 mm), and two different types of gravel (6-16 mm and 4-10 mm.)

2.4. RECYCLED GRAVEL

~~The recycled gravel used in this study was classified as recycled mixed ceramic aggregate due to its ceramic content (>30%).~~ The recycled aggregates showed a typical composition for CDW produced in the Mediterranean area, where clay-based materials are commonly used for façades, walls, roofs, etc. [5]. Hence, the recycled gravel was classified as recycled mixed ceramic aggregate due to the amount of ceramic present (>30%). The composition of non-floating components of the recycled aggregate is shown in the following table (Table 6)~~, this composition was obtained~~ according to EN 933-11 [56]. According to tho~~e~~se results, the recycled aggregate ~~i~~was classified as $R\textit{cu}_{50}$ (Rc+Ru=61.62% and $R\textit{b}_{50}$ (Rb=33.56%) [59].

Table 6: Non-floating components of the recycled aggregate

Component	~~% wt (~~Average value (wt%)
Concrete and mortar (Rc)	44.11
Ceramics (bricks, tiles,…) (Rb)	33.56
Unbound aggregates (natural aggregates without cement or mortar attached) (Ru)	17.51
Asphalt (Ra)	0.44
Glass (Rg)	0.75
Gypsum, Wood, metals, plastic and other impurities (X)	3.64

~~According to these results, the recycled aggregate is classified as $R\textit{cu}_{50}$ (Rc+Ru=61.62% and $R\textit{b}_{50}$ (Rb=33.56%) [57].~~

The recycled coarse aggregates or secondary gravel was obtained through a mechanical processing (classification, crushing and sieving) ~~of CDW in~~at the TEC-REC: Tecnología y Reciclado S.L. manufacturing plant located in the province of Madrid (Spain). Table 7 shows a summary of the average physical and mechanical properties of the recycled coarse aggregates, which have been studied according to EN 12620+A1 [59].

Table 7. Physical and mechanical properties of the recycled coarse aggregates

Properties	Test result	Limit value	Standard
Maximum particle size (mm)	16	-	EN 933-1 [60]
Minimum particle size (mm)	4	4	EN 933-1 [60]
D/d ratio	5.0	≥ 1.4	EN 933-1 [60]
Granulometric modulus	7.67	-	UNE 146406 [61]
Content of particles < 4 mm (%)	5	5	EHE-08 [62]
Undersize particle content (%) Sieve d	5	< 10	UNE 146121 [63]; EN 933-1 [60]
Oversize particle content (%) Sieve 2D	0	0	UNE 146121[63]; EN 933-1 [60]
Oversize particle content (%) Sieve D	2.21	< 10	UNE 146121 [63]; EN 933-1 [60]
Fines content (%)	0.04	≤ 1	UNE 146121 [63]; EN 933-1 [60]
Apparent density (Mg/m^3)	2.53	-	EN 1097-6 [64]
Oven-dried density (Mg/m^3)	2.08	-	EN 1097-6 [64]
Saturated surface dry density (Mg/m^3)	2.26	-	EN 1097-6 [64]
Water absorption (%)	8.53	≤ 7	EN 1097-6 [64]
Flakiness index (%)	14.75	≤ 35	EN 933-3 [65]
Los Angeles coefficient (%)	40.99	≤ 40-50(1)	EN 1097-2 [66]

(1) The current Spanish legislation accepts the use of coarse aggregates with a fragmentation resistance ranging from 40 to 50 in the Los Angeles test for the manufacture of mass or reinforced concrete not exceeding 30 N/mm^2, when previous experiences or studies exist that support its use without prejudice to the performance of concrete.

2.5. WATER

Tap water was used, complying with the EHE-08 [62] recommendations.

2.6. ADDITIVES

No additives (plasticizers or others) were used to obtain the different mixes.

2.7. DOSAGE

In this research work, the De la Peña method was employed for the proportioning of both the conventional and recycled concrete mixtures. Although not too well known at international level, some research papers [16,17] have previously referenced this technique as it is the most employed method in Spain since its development in 1955. The proportioning design was based on the attainment of a target 28 days characteristic compressive strength of 25 MPa. Moreover, to design the recycled mixtures, a constant water content was maintained, and the recycled aggregate substitution was made according to the direct weight replacement approach, which is commonly used [67,68]. In addition, the limits prescribed by EHE-08 [62] to achieve a durable concrete, i.e. a minimum cement content of 300 kg/m^3 and a maximum water/cement ratio of 0.55, were taken into account in the proportioning of all mixes.

Table 8 shows the detailed proportion of the different raw components used in the manufacture of the concrete mixtures.

