



# Mechanical and fracture properties of concrete with recycled concrete aggregates treated with acids and addition of aluminium sulphate

J.A. Forero<sup>a</sup>, J. de Brito<sup>b</sup>, L. Evangelista<sup>b,c,\*</sup>, Cláudio H.F. Pereira<sup>a</sup>

<sup>a</sup> Postgraduate Program in Structural Engineering and Construction (PECC), Prédio SG-12 Campus Darcy Ribeiro, University of Brasília, Brasília, DF CEP 70910-900, Brazil

<sup>b</sup> CERIS, Instituto Superior Técnico, Universidade de Lisboa, Av. Rovisco Pais, Lisboa 1049-001, Portugal

<sup>c</sup> ISEL-IPL, Dept. of Civil Engineering, Instituto Superior de Engenharia de Lisboa, Rua Conselheiro Emídio Navarro, 1, Lisbon 1959-007, Portugal

## ARTICLE INFO

### Keywords:

Mechanical properties  
Fracture energy  
Wedge splitting test  
Recycled concrete aggregate  
Pre-soaking treatment

## ABSTRACT

The use of recycled aggregates, particularly concrete waste aggregate, has gained increasing popularity and has become current practice in day-to-day applications in many countries. Enhancing the physical and morphological characteristics of this type of aggregate is crucial to promote its widespread adoption. This study investigates the mechanical and fracture properties of concrete incorporating 100 % recycled concrete coarse aggregate (RCA), both untreated and treated with acid solutions of hydrochloric acid (HCl) and sulphuric acid (H<sub>2</sub>SO<sub>4</sub>), and addition to the addition of aluminium sulphate (AS). X-ray diffraction (XRD) and X-ray fluorescence (XRF) techniques were employed to characterize the cement and aluminium sulphate (AS). The performance of all mixes was evaluated in terms of mechanical properties, including compressive strength, tensile strength, and secant modulus of elasticity. Experimental fracture tests were conducted using the wedge splitting method on notched specimens aged for 28 days. The results demonstrate an improvement in the mechanical behaviour and fracture resistance of mixes incorporating RCA treated with acid solutions at concentrations of 0.3 M, 1 M, and 3 M for HCl, as well as at a concentration of 0.3 M for H<sub>2</sub>SO<sub>4</sub>. However, a decrease in properties was observed at higher concentrations of H<sub>2</sub>SO<sub>4</sub> (1 M and 3 M). Furthermore, the addition of AS also resulted in improvements compared to mixes containing RCA.

## 1. Introduction

Within the construction industry, there has been a significant increase in the use of recycled materials and encouragement for their utilization in the production of new concrete. One of the materials commonly employed in this context is recycled concrete aggregate (RCA), which replaces natural aggregate (NA), despite restrictions imposed by regulations in some countries [1]. These restrictions are based on the inferior characteristics of RCA compared to NA, such as high porosity, high water absorption, low density, and differences in the characteristics of the interfacial transition zones (ITZ) present in RCA, varying depending on the origin of the RCA.

These characteristics result in inferior mechanical performance of concrete produced with RCA, including lower fracture energy, compared to equivalent conventional mixes using NA [2,3]. Fracture energy ( $G_F$ ) is an essential parameter representing the toughness of concrete and the amount of energy required to create and propagate a crack in the material [3–9]. Crack propagation is related to the quality of the ITZ due to its role in stress transfer between the aggregate and the cementitious matrix, adhesion, and formation of micro-cracks resulting from differences in properties and stresses between the aggregate and the cementitious matrix. RCA surface modification emerges as a promising approach to mitigate these ITZ-dependent micro-cracks, thus improving its characteristics and resulting in an enhanced mechanical

*Abbreviations:* 3PBT, Three point bending test; AS, Aluminium sulphate; CMOD, Crack mouth opening displacement; CTT, Compact tension test;  $E_{cm}$ , Secant modulus of elasticity of concrete;  $F_{cm}$ , Mean value of concrete cylinder compressive strength;  $f_{ctm}$ , Mean value of axial tensile strength of concrete;  $G_F$ , Fracture energy; HCl, Hydrochloric acid; ITZ, Interfacial transition zone; KI, Stress intensity factor; NA, Natural aggregate; P.a, Purity analytics; RCA, Recycled concrete aggregate; WST, Wedge splitting test method; XRD, X-ray diffraction; XRF, X-ray fluorescence.

\* Corresponding author at: CERIS, Instituto Superior Técnico, Universidade de Lisboa, Av. Rovisco Pais, Lisboa 1049-001, Portugal.

*E-mail addresses:* [jaaandresforero@gmail.com](mailto:jaaandresforero@gmail.com) (J.A. Forero), [jb@civil.ist.utl.pt](mailto:jb@civil.ist.utl.pt) (J. de Brito), [evangelista@dec.isel.pt](mailto:evangelista@dec.isel.pt) (L. Evangelista), [claudiopereira@unb.br](mailto:claudiopereira@unb.br) (C.H.F. Pereira).

<https://doi.org/10.1016/j.conbuildmat.2024.137947>

Received 16 August 2023; Received in revised form 7 August 2024; Accepted 15 August 2024

Available online 29 August 2024

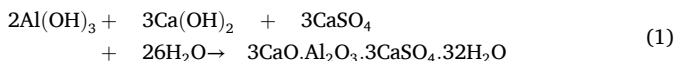
0950-0618/© 2024 Published by Elsevier Ltd.

response. In this context, the removal of mortar by means of acids improves the RCA surface, in addition to removing mortar fragments that are weakly adhered, or adding elements that solidify the ITZ. To evaluate this performance, concepts found in linear elastic fracture mechanics (LEFM) can be utilised, using parameters such as stress intensity factor (K) and fracture energy ( $G_F$ ) [10,11].

To improve the ITZ between the RCA and the cementitious matrix, different authors used acid concentrations of hydrochloric acid (HCl), sulphuric acid ( $H_2SO_4$ ) and phosphoric acid ( $H_3PO_4$ ), to remove the old mortar and improve the surface of RCA, obtaining improvements of the mechanical behaviour properties of concrete produced with treated RCA. Tam et al. [12], Ismail and Ramli [13], Purushothaman et al. [14] and Saravanakumar et al. [15] used acidic solutions at different concentrations of HCl and  $H_2SO_4$  phosphoric acid ( $H_3PO_4$ ), achieving improvements of the compressive strength, modulus of elasticity, and splitting tensile strength. This occurs because the acid treatment reduces the number of weakly adhered particles in RCA, leaving a cleaner and smoother surface with less sharp angles, thus improving the ITZ.

When increasing the degree of acidic molarity, Ismail and Ramli [13, 16] observed that the removal of the adhered mortar in RCA is greater due to the increase of hydrogen cations ( $H^+$ ), incrementing the dissociation of calcium hydroxide, which leads to a less porous RCA, with less water absorption, less mortar content and more uniform [17–19].

Another method to improve the ITZ of concrete with RCA is the addition of aluminium sulphate (AS). When a small amount of AS is added to cement, ettringite is mainly formed in small-size agglomerates, which are beneficial for the concrete's properties. When the aluminium sulphate content is high, calcium is consumed in small concentrations and the alkalinity of the solution decreases. Therefore, ettringite grows to a larger size and is widely spread. In short, on the one hand, the addition of aluminium sulphate consumes the calcium ion in solution, and on the other, it provides aluminium ion and sulphate ion that formed the ettringite, forming an environment for ettringite formation. Eq. 1 is the intermediate product - aluminium hydration - generally cannot exist constantly, so it soon turns to ettringite in the presence of calcium hydroxide and gypsum. At the same time, these reactions consume calcium hydroxide. The reduction of calcium hydroxide accelerates the hydration of calcium silicate. Consequently, the ettringite crystals grow and intersect in a large network, generating a cement paste skeleton, and the C-S-H gel helps to fill it, forming a denser structure. Then, the rapid and massive formation of ettringite and C-S-H gel accelerates cement setting [20–22].



The study by Briendl et al. [23] explored the initial hydration mechanisms of shotcrete binders. In this research, mixes containing low sulphate content were compared with those that included the addition of aluminium sulphate, an accelerator. The process in which  $Al^{3+}$  ions from the accelerator rapidly transformed into  $Al(OH)^4+$  ions in the alkaline conditions of the cement paste was detailed. These aluminium ions interacted with  $SO_4^{2-}$  ions and  $Ca^{2+}$  ions to form ettringite, which plays a crucial role in rapid hardening. The rapid formation of ettringite, together with the acidic nature of the accelerator, reduced  $Ca^{2+}$  levels and the pH in the pore solution, promoting the dissolution of alite and  $C_3A$ . This resulted in greater supersaturation in relation to calcium (aluminium) silicate hydrates (C-(A)-S-H) and the subsequent precipitation of nuclei of these phases. When analysing the compressive strength under different wet and dry curing conditions, it was observed that, compared to the dry mix, the samples subjected to wet curing had higher compressive strength values and lower total porosity, calculated after 24 hours and in later hydration phases. It is important to note that, although the accelerator contained approximately 65 % water by weight, it had a minimal effect on the water available for cement hydration. The addition of 7 % accelerator corresponded to 2.45 g of

$Al_2(SO_4)_3$  and 4.55 g of  $H_2O$  per 100 g of cement. This amount of accelerator was able to produce approximately 9 g/100 g of ettringite cement, containing around 4.13 g of  $H_2O$ . This indicates that most of the water present in the accelerator was consumed by the ettringite, formed as a result of the reaction between  $Al_2(SO_4)_3$  and  $Ca^{2+}$  from the pore solution. After 6 hours of hydration, the compressive strength was equivalent in both types of samples, while the calculated total porosity was lower in the sample with wet curing. Therefore, it is suggested that in the early stages of hydration, the morphology and mechanical properties of the phases formed are also crucial to the compressive strength of the cement matrix. After 6 hours of hydration, the compressive strength was equivalent in both types of samples, while the calculated total porosity was also lower in the sample with wet curing. It can therefore be assumed that, in the initial stages of hydration, the morphology and mechanical properties of the phases formed are also relevant to the compressive strength of the cementitious matrix.

