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Durability of rubberized concrete with recycled steel fibers from tyre recycling in aggresive enviroments

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ABSTRACT

An assessment of the chemical and physical behavior of rubberized concrete (RuC) during natural carbonation exposure, freeze–thaw degradation and acid degradation (sulfuric and acetic acid ($C_2H_4O_2$)) with two types of crumb rubber aggregates, is presented: crumb rubber (CR) containing only rubber; and fibers partially coated with crumb rubber (FCR). The mixtures studied with rubberized concrete reach the point of total substitution of stone aggregate (0/20/40/60/80/100 vol%). To analyze and understand the behavior of concrete, physical and mechanical properties have been also studied and related. Weight loss and ultrasonic pulse test were done throughout 3 months in rubberized concrete under accelerated attack, and strength variation recorded after the tests. As well, Scanning Electron Microscope images have been studied after the degradation processes tested. The use of CR and FCR reduces mechanical strength and increases porosity, but rubberized concrete with CR aggregates presents lower carbonation when volume is over 80% and strength reductions stability improves when FCR are used in freeze–thaw attack and under acid corrosion environments, as fibers improve matrix cohesion. Moreover, effectiveness of Ultrasonic Pulse Velocity (UPV) as a method to assess the stages of the attack and final resistance estimation is also indicated after the research results.

1. Introduction

Nowadays, sustainability and environmentally friendly strategies in construction industry are getting more attention as the weather climate change is being more noticeable [1,2], with new policies ruling the construction and building sector [3]. In the building industry, in which cement and steel production together are responsible of the 10% of all global Greenhouse gases (GHG) emissions [4] some of these strategies must be focused throughout the stability and long-term durability of materials, the use of low carbon-print materials, and the re-use and revalorization of materials [5]. These strategies are also enhanced when these new composites present low unit-weight, are thermal and acoustically isolators presenting bioclimatic principles [5]. Moreover, the building sector accounts for 40% of energy consumption and generates 25% of solid waste [6].

The use of lightweight recycled aggregates (LWRA) in concrete [7] reduces unit weight of concrete. Therefore, the load and stress on structures are also reduced by decreasing the reinforcement spenditure (usually steel) or by reducing the dimensions of beams or pillars of the structure. In addition, thermal conductivity and some acoustical properties and dumping properties can be also improved. Nevertheless, LWRA when used as concrete aggregates, reduce mechanical resistance, and exhibit different internal transport properties of concrete, and therefore concrete durability can be compromised.

On the one hand, according to their composition, LWRA can be organic [8] or inorganic [9], but also manufactured (from raw or discarded/composites materials) or natural from quarries [10]. The LWRA studied here are two types of recycled rubber from end-of-life tires, but from different recycling process: Crumbed Rubber (CR) and Fiber with Crumbed Rubber (FCR) [11]. On the other hand, metallic fibers (mainly steel made) can be from manufacture or from tire recycling [10–12]. Cement-based mixtures with these recycled fibers and aggregates have been studied to be included in the manufacture chain to increase their sustainability [10]. They have been used as reinforcement in Rubberized Concrete (RuC) for mechanical enhancement, especially ductility and toughness in slabs under flexure [13,14], fatigue and cracking control of

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concrete under dynamic loads [15], but also for early cracking control [11]. The addition of fibers also helps to volume stability of RuC, reducing cracking due to expansion during attack processes [16,17] but also reducing cracking due to drying shrinkage. In this research as in others, fibers also reduce strength loss after attack, even when weight loss is more accused [18] Moreover, reducing particle size of crumb rubber increase RuC freeze–thaw resistance [19], and carbonation resistance [13,16,20], observed improved flexural behaviour in RuC with a 20% of rubber aggregates, and steel fibers and silica fume. Silica fume in RuC, minimized the impact of rubber aggregates on the chloride permeability. RuC with fibers may also exhibit, when added in high volume fractions, a poor dispersion and low workability, leading to more entrapped air during mixing, although also improved durability of RuC [12]. Moreover, RuC with volume substitutions of stone aggregate up to 100% of CR or FCR has been studied to facilitate the understanding of the possibilities of rubber aggregates in construction materials. FCR is obtained in a simpler and coarser recycling process than CR, and less energy consumption is needed during the recycling process itself. Although, FCR are partially coated with rubber, the recovered steel fiber can improve the mechanical behaviour of RuC as studied previously [21]. RuC with volume of rubber aggregates over 40%, although has low resistance for structural proposes, it can be use in several construction material such as insulator in roofs, flooring, etc. [21]. Durability of RuC is in this research also studied, to follow one of the principles of sustainability and environmentalism of building industry, the durability of the materials even when revalued materials are used.