TABLE 8: Mix proportions per cubic meter

Mix proportions per cubic meter	CC	RC-RA	RC-RAC
Real w~~W~~ater (l~~kg~~)	155.21	155.21	155.21
Effective water (l)	155.21	128.13	128.13
CEM III/A 42,5 N/SR (kg)	312.50	312.50	0
Recycled cement CEM-ceramic (kg)	0	0	312.50
Sand 0/4 mm (kg)	96.98	96.98	96.98
Sand 0/5 mm (kg)	441.81	441.81	441.81
Gravel 4/10 mm (kg)	484.92	242.46	242.46
Gravel 6/16 mm (kg)	161.64	80.82	80.82
Recycled mixed ceramic aggregate 4/16 mm (kg)	0	323.28	323.28
Real w/c ratio	0.50	0.50	0.50
Effective w/c ratio	0.5	0.41	0.41

Real W/C ratio and effective W/C ratio were included in the dosage table due to the importance of both parameters on the subsequent recycled concrete properties as the amount of water absorbed by recycled aggregates and, therefore, unavailable for the cement hydration should be considered.

3. METHODS

3.1. CONSISTENCY

The consistency, which could be employed as an indirect measurement of the workability of the conventional and recycled concrete mixtures, was determined by means of two common methods: the slump-test [69] and the Vebe test [70].

3.2. DENSITY OF HARDENED CONCRETE

Hardened density of concrete was determined according to EN 12390-7 [71]. After 28 days of curing immersed in water (20±2 ºC), four specimens of each mixture were tested. Firstly, the surfaces were wiped using a damp cloth to remove any water excess, and then, each specimen was weighed. Afterwards, the volume of each specimen was determined by actual measurements with a caliper in accordance with EN 12390-1 [72]. Finally, by using the determined values, i.e. the saturated mass of the specimen divided by its volume, the hardened density of concrete in water saturation condition was determined.

3.3. COMPRESSIVE STRENGTH

The compressive strength tests were done by means of a hydraulic press conforming to EN 12390-4 [73] and EN 12390-3 [74]. The tests were implemented at different ages, 7, 21 and 28 days, by using cylindrical specimens of 150 x 300 mm^2, according to EN 12390-1 [75].

3.4. POROSITY

In order to determine the 28 day porosity and pore size distribution of the concrete samples, several tests were conducted using a Mercury Intrusion Porosimetry (MIP) technique. A Micromeritics AutoPore IV 9500 porosimeter was employed to examine a pressure range of 0.0034-227.5270 MPa and a pore diameter range from 0.006 μm to 175 μm. Previously to the test, cylindrical samples, cut from the centers of the specimens (~~of~~with a diameter of 20 mm and a height of 20 mm ~~approximately 1 cm^3~~) were dried at 40°C to constant weight and degassed for 30 minutes with a vacuum pump to ensure moisture removal.

3.5. MICROSTRUCTURE

Two types of microstructural tests were carried out in this study by means of a Hitachi S-4800 scanning electron microscope (SEM) with tungsten as X-ray source, a Si/Li detector and a Brucker XFlash 5030 EDS analyzer and SEM images and EDX mappings were obtained. The samples were prepared with a bi-adhesive graphite film and a carbon coating to ensure conductivity and avoid signal masking, all the samples were sited in a metallic holder to facilitate its placement in the microscope.

4. RESULTS AND DISCUSSION

4.1. CONSISTENCY

Table 9: Consistency of the different concrete mixes.

	Slump-test (cm)	Vebe-test (s)
CC	3.2	10 symmetrical
RC-RA	2.3	9 symmetrical
RC-RAC	2.3	9 symmetrical

Notwithstanding the observed variations, it could be established that the use of a partial replacement of the natural aggregates or the OPC by recycled ceramic materials did not negatively affect the consistency values of the concrete mixes employed in this research work. Furthermore, despite the lower consistency values, no problems in workability were detected when placing, compacting or casting the test specimens. Nonetheless, it is well recognized that the use of plasticizers or superplasticizers improves the workability of a concrete mixture. For instance, Cantero et al. [50], who employed a modified water-based polycarboxylate superplasticizer, reported fluid consistencies for all conventional and recycled mixtures. However, the authors also stated that no significant modifications in the slump values occurred for the concrete incorporating the recycled aggregate ~~replacement~~ nor the recycled powder brick ~~substitution~~ addition when compared to the conventional concrete.

4.2. DENSITY OF HARDENED CONCRETE

The density results of the saturated hardened concrete are given in Table 10. As expected, the conventional concrete presented the highest density value, 2379 kg/m^3, which is in accordance with the standard density of a conventional concrete, between 2300 and 2400 kg/m^3 [62].