Braz et al. [24] collected samples of metal waste from aluminium recycling in a tertiary aluminium industry. This waste is obtained from the black/salt dross from the secondary aluminium industry. In the tertiary industry, the raw material (black salt) is received in solid blocks from various locations in Brazil, broken up manually and leached with water in rotating drums to recover the metallic aluminium. In the study of [24], the incorporation of this aluminium waste into mortars containing 100 % of this material was investigated, resulting in significantly lower strengths compared to reference mortars containing cement. The values obtained were 42.25 %, indicating the absence of calcium silicate hydrate (C-S-H) formation, the component responsible for concrete strength. This absence is due to the lack of reactive silica in the composition containing the aluminium residue.

Kan et al. [21] showed that aluminium sulphate increases the curing time of concrete when 6 % of AS is used and decreases as the amount of AS is reduced (2.5 %). This effect is reflected on the fluidity of concrete, maintaining a constant fluidity in ranges from 1 % to 2 % of AS, whereas higher ranges of AS (2–10 %) fluidity is reduced because of the accelerating effect of AS. Kan et al. [21] also observed that the compressive strength increases with respect to the concrete mixes without AS when up to 5 % of AS was added. For additions greater than 8 %, the 28-day strength tends to be lower compared to the reference concrete. On the other hand, the tensile strength tends to be lower when any amount of AS is added.

Song et al. [25] studied concrete mixes with RCA and AS addition in amounts of 0.65 % and 1.10 % of the cement weight. The results showed that the mixes with 1.10 % AS addition increased the strength by around 14.7 % with respect to the reference concrete. Song et al. [25] attributes this improvement to the increase in the production of ettringite capable of reducing the porosity of the RCA and increasing the solidity of the mortar. At the same time, too much calcium hydroxide can reduce the preferential orientations of portlandite in ITZ and accelerate the process of consumption of tricalcium silicate/dicalcium silicate. These chemical reactions help to improve the ITZ and, therefore, improve the strength of the cement paste and of RCA.

However, in the literature there are studies that analyse the fracture behaviour of concrete RCA and found reduced performance compared to concrete with natural aggregates (NA). One of these studies was conducted by Ishiguro and Stanzl-Tschegg [9], who performed the total replacement of NA with RCA, resulting in a 60 % reduction in fracture energy when compared to a reference concrete. Other researchers, such as Casuccio et al. [26], Gesoglu et al. [27] and Garcia-Gonzales et al. [8] also observed a decrease in fracture energy, ranging from 20 % to 75 %, when using recycled aggregates compared to conventional concrete. This reduction depends on the incorporation ratio of RCA in concrete. In a study by Bordelon et al. [2], beams with 100 % replacement of natural aggregates with RCA were evaluated, obtaining a 34.89 % reduction in fracture energy in notched beam tests.

On the other hand, Kazemian et al. [3] showed a noticeable reduction of the fracture energy when RCA is incorporated, which produces

negative effects on crack growth that is more intense when adding RCA. When RCA was treated with 0.1 M HCl and calcium metasilicate was added, the fracture energy of concrete made with RCA was greater than that of mixes with untreated RCA. These results can be attributed to the treatment process and the availability of pozzolanic particles that coated RCA, which consequently reacted with cement during the hydration process and improved the ITZ.

To predict the fracture properties in concrete with incorporation of RCA, Ghorbel et al. [28] proposed a prediction model for fracture properties based on the model of Ghorbel and Wardeh [29], where the fit was improved with an inverse analysis of data collected in the literature, giving a good approximation of the  $G_F$ . The wedge splitting test method (WST) was chosen in this research article, because its approach eliminates the disadvantages of the three point bending test (3PBT) and compact tension test (CTT), i.e. the effect of the self-weight of the sample on the fracture result [30–32].

The main objective of this article is to evaluate the influence of RCA treated with acids and addition of AS on the mechanical behaviour and more specifically the fracture behaviour. The fracture properties of concrete with recycled aggregates is an essential element for the prediction of its service life and its behaviour, environmental and mechanical loads [33].

## 2. Materials and methods

### 2.1. Cement, aluminium sulphate and acids

The cement type used as the main binder in the composition of the reference concrete (RC) and the mixes with RCA was CEM I 42.5 R, which was produced by CIMPOR (Portugal). Two types of acids were used as a solvent for the removal of the adhered old paste in RA: hydrochloric acid (HCl) at 37 % purity analytics (p.a.) with a molecular weight of 36.46 g/mol, a specific gravity of 1.19 g/ml, pH < 1; and sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) at 95–97 % p.a. with a molecular weight of 98.08 g/mol, a specific gravity of 1.84 g/ml, pH < 1. These acids were prepared at three acid molarities: 0.3 M, 1.0 M and 3 M. The AS used comes from the Chem-Lab company, it is a solid crystal in powder with a molecular weight of 666.42 g/mol, density of 1.72 g/cm<sup>3</sup>, pH 3–4 and a solubility in water of 30 g/100 ml.

### 2.2. Aggregates and water

In this study, three types of NA were used: coarse aggregate CA1 (grain size 4–5.6 mm), CA2 (grain size 4–16 mm) and CA3 (grain size 4–22.4 mm); and two siliceous sands, FA1 (grain 0–2 mm) and FA2 (0–4 mm), all of them complying with the requirements of EN 12620 [34], to produce the RC. RCA was obtained by crushing concrete blocks at 28 days of age (using a jaw crusher as seen in Fig. 1a), followed by sieving in a range of sizes 0/25 mm, as presented in Fig. 1b. Tap water complying with Directive 98/83/CE [35] was used.

The size fraction of RCA from 4 mm to 22.4 mm was treated with two types of acids: H<sub>2</sub>SO<sub>4</sub> and HCl at three molarities (0.3 M, 1.0 M and 3 M). The method that was followed for the pre-soaking of the RCA is the one previously adopted by Ismail and Ramli [13], Tam et al. [12], Akbarnezhad et al. [36] and Purushothaman et al. [14]. First, the acidic solutions were prepared at 22 °C and then the aggregates were pre-soaked for 24 hours. After soaking, RCA underwent a single rinse with natural water and was dried at 105±5 °C for a period of 24 hours. Fig. 2 shows the steps of the pre-soaking procedure applied to RCA.

To evaluate the influence of AS on the concrete properties, 1.10 % of AS by weight of binder was added according to the study of Song et al. [17]. The AS was previously mixed in 1/3 of the mixing water and then placed in the mixer.

### 2.3. Mix design

The mixes' composition was based on the Faury's reference curve method [37], taking into account the following parameters: C30/37 strength class, XC3 exposure class and S3 slump in accordance with the EN 206–1 standard [38]. Using the Faury curve for the reference concrete (RC), 100 % coarse NA were replaced (particles size over 4 mm) with RCA through volumetric substitution performed by size fraction, ensuring the same equivalent volumetric fraction of the RC aggregates (Fig. 3).

The water absorption of RCA was higher than that of NA [14,39–41], which required compensation for the loss of free water in the mixes with RCA. RCA was expected to absorb approximately 92 % of the total water absorption of each type of RCA used, as measured in the absorption test over a 10-minute period (concrete production duration time): 3.72 % for untreated RCA; 3.35 % for RCA pre-soaked in 0.3 M HCl; 3.0 % for RCA pre-soaked in 1.0 M HCl; 2.98 % for RCA pre-soaked in 3.0 M HCl;



Fig. 1. - Jaw crusher used in the production of RCA (a); size fractions of RCA used in this study (b).



Fig. 2. - Pre-soaking treatment procedures for RCA.

3.52 % for RCA pre-soaked in 0.3 M  $H_2SO_4$ ; 3.60 % for RCA pre-soaked in 1.0 M  $H_2SO_4$ ; and 3.62 % for RCA pre-soaked in 3.0 M  $H_2SO_4$ . The concrete production process was divided into three phases. Initially, the three limestone gravel fractions CA1, CA2 and CA3 were mixed for 4 minutes using 2/3 of the total mixing water. Then, sands FA1 and FA2 were added and mixed for two minutes (second phase). Finally, the cement and 1/3 of the mixing water were added to the mixer, totalling 10 minutes of mixing. The proportions of the mixes are detailed in Table 1. The different mixes tested were named as follows: RC for the reference concrete (with NA); RC-RCA for the mixes with 100 % replacement of coarse NA ( $> 4$  mm) with RCA; RCA-0.3HCl for the mixes with 100 % RCA treated with 0.3 M HCl; RCA-1.0HCl for the mixes with 100 % RCA treated with 1.0 M HCl; RCA-3.0HCl for the mixes with 100 % RCA treated with 3.0 M HCl; RCA-0.3  $H_2SO_4$  for the mixes with 100 % RCA treated with 0.3 M  $H_2SO_4$ ; RCA-1.0  $H_2SO_4$  for the mixes with 100 % RCA treated with 1.0 M  $H_2SO_4$ ; RCA-3.0  $H_2SO_4$  for the mixes with 100 % RCA treated with 3.0 M  $H_2SO_4$ ; and RCA-AS for the mixes with 100 % RCA with added AS.

#### 2.4. Test methods and sample preparation

The chemical composition of cement was determined by the X-ray fluorescence technique (XRF). Likewise, to determine the main

crystalline phases present in cement and AS, X-ray diffraction (XRD) was used. In the fresh state, workability and density tests were performed, according to the EN 12350-2 [42] and EN 12350-6 [43] standards.

The compressive strength of hardened specimens was evaluated according to EN 12390-3 [43]. This test was performed at 7 and 28 days using three cylindrical specimens on 150 mm diameter  $\times$  300 mm height cylindrical specimens for each composition. The splitting tensile strength test followed the procedures presented in EN 12390-6 [44]. This test was performed on 150 mm diameter  $\times$  300 mm height cylindrical specimens at 28 days. The test procedure used to determine the secant elastic modulus of elasticity is described in LNEC E 397 [45]. This test was performed on 150 mm diameter  $\times$  300 mm height cylindrical specimens at 28 days.