CR modifies the physical properties of concrete with a reduction of workability from the fresh state [22]. Therefore, it has been found that the casting process is more difficult and air-void content in concrete is increased [22–25,37] and therefore the total porosity [16]. Moreover, the hydrophobic surface and jagged shapes of CR trends to entrap air during the mixing process of RuC [27] and a weaker composition of the Interface Transition Zone between CR aggregate and cement paste compared to stone aggregate has been observed [20–22,22–28]. Another process has been reported in the literature when high amounts of CR are used, ocurring a poor cementation and an increase of permeability channels and water absorption [29]. During the curing process, shrinkage is also increased with the use of larger volume of CR [23,30–32].

The above physical processes lead RuC to a more permeable composite [22,26], what eases the transport of water and aggressive compounds through capillary and pore network into concrete [32–34]. Hence, attack processes such as carbonation, acid attack and freeze–thaw degradation in concrete can be accelerated. Contradictory results have been found in the literature about the influence of rubber particle size, some authors have reported that the higher the size the lower the water permeability [27], and other set a maximum size of 0.5–5 mm [35]. However, water absorption of RuC can be reduced through the pretreatment of rubber aggregates -with NaOH, or with KMnO4- or with cement slurry, as a bond between treated rubber aggregates and cement hydration products is obtained. [28]. Attending to the microstructure, ITZ is affected by microcracking on the boundary which can be reduced with nano-silice and silanized rubber [36]. Fillers with pozzolanic action have also shown an improvement of durability of RuC, thanks to a reduction of water penetration and to the development of a less porous and more homogeneous ITZ [37]. ITZ is thicker for RuC has been observed to be up to three times than for conventional concrete [38], but ITZ width can be reduced when rubber is treated with NaOH and metakolin, also obtaining a more fine pore structure, increasing durability of RuC [39].

Carbonation is caused by the combination of carbon dioxide from the atmosphere with the portlandite in the cement paste (eq. (1). The carbonation process leads to a reduction of the pH of concrete, what eases the corrosion of steel reinforcement involved by the cement mass. Service life of reinforced concrete can be improved, as steel rods are not easily attacked when the carbonation process is delayed, through the increase of the amount of cement and thus portlandite reserves or by a less permeable microstructure of concrete [20].

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \tag{1}$$

Carbonation itself do not affect the resistance of concrete, but when the bar is corroded, the corrosion products can crack and disrupt its surrounding concrete. Rubberized concrete can be used in samples reinforced, and carbonation has been studied by several authors. Trilok et al. observed an increase of carbonation depth in mixtures with 20% replacement of aggregate in samples with low w/c ratio (0.35), but a reduction in samples with 0.55w/c ratio [22]. Several authors also observed a higher carbonation depth rates of rubberized concrete compared to conventional concrete [33,41,42]. However, Blessen Skariah Thomas studied a trend change with the volume of rubber aggregates above 12.5% volume replacement, with a stabilization of the results [41], and Jihen Mallek indicated that rubber promoted the resistance to carbonation [43]. Rubber content, particle size and w/c ratio played an important role in the results obtained, according to the authors studied [27,41,44–46]. Nevertheless, it has not been found results in the literature of RuC with fibers.

Disruption of concrete can also occur due to the increase in volume of free water upon freezing or the formation of new chemical components, which creates internal stresses and, subsequently, cracking and degradation. Freeze-thaw process has been studied in plain and rubberized concrete, observing several authors an improvement when rubber aggregates are used [16,17,33,35,47–49]. Freeze-thaw durability performance is also observed in RuC with a pretreatment of the surface of rubber aggregates to improve interfacial transition zones (ITZ) with the cement matrix [23,50], with pozzolan addition, such as silica fume [36,51] and with fiber reinforcement [30]. It is mentioned that rubber aggregates can relieve the pressure of ice crystallization, what reduces the damage of the matrix [52].