Table 10: Density of the hardened concrete mixtures

	Hardened density (kg/m^3)
CC	2379 ± 26.2
RC-RA	2295 ± 28.7
RC-RAC	2290 ± 42.1

Regarding the hardened density of the recycled mixtures, the average values were 2295 kg/m^3 for RC-RA and 2290 kg/m^3 for RC-RAC. It is well recognized that the density losses ~~exhibited by recycled concrete mixtures answers to~~are a result of the replacement of the natural aggregates by the recycled aggregates [7–10,13,23,76]. Due to the presence of adhered mortar, clay-based particles, impurities and floating materials (Table 7) in the recycled aggregates, a decrease of 3.55 % was noticed for RC-RA. Similar values of density declines have been reported ~~for ~~in the literature when a 50 % substitution level was applied: 2.11 % [10,23], 1.10 and 2.36 % [11], 3.91 % [7], 4.18 % [76]. On the other hand, the density of the RC-RAC mixture exhibited a decrease of 3.75 %, which is also in line with the results found in the literature, as Cantero et al. [50] observed a 4.47% density reduction for a concrete with a 50% replacement of coarse aggregates and ceramic cement additions~~-~~. Besides the effect of the recycled aggregates, ~~this further~~the additional decrease reveals the influence of the eco-efficient cement employed. Whereas the OPC density could be taken as 3.15 g/cm^3 [77], the incorporation of the clay brick powder from CDW (with a density of 2.54 g/cm^3 as shown in Table 2) would inevitablely result in a eco-efficient cement with a lower density. This would~~, which~~ cause~~d~~ ~~that RC-RAC displayed ~~a slightly lower density ~~due to a slightly~~of the hardened cement paste RC-RAC compared to both CC and RC-RA, even though both recycled mixtures were proportioned ~~for~~with the same W/C ratio.

4.3. COMPRESSIVE STRENGTH

As it could be observed in Table 11, the conventional and recycled mixtures exhibited 28 ~~-~~day compressive strength figures over 25 MPa, which complied with the target strength of the proportioning method. Hence, ~~and,~~ according to the Spanish standard EHE-08, ~~is the~~the recycled mixtures also conformed to minimum compressive strength value for structural purposes. ~~Equally~~In a similar manner, the current standard EN-1992-1-1 [78] recommends a minimum value of average compressive strength (fcm) of 20 MPa (tested in cylindrical samples), which was satisfied.

Table 11: Compressive strength of the samples (MPa)

Mixture	Compressive strength (N/mm^2)		
	7 days	21 days	28 days
CC	24.3 ± 0.2~~3~~	30.4 ± 0.3	35.0 ± 0.7
RC-RA	14.9 ± 0.9	26.3 ± 0.3	35.7 ± 3.6
RC-RAC	19.7 ± 1.0	26.9 ± 0.6	37.1 ± 1.4

The presence of adhered mortar, ceramic materials, as well as some other impurities such as wood, plastic, gypsum… in the recycled aggregates reduced the compressive strength performance of all recycled mixtures compared to the conventional concrete at early ages (7 and 21 days). Both recycled concrete mixtures exhibited greater decreases at the 7 day age (37.5 % and 16.7 % for RC-RA and RC-RAC, respectively), which eventually became less significant as the compressive strength developed due to cement hydration, which suggested that the recycled mixtures presented a higher strength development with age. This finding is in agreement with other researchers [8,9,11,79], who found that the differences between conventional and recycled mixtures incorporating recycled mixed aggregates were lower at ~~longer~~later~~-~~ ages. The effect could be explained by the presence of unhydrated cement particles and brick dust with pozzolanic activity within the recycled aggregates that contributed to the resistance gain. In any case, it is worth mentioning that, for the 7 and 21~~-~~ day compressive tests, the reduction experienced by the RC-RAC mixture was always lower than that of the RC-RA compared to the CC, which may be attributed to the positive effect of the clay brick cement replacement. In this regard, the results obtained by Naceri and Hamina [29], Kartini et al. [28] and Shao et al. [45] also pointed to a beneficial effect of the brick powder on the compressive strength of mortar but ~~pointed~~ mainly referring to a long-term effect (sometimes, beyond 60 to 90 days). Conversely, Heidari and Hasanpour [30] and Ge et al. [31] reported that the ceramic addition had a negative effect on the concrete strength, which was more significant up to the 28 days mark.

In this research work, a clear shift in the mechanical performance of the recycled concretes was observed at 28 days. Both the pozzolanic activity of the ceramic powder addition ~~as well as~~and the lower effective w/c ratio of the recycled mixtures resulted in recycled mixtures exhibiting similar, or even better, compressive strength levels compared to the conventional concrete. For the RC-RA mixture, a compressive strength increase of 2.9 % was observed compared to CC at 28 ~~-~~days. Moreover, the addition of the clay brick powder led to a further improvement, and the RC-RAC mixture showed a 5.7 % strength increase compared to CC at 28 ~~-~~days.

Firstly, the mechanical improvement exhibited by the RC-RA mixture could be attributed to the enhancement of the ITZ due to the lower effective w/c ratio, the internal curing caused by the recycled aggregates as well as the bonding effect produced by the rough and porous surface of the recycled materials. Etxeberria et al. [24] reported comparable compressive strength values between conventional and recycled concretes for replacement levels of recycled mixed aggregates up to 25 %. Similarly, Cachim [14], who replaced 50 % of the natural coarse aggregates by recycled ceramic aggregates, observed a slight gain in compressive strength for recycled concretes w/c ratios of 0.45 which were subjected to water absorption compensation.