The wedge splitting test (WST) [31] was performed on four 150 mm cubic samples (Fig. 4a-b) for each composition, in order to determine the fracture energy according to the guidelines of the NT BUILD 511 standard [46]. The fracture energy is measured in this method through the area under the  $F_{sp}$ -CMOD curve. The merit of this approach is to minimize the weight effect on the fracture energy, unlike other methods in which weight has an effect of 40–60 % on the fracture energy [30–32, 46]. In this method, a wedge was generated using special moulds in the casting and the starter and guide notches were made in the sample after 15 days of curing (wet chamber) using a diamond saw blade. To fix the

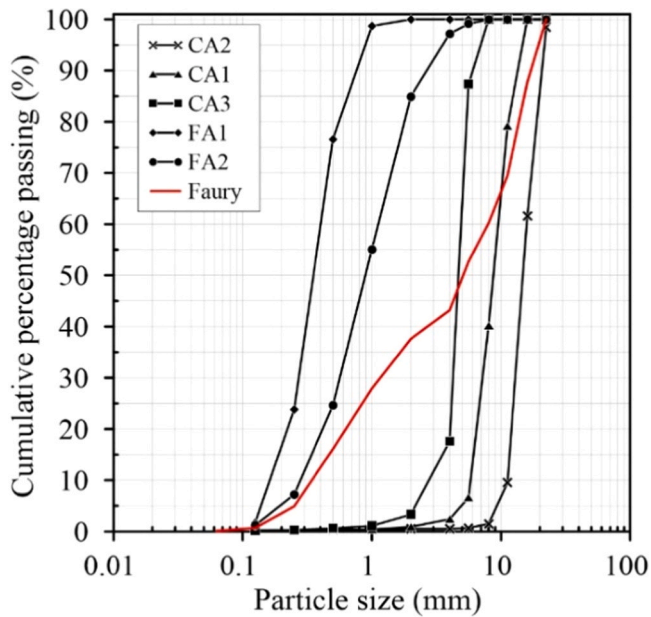


Fig. 3. - Particle size distribution of the aggregates and Faury’s reference curve.

specimen on the device, it was placed on a linear support and then two pieces with rollers were mounted on the sample to transfer the load. Afterwards, a hard steel profile with two identical wedges was attached to the device. The load was applied vertically ( $F_v$ ) with a controlled

deformation of 0.6 mm/s according to the NT BUILD 511 standard [46], dividing the sample into two sections where the crack mouth opening displacement (CMOD) is measured with the help of callipers. After the test, the depth and the width of the split surface ( $A_{lig}$ ) were measured. The procedures for carrying out the WST test are illustrated in Fig. 5, which shows the gradual application of force until failure occurs, corresponding to totally split surface ( $A_{lig}$ ).

$F_{sp}$  is calculated as per Eq. 2, where  $\alpha$  is the angle between the wedge and the vertical load line. The area of the  $F_{sp}$ -CMOD curve is the energy dissipated during the fracture  $W_{f,CMOD}$ , and is normalized with respect to the total split surface ( $A_{lig}$ ) to complement the fracture. This intermediate specific fracture energy is denoted  $G_F$  (kJ/mm) and is determined from the test result as per Eq. 3 [31,32,46]. The double K fracture criterion and the double G fracture criterion are based on asymptotic linear superposition, maintaining the relationship of the linear elastic fracture mechanics valid at all points of the  $F_{sp}$ -CMOD envelope. Therefore, the effective double  $K_I$  fracture is obtained in terms of equivalent stress intensity factors using the double  $G_F$  fracture model. Eq. 4 relates the two criteria; double  $K_I$  fracture model and double  $G_F$  fracture model [47].

$$F_{sp} = \frac{F_v}{2 \tan(\alpha)} \tag{2}$$

$$G_F = \frac{W_{f,CMOD}}{A_{lig}} \tag{3}$$

$$K_I = (G_F E_{cm})^{\frac{1}{2}} \tag{4}$$

Table 1  
Concrete mix proportions.

Material	Mix proportions (kg/m <sup>3</sup> )								
	RC	R-RCA	RCA-0.3HCl	RCA-1.0HCl	RCA-3.0HCl	RCA-0.3 H <sub>2</sub> SO <sub>4</sub>	RCA-1.0 H <sub>2</sub> SO <sub>4</sub>	RCA-3.0 H <sub>2</sub> SO <sub>4</sub>	RCA-AS
RCA incorporation ratio	0 %	100 %	100 %	100 %	100 %	100 %	100 %	100 %	100 %
Cement (CEM I 42.5 R)	350	350	350	350	350	350	350	350	350
Aluminium sulphate (AS)	-	-	-	-	-	-	-	-	3.85
Water	175	175	175	175	175	175	175	175	175
Additional water	-	20.9	18.4	17.6	17.6	18.7	19.0	19.1	-
W/C	0.5	0.55	0.55	0.55	0.55	0.55	0.55	0.55	0.5
FA1	179.4	179.4	179.4	179.4	179.4	179.4	179.4	179.4	179.4
FA2	446.8	446.8	446.8	446.8	446.8	446.8	446.8	446.8	446.8
CA1	365.7	-	-	-	-	-	-	-	-
CA2	640.0	-	-	-	-	-	-	-	-
CA3	182.8	-	-	-	-	-	-	-	-
RCA > 22.4 mm	-	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0
RCA 16–22.4 mm	-	232.0	232.0	232.0	232.0	232.0	232.0	232.0	232.0
RCA 11.2–16 mm	-	403.7	403.7	403.7	403.7	403.7	403.7	403.7	403.7
RCA 8–11.2 mm	-	193.4	193.4	193.4	193.4	193.4	193.4	193.4	193.4
RCA 5.6–11.2 mm	-	155.3	155.3	155.3	155.3	155.3	155.3	155.3	155.3
RCA 4–5.6 mm	-	152.8	152.8	152.8	152.8	152.8	152.8	152.8	152.8

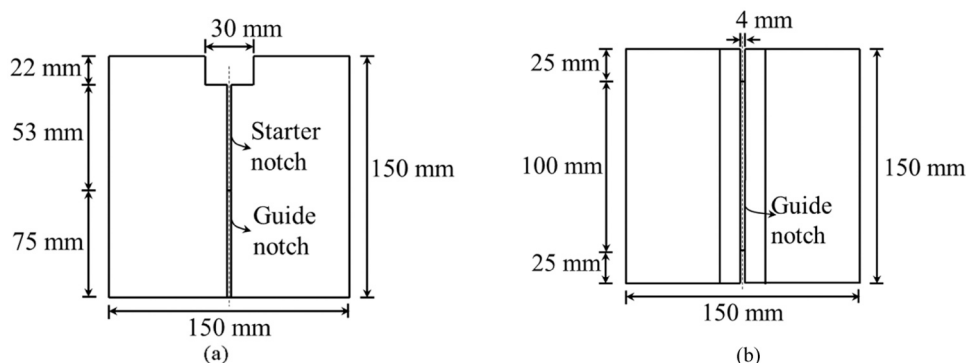


Fig. 4. - Illustration of the specimens' geometry: front view (a); top view (b) as per NT BUILD 511 [46].

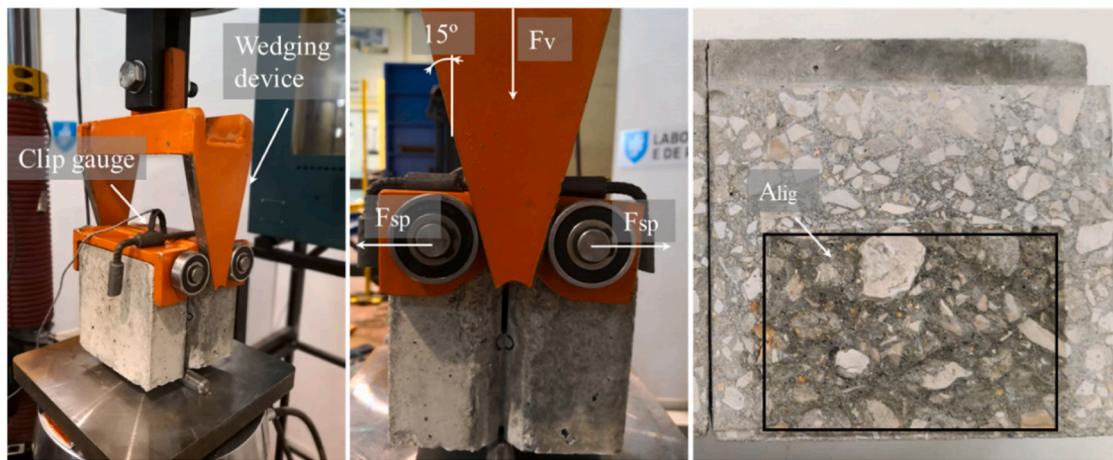


Fig. 5. - Illustration of the application of the splitting load.

### 3. Results and discussion

#### 3.1. Characterization of the materials

To determine the chemical composition of CEM I 42.5 R and the crystalline phases of AS, X-ray fluorescence (XRF) analysis and X-ray diffraction (XRD) tests were performed. The chemical composition of cement is presented in Table 2, which shows that the cement complies with EN 197 [48]. The main phases identified in CEM I 42.5 R are shown in Fig. 6a. The crystalline phases of AS were also determined, where the main crystalline phase is Alunogen (Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> · 17 H<sub>2</sub>O), as shown in Fig. 6b.

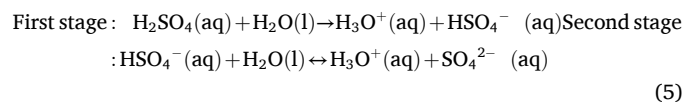
#### 3.2. Compressive strength

The average compressive strength, its standard deviation, and the improvement relative to RC and concrete with RC-RCA are presented in Fig. 7a, for concrete cylindrical specimens prepared with natural aggregates (NA), untreated RCA, and RCA treated with solutions of HCl, H<sub>2</sub>SO<sub>4</sub>, and the addition of AS, respectively. The figure shows variations in compressive strength across all mixes. Notably, the RC-RCA mix exhibits a reduction of 13.76 % and 19.80 % at 7 and 28 days, respectively, compared to the conventional RC mix. This reduction of the strength is explained due to the RCA's lower density than the NA due to the adhered mortar that also increases the water absorption and the w/c ratio needed to produce new concrete, generating greater contents of pores. Furthermore, RCA has more than one ITZ, which is the zone that undermines concrete strength [4,40,49–51].

The mixes with RCA treated with HCl and H<sub>2</sub>SO<sub>4</sub> showed similar compressive strength results at 7 days, with a reduction of only 1.71 % in the RCA-0.3HCl mix and an increase of 1.58 % in the RCA-0.3 H<sub>2</sub>SO<sub>4</sub> mix, compared to the reference concrete (RC-RCA). On the other hand, a remarkable increase in compressive strength at 28 days was observed for the RCA-0.3HCl, RCA-1.0HCl and RCA-3.0HCl mixes, of 7.76 %, 11 % and 10 %, respectively, compared to RC-RCA. This phenomenon can be attributed to the reduction of the amount of mortar adhered to the RCA, which makes the aggregate less porous and with a cleaner surface, improving the ITZ between the RCA and the cementitious matrix. Similar results were obtained by Ismael et al. [13,16], Purushothaman [14], Saravanakumar et al. [15], Pandurangan et al. [52], Kim et al. [53]

and Tang et al. [54].