Some unstable chemical phases from cement that increase their volume need to be combined with other chemicals, such as sulphate acids. Sulphury acid attack compared to sulphate attack is more active, as it presents a dissolution effect by the hydrogen ions combined to the attack by sulphate ions [41]. Ettringite is a point-shape crystal which decays concrete when formed after concrete hardening. When concrete presents a high amount of free sulphate after hardening, in its mass or it is introduced from the surface to the internal mass, it can be combined with celite or free calcium aluminate [20]. Corrosion of cementitious composites under sulfuric acid attack can be characterized by the following reactions [34].

$$Ca(OH)_2 + H_2SO_4 \rightarrow CaSO_4 \cdot H_2O \tag{2}$$

$$CaSiO_2 \cdot 2H_2O + H_2SO_4 \rightarrow CaSO_4 + Si(OH)_4 + H_2O \tag{3}$$

$$\begin{aligned} &3CaO \cdot Al_2O_3 \cdot 12H_2O + 3(CaSO_4 \cdot 2H_2O) + 14H_2O \\ &\rightarrow 3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O(Tricalciumaluminate) \\ &+ (Water) + (Gypsum) \rightarrow (Ettringite) \end{aligned} \tag{4}$$

The use of rubber aggregates to mitigate the corrosion of concrete under acid and sulphate attacks has been reported in the literature [30,31,33,41]. Li Y. et al, affirmed that rubber aggregates, which are elastic, absorbed the energy released in the sulphate attack by decreasing expansion and reducing cracking [27]. Blessen Skariah Thomas indicates an optimum rubber volume of a 20%, and A. Yung indicates that only a volume of 5% RC increases durability to sulphate attack by 15% [41,44,53]. Other authors also used pozzolanic additions, such as metakaolin, fly ash and silica fume in RuC to further improve the RuC sulphate resistance [18,28,51,54].

Concrete used in agro-industrial applications (wine industry, sugar industry, dairy products industry or animal rearing) may be attacked by waste water containing organic acids, such as acetic acid [39]. Acetic acid reacts with cement hydration products to form calcium acetate as follows [20]:

$$2CH_3COOH + Ca(OH)_2 \rightarrow Ca(CH_3COO)_2 + 2H_2O \quad (5)$$

$$2CH_3COOH + C-S-H \rightarrow SiO_2 + Ca(CH_3COO)_2 + 2H_2O \quad (6)$$

However, the use of rubber aggregates to mitigate the corrosion of concrete under acetic acid attack has not been found in the literature by the authors. Moreover, the durability assessment of concrete with high volume of rubber aggregates, rubber aggregates with fibers, specially recycled fibers, is also novelty in the literature as far as the authors know.

2. Materials and methods

2.1. Materials

Portland cement with developed early strength resistance of 42,5 MPa (CEM I 42,5 R) was used. Its main properties are as follows: 3.18 specific gravity; 3750 cm^2/g Blaine fineness, 90.25% weight of clinker, 4.75% weight of calcareous filler and 5 % weight of gypsum. Its initial and final setting time are 170 min and 220 min respectively (UNE-EN 196–3).

Two siliceous aggregates were used in mixes, sand from river banks (0–4 mm), and rolling gravel (4–8 mm) and their nominal density was 2.61 g/cm^3. To compare the chemical properties of the mixes, no additives were used.

CR and FCR of 4–8 mm size from end-of-life tires were used. CR is usually commercialized in the concrete industry and steel and plastic fibers (5–30 mm length) coated with crumb rubber FCR (Fig. 1). CR follows European Normative: Final Draft FRCEN/TS 1424 Materials produced from end-of-life tires –Specification of categories based on their dimension and impurities and methods for determining their dimension and impurities. FCR in concrete has been previously studied by the authors [11,21]. FCR aggregates present steel and fabric fibers partially coated with rubber and rubber crumb aggregate, as a process of recycling without total separation and recovery of raw materials from tires. FCR main components are 50.35 % weight of CR aggregates, 27.00% CR bonded to textile fibers, 9.43% CR bonded to steel fiber, 5.81% of CR linked to both fibers, and 5.04% steel and 2.07% plastic fibers [21]. The nominal density was determined with an Helium Stereopycnometer of Quantachrome, being for CR 0.90 g/cm^3, and for FCR 1.10 g/cm^3. Composition and mix proportion of mixtures are presented in Table 1.