Nevertheless, losses of compressive strength have also been extensively reported in the literature when recycled mixed aggregates are incorporated into ~~the~~ recycled concrete. For a 50 % substitution of the natural coarse aggregates such as the one described in this research work, some authors have stated declines in the 28 ~~-~~days compressive strength of 6.34 % and 7.31 %, depending on the ceramic content -34 % and 68 %, respectively - [11], 10 % [50], 12.3 % [22], 18.44 % [10,23], and 26.79 % [9] compared to their respective control concrete mixtures. Moreover, increases o~~n~~f the replacement ratios have been linked to larger declines in the performance of the recycled mixtures. For instance, complete substitutions of the natural coarse aggregates have~~d~~ resulted in greater compressive strength drops: 26.55 % [22] and 41.53 % [9]. Secondly, it is also necessary to contemplate the greater strength increase achieved by the incorporation of the brick powder addition into the recycled cement. The observed improvement may be attributed to the pozzolanic action of the selected addition and it has also been reported by other authors who studied the influence of the brick powder cement replacement in both mortar and concrete specimens. Up to 7 % strength gain was observed by Kirgiz [80] for a 20 % replacement. Naceri and Hamina [29] and Shao et al. [45] concluded that an increased mechanical performance of mortar could be expected up to a 10 % and 20 % replacement level, respectively, but only at long term (60 to 90 days). Albeit Olofinnade et al. [32] registered up to a 9 % strength increase of concrete at 10 % replacement, the authors also noted that the behavior was reversed when the powder replacement reached a certain value (20 %). A similar trend was observed by several researchers and different substitution values have been stated as a limit prior to a significant compressive strength decline: 15 % [33], 20 % [26,34], 25 % [35,47] and 30 % [48]. Nevertheless, it is worth mentioning that, in a later study, Ge et al. [31] observed lower compressive strength in concrete mixtures incorporating 10-30 % brick dust replacements. The same findings were reported by Kartini et al. [28], who observed a 4 %, 8 % and 15 % decline in the compressive strength for 10 %, 20 % and 30 % ground clay brick replacements, respectively. Similarly, Aliabdo et al. [46] observed a 8.3 %, 14.0 %, 18.7 %, 14.2 % and 25.2 % decrease in the compressive strength of mortar for 5 %, 10 %, 15 %, 20 % and 25 % substitution levels, respectively. Interestingly, ~~the contrary results~~increases in strength were reported when the brick powder was incorporated as an additive to cement instead of a partial replacement of cement.

Nevertheless, the aforementioned 5.7 % compressive strength gain exhibited by the RA-RAC mixture should be attributed to the effect of both the recycled aggregates and the cement addition. In this regard, the obtained result is in line with that of Letelier et al. [49], who stated that concrete mixtures incorporating a 30 % substitution of recycled aggregate and a 5 % waste brick powder as cement replacement showed a 9 % strength increase compared to the control concrete. However, Letelier et al. [49] established a 5 % ceramic replacement as the limit value to the positive effect of the addition on the compressive strength of recycled concrete.

Contrarily to the results shown~~ed~~ in this research work, Cantero et al. [50] found a negative effect of the brick powder cement substitution in recycled concrete mixes. The authors reported a greater compressive strength reduction when both recycled aggregates and brick powder were incorporated to concrete (25 % and 23 % for 25 % and 50 % replacements at 28 days) compared to the one exhibited by the recycled concrete mixes with recycled aggregates (3 % and 10 % for 25 % and 50 % replacements at 28 days).

4.4. POROSITY

The effect of the ceramic replacement on the porosity is represented in Figure 5. The graph illustrates the cumulative volumes of mercury intruded, during both the intrusion and extrusion phases, as a function of the pore size diameter for the CC, RC-RA and RC-RAC samples.