Studies by Ismail and Ramli [13], Juan and Gutierrez [55], Kim [53], Abbas et al. [56], Al-Bayati et al. [57] and Saravankumar et al. [15] have shown that, by increasing the acid concentration (M), the removal of the paste adhered to the RCA is greater because the dissolution of calcium hydroxide caused by acid attack proceeds in two phases. The first one is the reaction of the acid with the calcium hydroxide (base) in the cement paste. The second phase is the chemical reaction with the calcium silicate hydrate. As expected, the second phase does not begin until all calcium hydroxide is consumed [58–60]. When the acid molarity is increased, the pH lowers, and the dissolution of calcium hydroxide occurs more quickly, reducing the amount of mortar in the RCA, improving its quality. For this reason, it is reasonable to assume that there is a relationship between molarity and compressive strength. Fig. 7b shows the positive correlation between HCl concentration and compressive strength at 7 and 28 days with a linear fit with good correlation (R<sup>2</sup> = 0.80) at 7 days and a linear fit (R<sup>2</sup> = 0.56) at 28 days. For concentrations of H<sub>2</sub>SO<sub>4</sub> (Fig. 7c), there is a negative correlation when the molarity is increased, with a linear fit (R<sup>2</sup> = 0.83 and 0.62, at 7 and 28 days, respectively). This can be explained because of the ionization of H<sub>2</sub>SO<sub>4</sub> (Eq. 5) [61], in which, in the first stage, an H<sub>2</sub>SO<sub>4</sub> molecule reacts with a water molecule. During this process, H<sub>2</sub>SO<sub>4</sub> donates a proton to the water molecule, generating a hydronium ion and the hydrogen sulphate ion. In the second stage, the hydrogen sulphate ion interacts with a water molecule, establishing a balance between reactants and products. This leads to the formation of a hydronium ion and a sulphate ion. In other words, it is plausible that a new degradation reaction occurs in the RCA when it is exposed to the water from the mix. Measuring the pH of the treated RCA revealed a result of 5.2, indicating the presence of the hydronium ion.



In the mixes with addition of AS, RCA-AS, the compressive strength increased by 16.6 % with respect to RC-RCA concrete, and 0.61 % to RC concrete. This high initial strength is explained by the fact that AS increases the acceleration of cement hydration by increasing the formation of calcium hydroxide, visible in the strength at 7 days [21]. This

Table 2  
Chemical composition of cement (% by mass).

Component	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	Cl <sup>-</sup>	LOI	Insoluble residue
CEM I 42.5 R	19.59	5.24	3.17	62.71	2.23	3.13	0.01	2,94	1.37

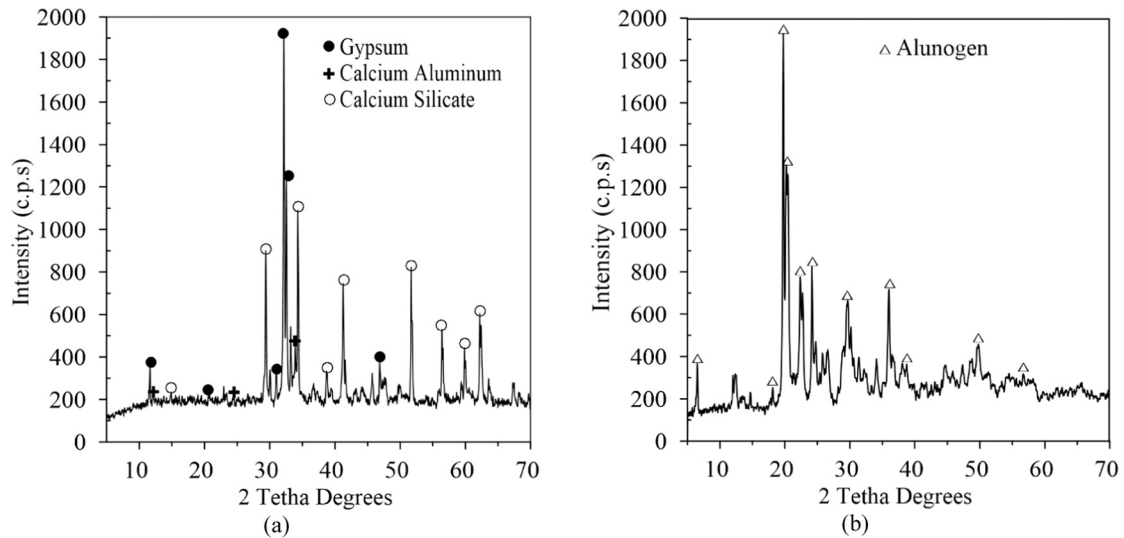


Fig. 6. - XRD pattern of CEM I 42.5 R (a) and AS (b).

behaviour continues until 28 days, where the difference to the strength at 7 days is not significant, since most of the calcium hydroxides are already formed.

### 3.3. Tensile strength

Fig. 8a, as well as Table 4, present the splitting tensile strength results, demonstrating a significant increase with increasing molarity of HCl and H<sub>2</sub>SO<sub>4</sub>, as well as with the addition of AS relative to the RC-RCA reference mix. Notably, RCA mixes treated with HCl exhibit higher tensile strength compared to those treated with H<sub>2</sub>SO<sub>4</sub> and AS after 28 days of curing. The higher values for RCA-0.3HCl, RCA-1.0HCl, RCA-3.0HCl, RCA-0.3 H<sub>2</sub>SO<sub>4</sub>, RCA1.0 H<sub>2</sub>SO<sub>4</sub> and RCA-AS mixtures are 34.61 %, 39.21 %, 40.82 %, 29.92 %, 5.55 % and 14.10 %, respectively, compared to RC-RCA. This trend can be attributed to the same factors discussed earlier as mentioned in the previous section that addressed compressive strength, notably the improvement of the ITZ due to loss of porosity. Similarly, mixes containing RCA treated with H<sub>2</sub>SO<sub>4</sub>, specifically RCA-3.0 H<sub>2</sub>SO<sub>4</sub>, showed a 2.57 % reduction compared to RC-RCA. Fig. 8b illustrates the relationship between compressive strength and tensile strength in cylindrical specimens for all mixes after 28 days of curing. The results indicate that all mixes are within the confidence intervals for the tensile strength of conventional concrete ( $f_{ctk}$ ). This suggests that the mixes analysed have a satisfactory behaviour and are adequate in terms of tensile strength compared to conventional concrete.

### 3.4. Secant modulus of elasticity

The results of the modulus of elasticity of all tested concrete mixes can be seen in Fig. 9. Fig. 9a shows the reduction of this property as RCA is incorporated in the mix. After 28 days, the modulus of elasticity ( $E_{cm}$ ) of the RC-RCA mix exhibits a reduction of around 26.0 % relative to RC, whereas for the values for the mixes with treated RCA the reduction is lower, i.e. 21 %, 19.45 %, 16.58 %, 24.29 %, 33.43 %, 35.33 % and 20.71 % for the RCA-0.3HCl, RCA-1.0HCl, RCA-3.0HCl, RCA-0.3 H<sub>2</sub>SO<sub>4</sub>, RCA-1.0 H<sub>2</sub>SO<sub>4</sub>, 3.0 H<sub>2</sub>SO<sub>4</sub> and RCA-AS mixes, respectively. The lower values of  $E_{cm}$  of the mixes with RCA can be attributed to higher porosity and lower stiffness of RCA in comparison with NA [62–65]. According to Beshr et al. [66], the stiffness of coarse aggregates greatly influences the concrete's modulus of elasticity. Aitcin and Mehta [67] also pointed out that the low strength of the aggregate can result in a concrete with a

lower modulus of elasticity. It has been observed that treating RCA with 0.3 M, 1.0 M, and 3.0 M HCl, as well as 0.3 M H<sub>2</sub>SO<sub>4</sub> solutions, leads to a noticeable improvement in the  $E_{cm}$  values. This improvement can be attributed to the enhanced interface connections between the aggregate and cement paste, effectively reducing crack propagation during loading. These results are similar to those found by several authors [12–14,16]. In the case of RCA-1.0 H<sub>2</sub>SO<sub>4</sub> and RCA-3.0 H<sub>2</sub>SO<sub>4</sub> mixes, the  $E_{cm}$  reduction relative to the RC-RCA mix was found to be 10.0 % and 12.64 %, respectively, which aligns with the observed reduction in compressive strength for these mixes. Conversely, in the RC-AS mix,  $E_{cm}$  exhibited an increase, attributed to the positive influence of AS. AS contributes to the improvement of the ITZ by reducing porosity and enhancing the solidification of the cement mortar.

Fig. 9b illustrates the relationship between the theoretical values, calculated according to Eq. 6 in accordance with the expression recommended by Eurocode 2 [68], and the experimental results obtained. This graph shows a remarkable correlation between the compressive strength ( $f_{cm}$ ) and modulus of elasticity ( $E_{cm}$ ) values. It is worth noting that, when analysing the evolution of the results, a consistent improvement can be seen in the mixes subjected to the aforementioned RCA treatments, with the exception of the RCA-1.0 H<sub>2</sub>SO<sub>4</sub> and RCA-3.0 H<sub>2</sub>SO<sub>4</sub> mixes, which do not show the same positive progression in the relationship between  $f_{cm}$  and  $E_{cm}$ . It is important to emphasise that a slight improvement in the evolution of the mixes' results was identified, especially when they were subjected to the RCA treatments described above. However, it is stressed that the RCA-1.0 H<sub>2</sub>SO<sub>4</sub> and RCA-3.0 H<sub>2</sub>SO<sub>4</sub> mixes did not follow this trend of improvement in the ratio between  $f_{cm}$  and  $E_{cm}$ .