2.2. Test methods

2.2.1. Physical and mechanical test

Reference concrete with nominal compressive strength of 45 MPa was used, with w/c ratio of 0.5 and without water reducer. Consistency of mixes was determined using the Slump test in concrete with CR, according to code UNE-EN 12350–3:2020 [Testing fresh concrete - Part 2: Slump test] and with the Vebe test in concrete with FCR according to code UNE-EN 12350–3:2020 [Testing fresh concrete - Part 3: Vebe test].

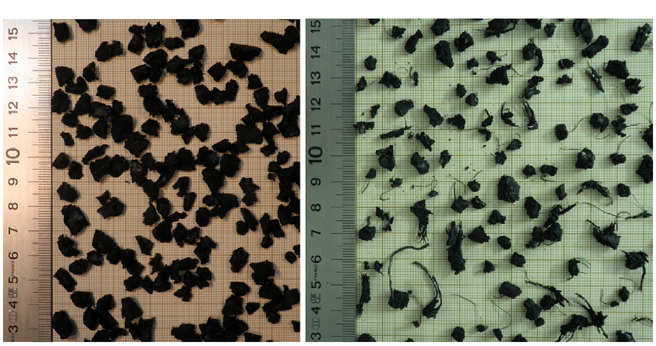

Fig. 1. CR and FCR used as raw material.

Reference concrete was modified with a gravel volume substitution of 20% to 100% with CR and FCR separately. Composition of mixtures is presented in Table 2. For testing mixtures were casted in different molds: 10 × 10 × 10 cm^3 cubic and 20 × (diameter)10 cm^3 cylindrical, 30 × (diameter)15 cm^3 cylindrical. Specimens were cured for 7 days under water according to UNE-EN 12390–2 and stored until testing under laboratory conditions (20 ± 2 °C and 65 ± 5 % RH).

Compression strength and three-point bending strength were determined according to UNE-EN 12390–4:2020 [Testing hardened concrete - Part 4: Compressive strength - Specification for testing machines] and to UNE-EN 12390–5:2020 [Testing hardened concrete - Part 5: Flexural strength of test specimens] respectively. Non-destructive ultrasonic pulse velocity tests (UPVT) were performed on the prismatic specimens, at 28 days, determining the ultrasonic modulus (Es) [21] Bulk density and bulk porosity were calculated by EN 1015–https://doi.org/10:1999/A1:2006 standard [Methods of test for mortar for masonry - Part 10: Determination of dry bulk density of hardened mortar].

2.2.2. Carbonation

Carbonation of concrete was determined in cylindrical samples (20x10cm^3) stored during 5 years in external conditions nearby the laboratory of the Tehcnical University of Madrid. These samples were exposed to rain and weather conditions, with temperatures varing from −5°C up to 42 °C. After these periods, the samples were cut into pieces under indirect tension through brazilian test using a hydraulic press, and inmediately sprayed with phenolphthalein following the process described in UNE 112011:2011 [Corrosion of concrete reinforcement steel. Determination of the carbonatation depht for in-service concrete]. Fig. 2 shows the samples after sprayed with phenolphthalein indicator, showing in magent the non carbonated areas.

2.2.3. Acid attack

Hardened 10x10x10cm^3 samples were immersed in two different basins with acid baths, one with acetic acid and another one with sulfuric acid. Fig. 3 shows the disposition of the samples before the bath and the section with the separation measures kept during the time the samples were tested. Disposition of samples allowed the acid to attack in all the faces of the sample homogeneously. Both acids were prepared through the dilution of 3% of each acid, up to a pH of 3, wich was stabilized after every measurement. After several days of immersion, the samples were dried in an oven at 30 °C, and their dry weight and ultrasonic pulse velocity (UPV) recorded before being immersed again, up to a period of 3 months. UPV measurement was performed after every acid immersion and drying cycle. It was made to assess the degradation of concretes in compliance with UNE-EN 12504–4:2006 [Testing concrete – Part 4: Determination of ultrasonic pulse velocity], The reduction of UPV indicates a lower density or continuity of the mass [23,30], that can be caused by the acid attack. Moreover, after the whole process of the acid immersion, the compression of the samples was also determined to asses the degradation of the mixtures. Besides, the miscrostructure of some samples, was studied after the attack using Scanning Electron Microscopy - Energy Dispersive X-ray spectroscopy (SEM-EDX). The mineralogical composition of residual products observed in the ITZ of rubber aggregates after acetic acid attack were determined by X-ray diffraction (Philips diffractometer, Netherlands).