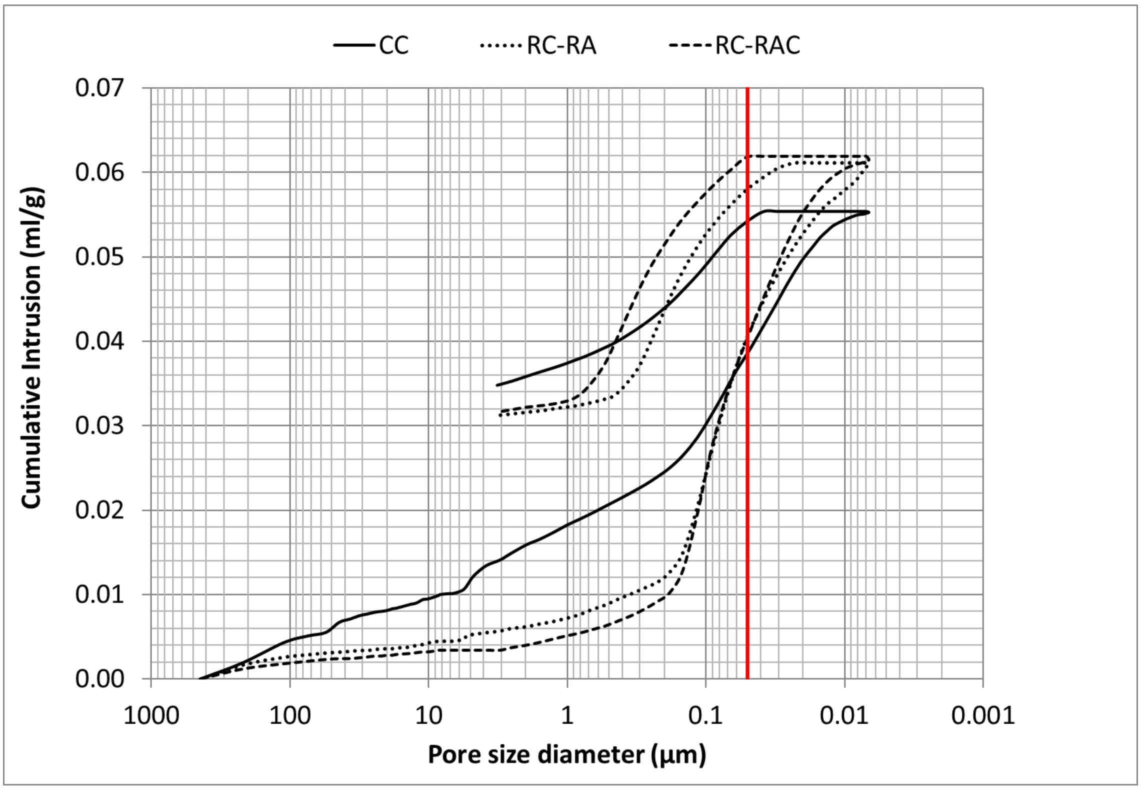


Figure 5: Cumulative intrusion *vs* pore size distribution of CC, RC-RA and RC-RAC samples at 28 days.

The total porosity obtained through the MIP test was slightly higher for the recycled mixtures (12.44 % for RC-RA and 13.08 % for RC-RAC) than the conventional concrete (12.37 %). Nevertheless, the study of the pore size distribution allowed to lessen the concerns about the negative effects of the ceramic incorporation ~~in~~ on the concrete durability. Firstly, the incorporation of the recycled aggregates resulted in a pore size distribution change compared to the control mixture. The volumes of mercury intruded during the intrusion phase for RC-RA and RC-RAC samples were significantly smaller than that of the CC for pores greater than 0.07 µm. Greater differences were even noticeable for pore sizes greater than 0.1 µm, especially for the RC-RAC sample, which showed the lower cumulative intrusion value and, thus, attested to the positive effect of ceramic cement addition on concrete porosity. Therefore, the higher porosity of the recycled mixtures could be attributed to the greater presence of pores smaller than 0.07 µm. In this regard, several authors [81,82] have remarked that the volume of pores with diameter lower than 0.1 µm ~~produces~~ plays a minor role in the water absorption of cement-based materials.

Similar results have been reported by Penteado et al. [38], who replaced 5 % to 30 % of cement by ceramic tile waste. The authors observed a minor tendency to greater porosities as the percentage of replacement increased. Kulovaná et al. [36] showed that concretes containing up to 20 % ceramic powder in the blended cement had similar open porosity values. Nevertheless, the open porosity rose up to 15 % and 23 % for 40% and 60 % replacements, respectively.

Concerning permeable pores, several authors have also reported the positive effects of the cement replacement by a ceramic fraction on the performance of cement-based materials. Kannan et al. [83] noted that the inclusion of ceramic waste powder on high performance concrete resulted in a reduction of the permeable pores between 3 % and 24 % at 90 days compared with the conventional concrete mixture. It is worth mentioning that the authors observed the greater decreases of the permeable pore volume for concretes with a 20 % ceramic powder addition; whereas mixtures with 40 % of cement waste powder resulted in lower reductions. On the contrary, later on, El-Dieb and Kanaan et al. [42] found that as the replacement level of ceramic waste powder increased, the permeable pores percentage showed an overall reduction compared to the control mixtures. The authors attributed this reduction to the physical microfilling effect of the ceramic powder, which altered the pore structure and reduced the volume of permeable pores by improving the packing of particles, ~~which was~~ especially ~~noticeable~~ at the aggregate-paste interfacial zone. Asensio de Lucas et al. [54], who manufactured recycled concrete incorporating a pozzolanic addition from clay-based CDW, concluded that the ceramic addition ~~originated~~ resulted in ~~in~~ a pore structure refinement that provided a better anti-corrosion performance of concrete exposed to sulfate attack.