Finally, Table 5 shows the percentage variation in modulus of elasticity values for each mix compared to the reference concrete mixes containing untreated natural (NA) and recycled (RCA) aggregates.

$$E_{cm} = 22 \left( \frac{f_{cm}}{10} \right)^{0.3} \quad (6)$$

### 3.5. Fracture energy

The results of fracture energy are presented in Table 6, along with their respective standard deviations and the percentage variation relative to the RC and RC-RCA mixes. It is evident that the RC-RCA mix exhibits a decrease of 54.5 % compared to the RC mix. This trend has been observed in previous studies by Casuccio et al. [20],

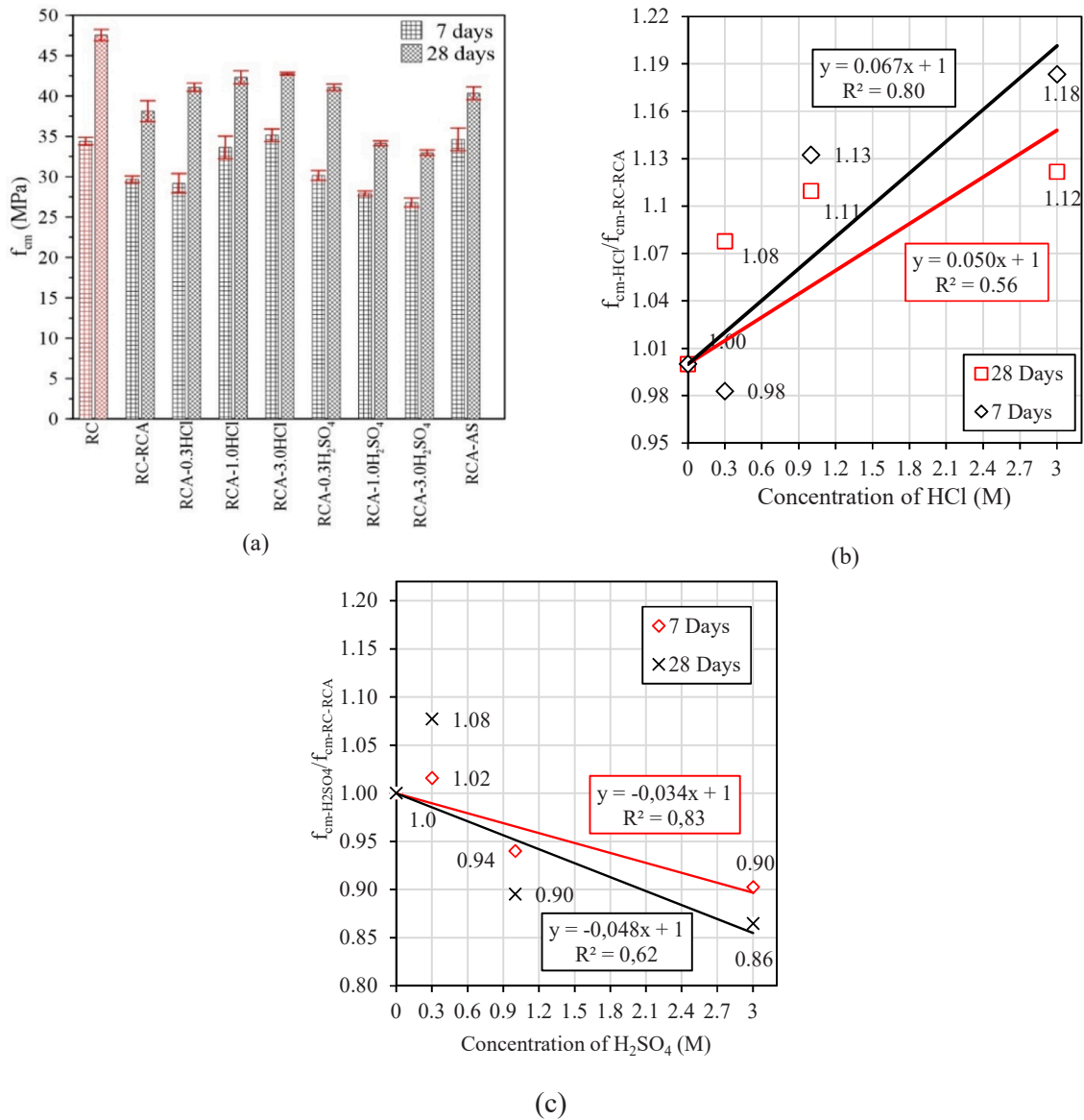


Fig. 7. - Compressive strength of cylindrical specimens ( $f_{cm}$ ) at 28 days (a); normalisation of compressive strength ( $f_{cm}$ ) versus acid concentration of HCl (M) (b) and normalisation of compressive strength ( $f_{cm}$ ) versus acid concentration of H<sub>2</sub>SO<sub>4</sub> (M) (c).

Table 3  
- Compressive strength of cylindrical specimens ( $f_{cm}$ ).

Mix	7 days			28 days				
	$f_{cm}$ (MPa)	$\sigma$	% $f_{cm}$ improvement relative to:		$f_{cm}$	$\sigma$	% $f_{cm}$ improvement relative to:	
			RC	RC-RCA			RC	RC-RCA
RC	34.41	0.47	-	-	47.54	0.70	-	-
RC-RCA	29.70	0.40	↓ up to 13.72 %	-	38.13	1.30	↓ up to 19.80 %	-
RCA-0.3HCl	29.19	1.20	↓ up to 15.20 %	↓ up to 1.71 %	41.09	0.50	↓ up to 13.56 %	↑ up to 7.76 %
RCA-1.0HCl	33.63	1.40	↓ up to 2.29 %	↑ up to 13.72 %	42.31	0.80	↓ up to 11.00 %	↑ up to 11.0 %
RCA-3.0HCl	35.15	1.43	↑ up to 2.10 %	↑ up to 18.34 %	42.77	0.16	↓ up to 10.03 %	↑ up to 12.1 %
RCA-0.3 H <sub>2</sub> SO <sub>4</sub>	30.17	2.20	↓ up to 12.36 %	↑ up to 1.58 %	41.07	1.00	↓ up to 13.60 %	↑ up to 7.71 %
RCA-1.0 H <sub>2</sub> SO <sub>4</sub>	27.92	1.50	↓ up to 18.88 %	↓ up to 6.00 %	34.14	0.30	↓ up to 28.18 %	↓ up to 10.46 %
RCA-3.0 H <sub>2</sub> SO <sub>4</sub>	26.81	0.55	↓ up to 22.15 %	↓ up to 9.74 %	32.97	0.34	↓ up to 30.64 %	↓ up to 13.53 %
RCA-AS	34.63	1.40	↑ up to 0.61 %	↑ up to 16.60 %	40.34	0.80	↓ up to 15.14 %	↑ up to 5.80 %

Garcia-Gonzalez [5], Ishiguro and Stanzl-Tschegg [7], Bordelon et al. [63], and Sato et al. [62], and has been attributed to the substantial amount of broken coarse aggregates with less crack branching within the cementitious matrix. Ghorbel and Warden [23] reported a decrease

of approximately 35 %, 39 %, and 40 % for RCA incorporations of 30 %, 65 %, and 100 %, respectively, relative to the reference mix without recycled aggregates. They also demonstrated that there is no sharp decrease of ( $G_f$ ) for the three mixes containing recycled gravel, as the

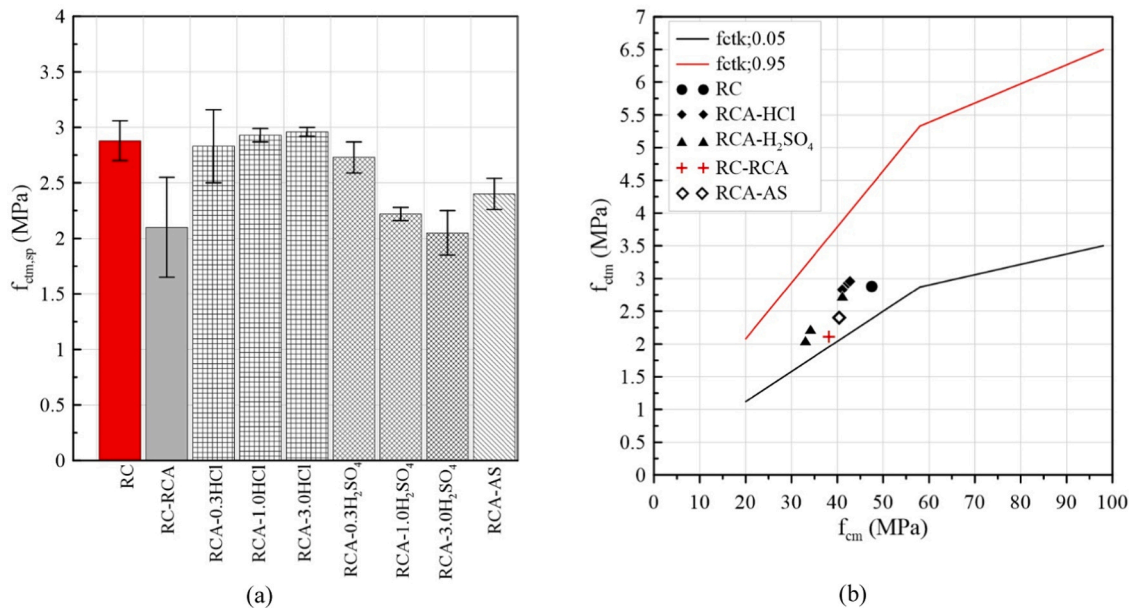


Fig. 8. - Splitting tensile strength ( $f_{ctm,sp}$ ) in mixes with NA and untreated and treated RCA (a) relationship between splitting tensile strength ( $f_{ctm}$ ) and compressive strength in cylindrical specimens ( $f_{cm}$ ) at 28 days (b).

Table 4  
- Splitting tensile strength ( $f_{ctm}$ ) at 28 days.

Mix	28 days			
	$f_{ctm}$ (MPa)	$\sigma$	% $f_{ctm}$ improvement relative to	
			RC	RC-RCA
RC	2.88	0.18	-	-
RC-RCA	2.10	0.45	↓up to 26.87 %	-
RCA-0.3HCl	2.83	0.33	↓up to 1.56 %	↑up to 34.61 %
RCA-1.0HCl	2.92	0.06	↑up to 1.56 %	↑up to 39.21 %
RCA-3.0HCl	2.96	0.04	↑up to 2.75 %	↑up to 40.82 %
RCA-0.3 H <sub>2</sub> SO <sub>4</sub>	2.73	0.14	↓up to 5.20 %	↑up to 30.00 %
RCA-1.0 H <sub>2</sub> SO <sub>4</sub>	2.22	0.06	↓up to 22.97 %	↑up to 5.55 %
RCA-3.0 H <sub>2</sub> SO <sub>4</sub>	2.05	0.20	↓up to 28.90 %	↓up to 2.57 %
RCA-AS	2.40	0.14	↓up to 16.56 %	↑up to 14.10 %

impact is compensated by the increase in paste volume.