Furthermore, the positive effects of the use of recycled ceramic aggregates on the concrete manufacture have already been reported in the literature ~~by other researchers~~. Poon and Chan

[84] and Xiao et al. [85] claimed that the finer clay brick aggregates particles were responsible for a more comprehensive filling of the voids and, thus, reduced the porosity of recycled concretes. Rodríguez et al. [12], who produced concrete with 25% of recycled mixed aggregates (RMA), ~~and~~ observed that the recycled concrete had a lower total porosity and a higher amount of pores of smaller size. Nevertheless, the authors also reported that 50 % and 75 % RMA replacements resulted in concrete mixtures displaying a higher number of larger pores.

4.5. MICROSTRUCTURE

~~The instrumental techniques used to study the recycled concrete paste microstructure in this research work detected a normal presence of portlandite, similar to the one detected for ordinary Portland concrete, able to react with silica, and common ettringite formations.~~ The microstructural tests revealed the formation of common hydration products (i.e. portlandite and calcium-silicate-hydrate gel). The instrumental techniques used to study the recycled concrete paste microstructure in this research work detected a normal presence of portlandite, similar to the one found in the control concrete, which was able to react with silica, and common ettringite formations. Figure 6 shows column-shaped aggregates of portlandite (CH) and Figure 7 plates of CH. Figure 8 displays the transformation of calcium silicate hydrates (C-S-H) needle forms into a “honeycomb” structure. For instance, Mas et al. [39], who used ceramic tiles waste as replacement material in Portland cement, also observed the presence of ettringite needles, hexagonal plates of portlandite and calcium silicate hydrate amorphous products in their recycled concrete samples. In the study carried out by Asensio de Lucas et al. [54], who employed a blended cement with a pozzolanic addition from clay-based CDW, the ceramic addition also produced primary ettringite, which provided the cement paste with better behavior against sulfate attacks. Conversely, Kannan et al. [83], who also conducted a microstructural investigation on concrete incorporating a partial cement replacement by ceramic waste powder, stated that insufficient amounts of CH were present to react with all the available silica when ceramic waste powder was incorporated.

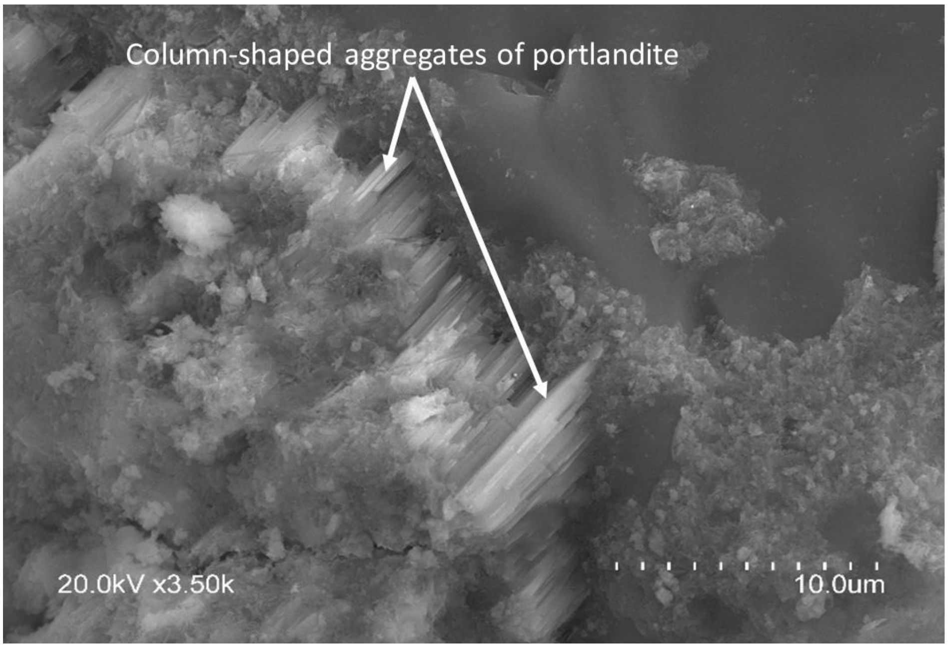


Figure 6: Column-shaped aggregation of portlandite.