When RCA is treated with acid solutions, a significant improvement in  $G_F$  is observed, resulting in reductions of 41.5 %, 16.25 %, 14.77 % and 33.64 % for the RCA-0.3HCl, RCA-1.0HCl, RCA-3.0HCl and RCA-0.3 H<sub>2</sub>SO<sub>4</sub> mixes, respectively, compared to untreated RCA (RC). This improvement is attributed to the acid treatments enhancing the RCA surface leading to an improved interfacial transition zone (ITZ), with the exception of the RCA-1.0 H<sub>2</sub>SO<sub>4</sub> and RCA-3.0 H<sub>2</sub>SO<sub>4</sub> mixes, which exhibited a less favourable behaviour with a reduction of 58.9 % and 59.34 %, respectively. This can be explained by the continuous degradation of RCA resulting from the persistence of sulphate ions, which were not effectively removed during the previous washing process. Thus, the presence of these ions in the RCA matrix may have compromised the positive effects observed in the other acid treatments. These results suggest that the choice of acid solution and the washing process are crucial factors to obtain significant improvements in the  $G_F$  of RCA. In a study by Kazemian et al. [6], a non-significant variation of 5 % was observed with 25 % and 50 % incorporations of RCA treated in two stages, involving HCl and calcium metasilicate. This indicates an improvement in fracture energy absorption with this type of treated RCA. However, the RCA-AS mix (Fig. 10e) indicated that concrete mixes with addition of 1.10 % aluminium sulphate (AS) showed a more brittle response. The softening curve was lower, which translated into a slight reduction in fracture energy compared to the RC-RCA reference

concrete. The production of ettringite, which reduces the porosity of RCA, together with the consumption of calcium hydroxide, which reduces the orientations of portlandite in the ITZ, possibly left this zone more prone to micro-crack formations. This embrittlement has been discussed in previous works [20,21,69,70]. The mixes with AS had a reduction of 6.48 % compared to RC-RCA.

Fig. 10 presents the Fps-CMD response, where different mixes are shown: RC and RC-RCA in Fig. 10a, RCA-HCl mixes in Fig. 10b, RCA-H<sub>2</sub>SO<sub>4</sub> mixes in Fig. 10c and mixes with AS - RCA-AS in Fig. 10d. From these results, it can be observed that the RCA-AS and RCA-0.3 H<sub>2</sub>SO<sub>4</sub> mixes show a lower toughness compared to the RCA-1.0HCl, RC-3.0HCl and RCA-0.3 H<sub>2</sub>SO<sub>4</sub> mixes. These results suggest that the mixes treated with RCA-AS and RCA-0.3 H<sub>2</sub>SO<sub>4</sub> may not have proved as effective in terms of toughness when compared to the other compositions analysed. Garcia-Gonzalez [8] showed that there is a good correlation between compressive strength and  $G_F$  for the RCA incorporation ratios used. In Fig. 10e and f, the relationship between normalized compressive strength and fracture energy is shown for each concrete mix with RCA treated with HCl (Fig. 10a) and concrete with RCA treated with H<sub>2</sub>SO<sub>4</sub> (Fig. 10 f). It can be concluded that, as compressive strength increases, the fracture energy proportionally increases, indicating a greater capacity to resist fracture. It can also be concluded that as the stress intensity factor ( $K_I$ ) increases, the energy required to propagate the crack also increases. This indicates that the mixes with RCA treated with HCl and H<sub>2</sub>SO<sub>4</sub> require higher fracture energy for crack propagation.

In Table 6, it is possible to observe the average values of the stress intensity factor ( $K_I$ ) for all mixes, together with  $G_F$  and its variation in relation to the RC and RC-RCA reference mixes. It is concluded that the treatment with HCl is more effective in cracking resistance than the treatment with H<sub>2</sub>SO<sub>4</sub>, due to its effectiveness in higher concentrations. Finally, the incorporation of AS did not show significant improvements in comparison with the RC-RCA reference concrete.

#### 4. Conclusions

In summary, after analysing the mechanical and fracture behaviour of several concrete mixes with RCA not treated and treated with HCl, H<sub>2</sub>SO<sub>4</sub> and AS, the following conclusions are drawn:

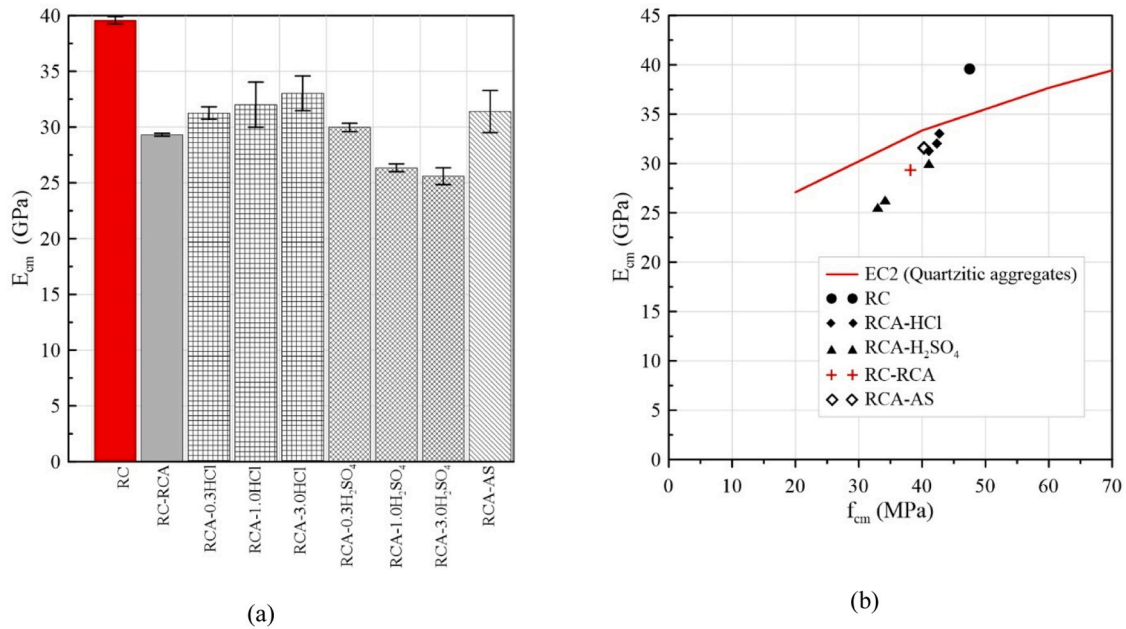


Fig. 9. - Modulus of elasticity (E<sub>cm</sub>) at 28 days (a); relationship between E<sub>cm</sub> and f<sub>cm</sub> at 28 days (b).

Table 5  
Modulus of elasticity (E<sub>cm</sub>) at 28 days.

Mix	28 days			
	E <sub>cm</sub> (GPa)	σ	% E <sub>cm</sub> improvement relative to:	
			RC	RC-RCA
RC	39.58	0.33	-	-
RC-RCA	29.31	0.13	↓up to 26.00 %	-
RCA0.3HCl	31.25	0.56	↓up to 21.06 %	↑up to 6.62 %
RCA1.0HCl	32.01	2.02	↓up to 19.45 %	↑up to 9.21 %
RCA3.0HCl	33.02	1.56	↓up to 16.58 %	↑up to 12.68 %
RCA0.3 H <sub>2</sub> SO <sub>4</sub>	30.00	2.02	↓up to 24.29 %	↑up to 2.26 %
RCA1.0 H <sub>2</sub> SO <sub>4</sub>	26.35	0.38	↓up to 33.43 %	↓up to 10.00 %
RCA3.0 H <sub>2</sub> SO <sub>4</sub>	25.60	0.75	↓up to 35.33 %	↓up to 12.64 %
RCA-AS	31.60	0.35	↓up to 20.71 %	↑up to 7.09 %

Table 6  
- Fracture energy (G<sub>F</sub>) and fracture toughness (K<sub>I</sub>) results.

Mix	28 days				
	G <sub>F</sub> (N/m)	σ	K <sub>I</sub> (MPa·m <sup>1/2</sup> )	% G <sub>F</sub> improvement relative to	
				RC	RC-RCA
RC	139.0	9.44	2.34	-	-
RC-RCA	63.35	3.79	1.36	↓up to 54.50 %	-
RCA-0.3HCl	81.26	8.21	1.59	↓up to 41.55 %	↑up to 28.50 %
RCA-1.0HCl	116.4	12.13	1.93	↓up to 16.25 %	↑up to 84.00 %
RCA-3.0HCl	118.5	4.5	1.98	↓up to 14.77 %	↑up to 87.30 %
RCA-0.3 H <sub>2</sub> SO <sub>4</sub>	92.26	3.53	1.64	↓up to 33.64 %	↑up to 45.85 %
RCA-1.0 H <sub>2</sub> SO <sub>4</sub>	57.26	4.34	1.23	↓up to 58.86 %	↓up to 9.58 %
RCA-3.0 H <sub>2</sub> SO <sub>4</sub>	56.53	5.89	1.20	↓up to 59.34 %	↓up to 10.65 %
RCA-AS	59.16	3.0	1.36	↓up to 57.44 %	↓up to 6.46 %

- The compressive strength of concrete mixes containing recycled aggregates (RCA) treated with HCl showed that this treatment is an effective solution of improving the characteristics of RCA. A notable increase of 10 % and 12.17 % in compressive strength was observed for the highest concentration, compared to the RC and RC-RCA reference concrete mixes, respectively;
- The use of H<sub>2</sub>SO<sub>4</sub> improves the strength when used at low molar concentrations, but in high concentrations the degradation process of RCA continues, possibly due to the dihydropic ionization of H<sub>2</sub>SO<sub>4</sub>, owing to the cleaning process used; this led to a low pH in the RCA, allowing the existence of sulphate ions, which when in contact with the mixing water can activate this reaction;
- Finally, in relation to the addition of AS, it can be concluded that the main improvement in strength occurs in the first ages of curing, with no differences in the more advanced ages;
- The splitting tensile strength and E<sub>cm</sub> have also improved due to improved surface and reduced water absorption of the RCA, with the exception of the acidic 1.0 M H<sub>2</sub>SO<sub>4</sub> And 3.0 H<sub>2</sub>SO<sub>4</sub> concentration; the improvements found were up to 38 % in splitting tensile strength and 9 % in E<sub>cm</sub>;
- The wedge split test method may be a suitable way to evaluate the energy absorption capacity of RCA under fracture, reducing the effect of the sample's own weight on the fracture test results; these showed that there is an improvement when RCA is treated with HCl at 1 M and 3.0 M, with a reduction of 16 % and 14.77 % compared to RC; the vertical force *versus* COMD curves for mixes with treated RCA showed a greater ductility than the NA mixes showing that the incorporation of RCA can be an alternative to the use of fibres presented by some authors to increase ductility; this can be attributed to the rough texture and angular shape of RCA compared to NA, which has led to better interlocking and shear resistance. Therefore, the weakness of the interface bond between treated RCA and RCA treated and cement paste may slightly offset the shear performance.

This article intends to highlight how improving the removal of mortar from recycled concrete aggregates (RCA), using acid solutions and the addition of aluminium sulphate, has a positive impact on mechanical properties and durability. The results indicate that lower concentrations are suitable for this method, demonstrating that higher concentrations, in addition to requiring greater acid consumption, do

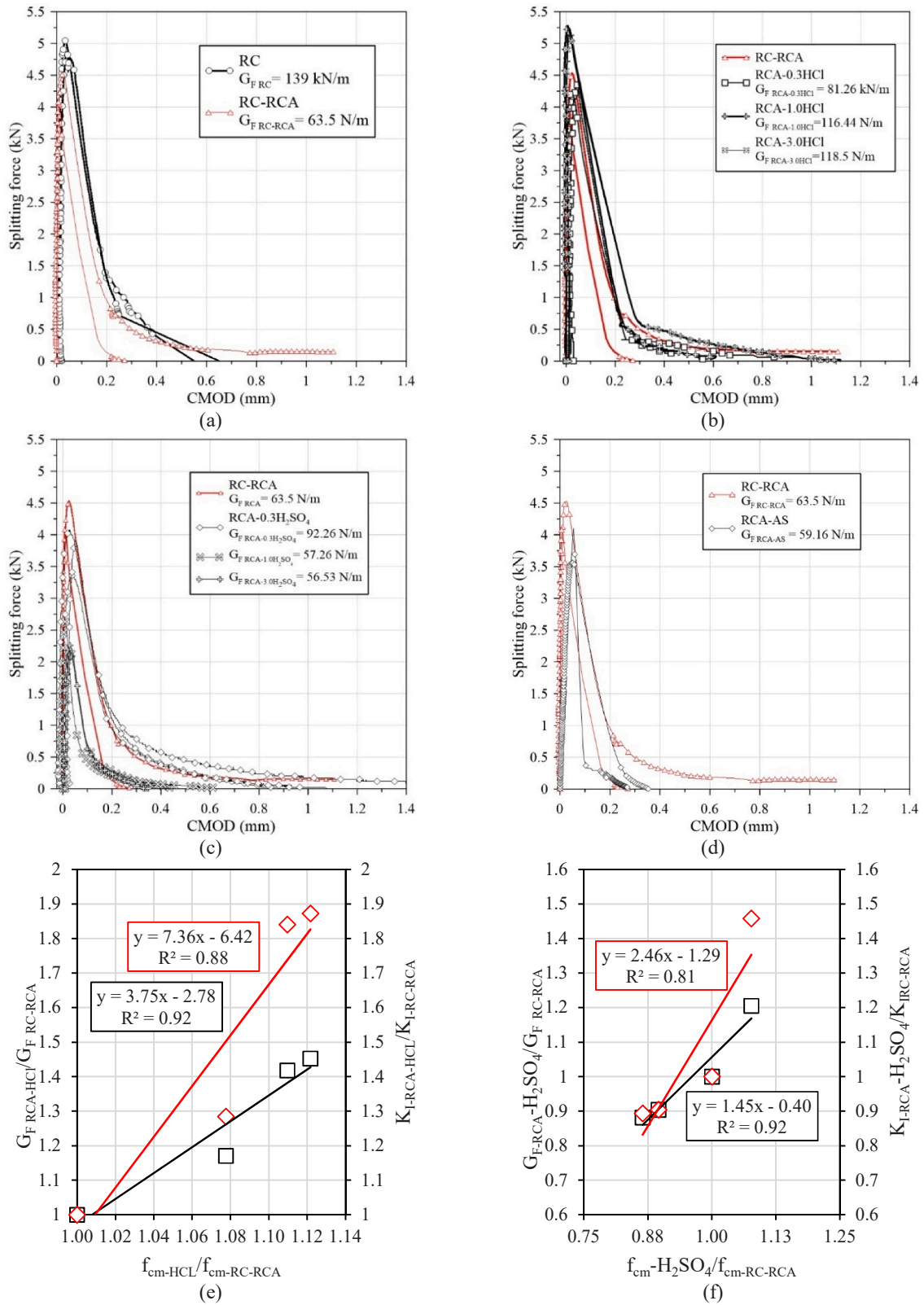


Fig. 10. - Splitting force versus CMOD curves for mixes: RC, RC-RCA (a), RCA-0.3HCl, RCA-1.0HCl and RCA-3.0HCl (b), RCA-0.3  $\text{H}_2\text{SO}_4$ , RCA-1.0  $\text{H}_2\text{SO}_4$  and RCA-3.0  $\text{H}_2\text{SO}_4$  (c), RCA-AS (d); normalised relationships between compressive strength ( $f_{cm}$ ), fracture energy ( $G_F$ ) and intensity factor ( $K_I$ ) of concrete mixes with RCA treated with HCl (e);  $\text{H}_2\text{SO}_4$  (f).

not provide a significant difference. With regard to aluminium sulphate, its addition proves to be a viable solution, since this component can be reused from various water flocculation processes present in water treatment plants

### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

### Data Availability

Data will be made available on request.

### Acknowledgements

The authors gratefully acknowledge the support of CERIS (Civil Engineering Research and Innovation for Sustainability) and FCT (Foundation for Science and Technology).