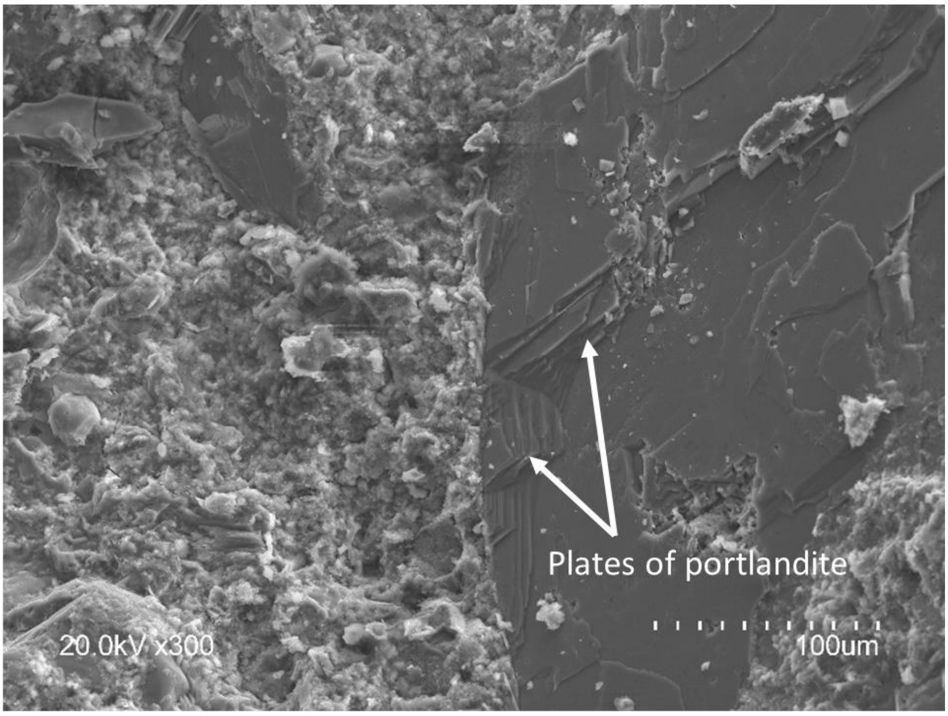


Figure 7: Plates of portlandite

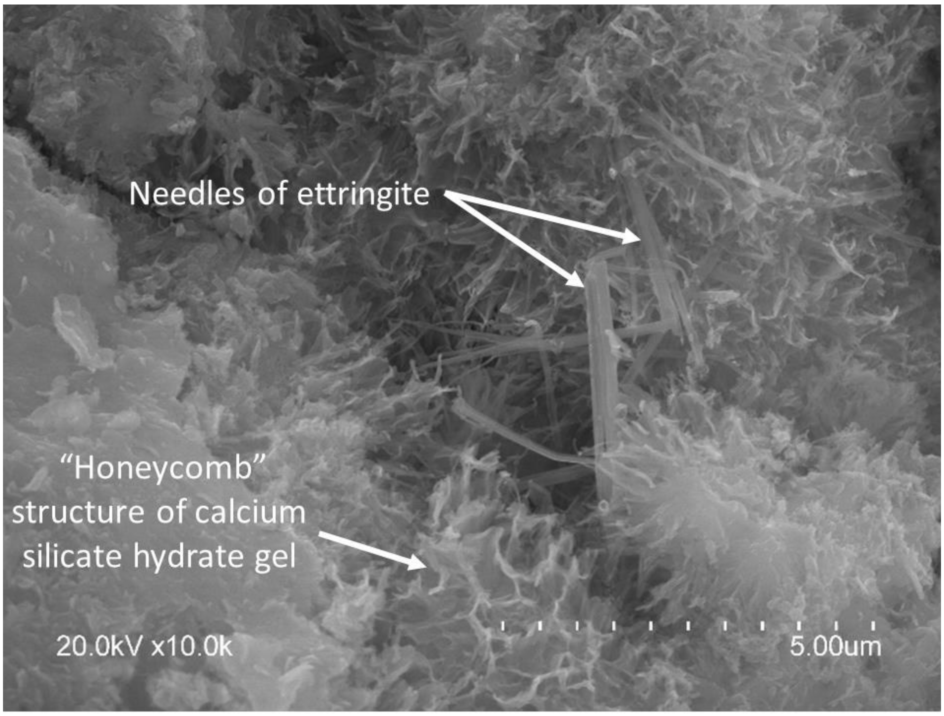


Figure 8: “Honeycomb” structure of calcium silicate hydrate gel and ettringite needles.