### References

- [1] IEA, Global Energy Review: CO2 Emissions in 2021-Global Emissions Rebound Sharply to Highest Ever Level, International Energy Agency Paris, France, 2022
- [2] A. Bordelon, V. Cervantes, J.R. Roesler, Fracture properties of concrete containing recycled concrete aggregates, *Mag. Concr. Res.* 61 (9) (2009) 665–670.
- [3] F. Kazemian, H. Rooholamini, A. Hassani, Mechanical and fracture properties of concrete containing treated and untreated recycled concrete aggregates, *Constr. Build. Mater.* 209 (2019) 690–700.
- [4] C.S. Poon, Z. Shui, L. Lam, Effect of microstructure of ITZ on compressive strength of concrete prepared with recycled aggregates, *Constr. Build. Mater.* 18 (6) (2004) 461–468.
- [5] L. Evangelista, J. de Brito, Concrete with fine recycled aggregates: a review, *European, J. Environ. Civ. Eng.* 18 (2) (2014) 129–172.
- [6] A. Sidorova, E. Vazquez-Ramonich, M. Barra-Bizinotto, J.J. Roa-Rovira, E. Jimenez-Pique, Study of the recycled aggregates nature's influence on the aggregate–cement paste interface and ITZ, *Constr. Build. Mater.* 68 (2014) 677–684.
- [7] R. Silva, R. Neves, J. de Brito, R. Dhir, Carbonation behaviour of recycled aggregate concrete, *Cem. Concr. Compos.* 62 (2015) 22–32.
- [8] J. García-González, T. Barroqueiro, L. Evangelista, J. de Brito, N. De Belie, J. Morán-del Pozo, A. Juan-Valdés, Fracture energy of coarse recycled aggregate concrete using the wedge splitting test method: influence of water-reducing admixtures, *Mater. Struct.* 50 (2) (2017) 120.
- [9] S. Ishiguro, S. Stanzl-Tschegg, E. Tschegg, R. Travnicek, Mode I fracture behaviour of recycled concrete, *Fract. Mech. Concr. Structures, ProceediRgs FRAMCOS 2* (1995) 145–154.
- [10] A. Carpinteri, A. Ingrassia, Z. Bazant, Fracture Mechanics of Concrete: Material Characterization and Testing, (1986).
- [11] Z. Li, Advanced Concrete Technology, John Wiley & Sons, 2011.
- [12] V.W.Y. Tam, C.M. Tam, K.N. Le, Removal of cement mortar remains from recycled aggregate using pre-soaking approaches, *Resour. Conserv Recy* 50 (1) (2007) 82–101.
- [13] S. Ismail, M. Ramli, Engineering properties of treated recycled concrete aggregate (RCA) for structural applications, *Constr. Build. Mater.* 44 (2013) 464–476.
- [14] R. Purushothaman, R.R. Amirthavalli, L. Karan, Influence of treatment methods on the strength and performance characteristics of recycled aggregate concrete, *J. Mater. Civ. Eng.* 27 (5) (2014) 04014168.
- [15] P. Saravanakumar, K. Abhiram, B. Manoj, Properties of treated recycled aggregates and its influence on concrete strength characteristics, *Constr. Build. Mater.* 111 (2016) 611–617.
- [16] S. Ismail, M. Ramli, Mechanical strength and drying shrinkage properties of concrete containing treated coarse recycled concrete aggregates, *Constr. Build. Mater.* 68 (2014) 726–739.
- [17] K. Scrivener, J.F. Young, Mechanisms of chemical degradation of cement-based systems, CRC Press, 2014.
- [18] S. Sarkar, S. Mahadevan, J. Meeussen, H. Van der Sloot, D. Kosson, Numerical simulation of cementitious materials degradation under external sulfate attack, *Cem. Concr. Compos.* 32 (3) (2010) 241–252.
- [19] J.A. Forero, J. de Brito, L. Evangelista, C. Pereira, Improvement of the Quality of Recycled Concrete Aggregate Subjected to Chemical Treatments: A Review, *Materials* 15 (8) (2022) 2740.
- [20] S. Chatterji, J. Jeffery, Studies of early stages of paste hydration of different types of portland cements, *J. Am. Ceram. Soc.* 46 (6) (1963) 268–273.
- [21] C.Y. Kan, M.Z. Lan, L.M. Kong, J.B. Yang, Effect of aluminium sulfate on cement properties. *Materials Science Forum*, Trans Tech Publications Ltd, 2013, pp. 285–291.
- [22] Y. Wang, X. He, Y. Su, B. Ma, J. Chu, B. Jiang, Effect of aluminium phases on thaumasite formation in cement slurries containing limestone powder, *Mag. Concr. Res.* 70 (12) (2018) 610–616.
- [23] L.G. Briendl, F. Mittermayr, R. Röck, F.R. Steindl, M. Sakoparnig, J. Juhart, F. Iranshahi, I. Galan, The hydration of fast setting spray binder versus (aluminum sulfate) accelerated OPC, *Mater. Struct.* 55 (2) (2022) 74.
- [24] I.G. Braz, M.C. Shinzato, T.J. Montanheiro, T.M. de Almeida, F.M. de Souza Carvalho, Effect of the addition of aluminum recycling waste on the pozzolanic activity of sugarcane bagasse ash and zeolite, *Waste Biomass-- Valoriz.* 10 (2019) 3493–3513.
- [25] X. Song, P. Qiao, H. Wen, Recycled aggregate concrete enhanced with polymer aluminium sulfate, *Mag. Concr. Res.* 67 (10) (2015) 496–502.
- [26] M. Casuccio, M. Torrijos, G. Giaccio, R. Zerbino, Failure mechanism of recycled aggregate concrete, *Constr. Build. Mater.* 22 (7) (2008) 1500–1506.
- [27] M. Gesoglu, E. Güneysi, H.Ö. Öz, I. Taha, M.T. Yasemin, Failure characteristics of self-compacting concretes made with recycled aggregates, *Constr. Build. Mater.* 98 (2015) 334–344.
- [28] E. Ghorbel, G. Wardeh, H. Fares, Mechanical and fracture properties of recycled aggregate concrete in design codes and empirical models, *Struct. Concr.* 20 (6) (2019) 2156–2170.
- [29] E. Ghorbel, G. Wardeh, Influence of recycled coarse aggregates incorporation on the fracture properties of concrete, *Constr. Build. Mater.* 154 (2017) 51–60.
- [30] H. Linsbauer, E. Tschegg, Fracture energy determination of concrete with cube-shaped specimens, *Zem. und Beton* 31 (1) (1986) 38–40.
- [31] E. Brühwiler, F. Wittmann, The wedge splitting test, a new method of performing stable fracture mechanics tests, *Eng. Fract. Mech.* 35 (1-3) (1990) 117–125.
- [32] S. Khalilpour, E. BaniAsad, M. Dehestani, A review on concrete fracture energy and effective parameters, *Cem. Concr. Res.* 120 (2019) 294–321.
- [33] M. Guo, S.Y. Alam, A.Z. Bendimerad, F. Grondin, E. Rozière, A. Loukili, Fracture process zone characteristics and identification of the micro-fracture phases in recycled concrete, *Eng. Fract. Mech.* 181 (2017) 101–115.
- [34] NP EN 12620:2002+A1: Aggregates for concrete, IPQ, Caparica Portugal 2010, p. 61.
- [35] C.D. EU, 98/83/EC of 3 November 1998 on the quality of water intended for human consumption, Official Journal of the European Communities (1998).
- [36] A. Akbarnezhad, K. Ong, M. Zhang, C. Tam, Acid treatment technique for determining the mortar content of recycled concrete aggregates, *J. Test. Eval.* 41 (3) (2013) 441–450.
- [37] J. Faury, Le béton, 3ème édition ed., Paris, 1985.
- [38] NP EN 206:2013+A2: Concrete Specifications, Performance, Production and Conformity, IPQ, Caparica Portugal, 2021, p. 102.
- [39] J. de Brito, N. Saikia, Recycled aggregate in concrete: use of industrial, construction and demolition waste, Springer Science & Business Media, 2012.
- [40] H. Cui, X. Shi, S.A. Memon, F. Xing, W. Tang, Experimental study on the influence of water absorption of recycled coarse aggregates on properties of the resulting concretes, *J. Mater. Civ. Eng.* 27 (4) (2014) 04014138.
- [41] S.C. Angulo, P.M. Carrijo, A.D. Figueiredo, A.P. Chaves, V. John, On the classification of mixed construction and demolition waste aggregate by porosity and its impact on the mechanical performance of concrete, *Mater. Struct.* 43 (4) (2010) 519–528.
- [42] NP EN 12350-2: Testing fresh concrete - Part 2: Slump test, IPQ, Caparica Portugal, 2019, p. 8.
- [43] NP EN 12390-3: Testing hardened concrete - Part 3: Compressive strength of test specimens, IPQ, Caparica Portugal 2019, p. 20.
- [44] NP EN 12390-6, Testing hardened concrete - Part 6: Tensile splitting strength of test specimens, IPQ, Caparica Portugal, 2023, p. 15.
- [45] E. LNEC, 397, "Betões: determinação do módulo de elasticidade em compressão", LNEC Lib. (1993).
- [46] NT BUILD 511: Wedge splitting test method (WST): Fracture testing of fibre-reinforced concrete (Mode I), Nordic Innovation Centre, Norway, 2005.
- [47] S. Kumar, S.V. Barai, Concrete fracture models and applications, Springer Science & Business Media, 2011.
- [48] NP EN 197-1: Cement - Part 1: Composition, specifications and conformity criteria for common cements, IPQ, Caparica Portugal 2012, p. 39.
- [49] R. Silva, J. de Brito, R. Dhir, The influence of the use of recycled aggregates on the compressive strength of concrete: a review, *European, J. Environ. Civ. Eng.* 19 (7) (2015) 825–849.
- [50] J. Xiao, W. Li, Z. Sun, D.A. Lange, S.P. Shah, Properties of interfacial transition zones in recycled aggregate concrete tested by nanoindentation, *Cem. Concr. Compos.* 37 (2013) 276–292.
- [51] A. Kabir, N.U. Ahmed, M.S. Alam, Effect of cement proportion on strength of recycled aggregate concrete, *Int. J. Struct. Civ. Eng. Res.* 4 (4) (2015) 341–346.
- [52] K. Pandurangan, A. Dayanithy, S.O. Prakash, Influence of treatment methods on the bond strength of recycled aggregate concrete, *Constr. Build. Mater.* 120 (2016) 212–221.
- [53] Y. Kim, A. Hanif, S.M.S. Kazmi, M.J. Munir, C. Park, Properties enhancement of recycled aggregate concrete through pretreatment of coarse aggregates - Comparative assessment of assorted techniques, *J. Clean. Prod.* 191 (2018) 339–349.
- [54] A.J. Tang, R. De Jesus, A. Nunanan, Microstructure and mechanical properties of concrete with treated recycled concrete aggregates, *Int. J.* 16 (57) (2019) 21–27.
- [55] M.S. De Juan, P.A. Gutiérrez, Study on the influence of attached mortar content on the properties of recycled concrete aggregate, *Constr. Build. Mater.* 23 (2) (2009) 872–877.

- [56] A. Abbas, G. Fathifazi, O.B. Isgor, A.G. Razaqpur, B. Fournier, S. Foo, Proposed method for determining the residual mortar content of recycled concrete aggregates, *J. ASTM Int.* 5 (1) (2007) 1–12.
- [57] H.K.A. Al-Bayati, P.K. Das, S.L. Tighe, H. Baaj, Evaluation of various treatment methods for enhancing the physical and morphological properties of coarse recycled concrete aggregate, *Constr. Build. Mater.* 112 (2016) 284–298.
- [58] A. Allahverdi, F. ŠKVÁRA, Acidic corrosion of hydrated cement based materials, *Ceramics–Silik. áty* 44 (4) (2000) 152–160.
- [59] R.E. Beddoe, H.W. Dorner, Modelling acid attack on concrete: Part I. The essential mechanisms, *Cem. Concr. Res.* 35 (12) (2005) 2333–2339.
- [60] J.R. Clifton, J. Pommersheim, Sulfate attack of cementitious materials: volumetric relations and expansions, NIST, IR, 1994.
- [61] T.L. Brown, H.E. LeMay Jr, B.E. Bursten, J.R. Burdge, *Química: la ciencia central*, Pearson educación 2004.
- [62] M. Etxeberria, E. Vázquez, A. Mari, M. Barra, Influence of amount of recycled coarse aggregates and production process on properties of recycled aggregate concrete, *Cem. Concr. Res.* 37 (5) (2007) 735–742.
- [63] S.W. Tabsh, A.S. Abdelfatah, Influence of recycled concrete aggregates on strength properties of concrete, *Constr. Build. Mater.* 23 (2) (2009) 1163–1167.
- [64] D. Pedro, J. de Brito, L. Evangelista, Influence of the use of recycled concrete aggregates from different sources on structural concrete, *Constr. Build. Mater.* 71 (2014) 141–151.
- [65] J. Thomas, N.N. Thaickavil, P. Wilson, Strength and durability of concrete containing recycled concrete aggregates, *J. Build. Eng.* 19 (2018) 349–365.
- [66] H. Beshr, A. Almusallam, M. Maslehuddin, Effect of coarse aggregate quality on the mechanical properties of high strength concrete, *Constr. Build. Mater.* 17 (2) (2003) 97–103.
- [67] P.-C. Aitcin, P.K. Mehta, Effect of coarse aggregate characteristics on mechanical properties of high-strength concrete, *Mater. J.* 87 (2) (1990) 103–107.
- [68] NP EN 1992-1:2010: Eurocode 2: Design of concrete structures-part 1–1: general rules and rules for buildings, British Standard Institution, London, 2005, pp. 659–668.
- [69] X. Liu, B. Ma, H. Tan, B. Gu, T. Zhang, P. Chen, H. Li, J. Mei, Effect of aluminum sulfate on the hydration of Portland cement, tricalcium silicate and tricalcium aluminate, *Constr. Build. Mater.* 232 (2020) 117179.
- [70] J. Han, K. Wang, Y. Wang, J. Shi, Study of aluminum sulfate and anhydrite on cement hydration process, *Mater. Struct.* 49 (4) (2016) 1105–1114.