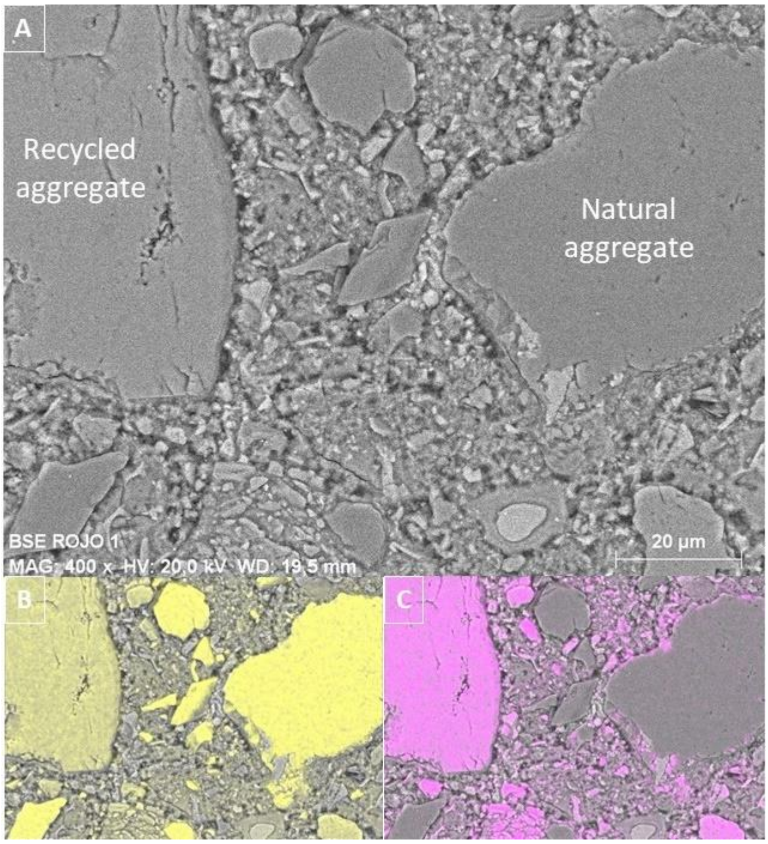


Figure 9. Elemental maps of RC-RAC sample: (A) Base image. (B) Elemental mapping for silicon (yellow). (C) Elemental mapping for aluminum (magenta).

Figure 9 shows the elemental mapping for aluminum (magenta) and silicon (yellow) present~~ce~~ in the RC-RAC sample at 28 days. The content of aluminum is typical in the recycled ceramic aggregates, whilst the silicon is the main component of the natural gravel. In addition, it is possible to appreciate the ITZ between the aggregates (natural and recycled) and the paste. As it could be observed, both ITZs ~~are~~ were very similar and appeared adequate and robust, which is in accordance to the findings ~~form~~ from the mechanical studies carried out in this investigation. Cantero et al. [50], who studied the structure of recycled concrete incorporating ceramics as cement addition and coarse recycled aggregates through an optical microscope, ~~established~~ stated that the aggregates bonded effectively to the matrix, irrespective of cement type, i.e. recycled or conventional cement.

5. CONCLUSIONS

The following conclusions have been drawn from the development and results obtained in ~~of~~ this research study:

1. ~~Adequate workability could be achieved for r~~Recycled concrete mixtures incorporating recycled mixed ceramic aggregates and cement additions from clay brick powder could reach an adequate workability. ~~The~~ Nevertheless, the results ~~obtained from~~of the slump and Vebe tests evidenced ~~drieds~~dry consistencies that could be improved by the use of plasticizing admixtures.

2. Due to the presence of the ceramic fraction within the recycled aggregates and cement, ~~T~~the density values exhibited by the recycled concrete mixes were slightly lower than that of the control concrete~~ due to the presence of the ceramic fraction within the recycled aggregates and recycled cement~~. Compared to CC, density reductions up to a 3.5 % and 3.7 % were observed for RC-RA and RA-RAC, respectively.

3. ~~As compressive strength is one of the most important characteristics of concrete, it is worth to mention that the~~The recycled mixtures ~~surely~~ safely complied with the of 25 MPa limit established by the Spanish Standard for Structural Concrete, as well as the 20 MPa limit established by the EN-1992-1-1. Thus, it is possible to ensure that the recycled concrete mixes could be used for structural purposes.

4. The porosity exhibited by the three concrete samples was similar and ranged between 12.4 % and 13 %. Nonetheless, changes in the pore size distribution were detected ~~for the different mixes~~. A clear refinement of the pore structure was observed for the recycled concretes due to the presence of the ceramic materials, which was especially noticeable for pore sizes lower than 10 µm.

5. The microstructural studies carried out in the recycled concrete mixtures detected the presence of the standard hydration products, such as portlandite, CSH gel and primary ettringite. Moreover, the assessment of the ITZ showed strong and continuous bonds between the cement paste and recycled ceramic aggregates, which were similar to those formed between the cement paste and natural coarse aggregates.

Therefore, ~~it could be concluded that~~ based on the mechanical results (density, consistency and compressive strength), as well as the microstructural properties such as porosity, elemental mapping analysis, hydration products and interfacial transition zones (ITZ), ~~showed~~ it could be concluded that the incorporation of the ceramic material as recycled aggregates or as cement addition did not imply an appreciable loss of the concrete performance. ~~and~~Moreover, the recycled concretes ~~resulted in mixtures that~~ meet the resistance requirements for structural applications established in the European standards ~~for the use of concrete in different structural applications~~. ~~However,~~

~~n~~Notwithstanding the potential shown by these materials at research level, ~~its~~ the technological transfer to field applications is still a distant reality.~~,~~ ~~since~~ To date, recycled mixed and ceramic aggregates are still considered with suspicion as a reliable source of secondary aggregates/additions for the concrete manufacture, especially for structural purposes. Thus, further research on the feasibility of the different uses of recycled aggregates from CDW in the concrete manufacture is necessary to reduce the uncertainty linked to ~~a~~ future field applications beyond their current use as unbound materials in earthworks, backfilling and road constructions, which would further extend the circularity and sustainability possibilities of such wastes.

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