2.2.4. Freeze-thawn attack

The freeze–thaw test has been carried out according to UNE-CEN/TS 12390–9:2008 EX [Testing hardened concrete - Part 9: Freeze-thaw resistance – Scaling] on 10 × 10 × 10 cm^3 specimens for a total of 84 freeze–thaw cycles. Samples were immersed in a water bath 24 h to complete hydration before exposed to freeze (Fig. 4). Likewise, UPV and dry weight were measured. At the end of the proccess, the residual compression resistance of the samples was tested.

Table 1
Composition and mix proportion of tested mixtures.

Rubber	**Cement42.5 MPa**	**Aggregate4-8 mm**	**Recycled Rubber4-8 mm**	**SandO-4 mm**	Water
Substitution (CR or **FCR) (% volume)**	(kg/m3)	(kg/m3)	(kg/m3)	(kg/m3)	(kg/m3)
0% (REF. CONCRETE)	360	1103.0	0.0	722.8	180
20%	360	882.4	84.5	722.8	180
40%	360	661.8	169.0	722.8	180
60%	360	441.2	253.5	722.8	180
80%	360	220.6	338.1	722.8	180
100%	360	0.0	422.6	722.8	180

Table 2
Physical and mechanical results of the different mixtures studied before exposure to aggressive testing.

Mixture Reference	**Consistency**	**Shrinkage**	**Bulk Density**	**Bulk Porosity**	**US Modulus**	**Compressive**	**Bending**
	Vebe test (s)	**(mm/m)**	**(lcg/m^3)**	(%)	**(Gpa)**	**Strength (Mpa)**	**Strength (Mpa)**
RC	9,03(7.5)	1,434(3.5)	2422(15.2)	9.04(15.8)	49(7.5)	47.78(12.6)	4.75(14.5)
CR-20	11,07(15.2)	1,204(5.6)	2264(16.2)	8.97(15.9)	42(8.5)	27.71(15.3)	3.75(18.6)
CR-40	9,34(11.2)	1,461(8.8)	2156(17.5)	9.29(18.2)	31(7.7)	17.71 (16.8)	2.90 (17.8)
CR-60	10,6(7.6)	1,186(8.7)	2026(14 5)	9.11(14.8)	27(8.8)	13.58 (19.5)	2.75 (22.5)
CR-80	6,75(9.5)	0,728 (9.7)	1858(18.5)	9.68(18.6)	19(9.1)	8.60(25.5)	2.05 (19.9)
CR-100	8,31(8.6)	1,252(11.2)	1742(21.3)	11.54(20.8)	13(11.3)	6.33(26.8)	1.86(22.6)
FCR-20	10,44(5.8)	1,144(5.9)	2313 (17.5)	9.17(17.6)	44(5.8)	30.09(18.9)	4.30(19.2)
FCR-40	7,32(7.7)	0,574(7.7)	2139(16.4)	1138(16.8)	34)4(8.1)	22.84(19.5)	3.43(21.5)
FCR-60	8,4(9.9)	0,878(9.6)	2032(17.1)	14 M1 (17.9)	28,5 (9.6)	15.82 (20.6)	3.32 (20.3)
FCR-80	18,97(15.8)	0,198(10.1)	1851(18.2)	18.31(17.9)	22(8.7)	9.60(20.5)	3.24(28.5)
FCR-100	24,38(17,1)	0,152(12.8)	1668(19.5)	2137(19.6)	7(12.8)	4.64(28.5)	1.68(22.8)

Fig. 2. Carbonated sections of samples of RuC with and without fibers exposed to outdoor conditions and tested later with phenophtalein indicator (colored parts are not carbonated).

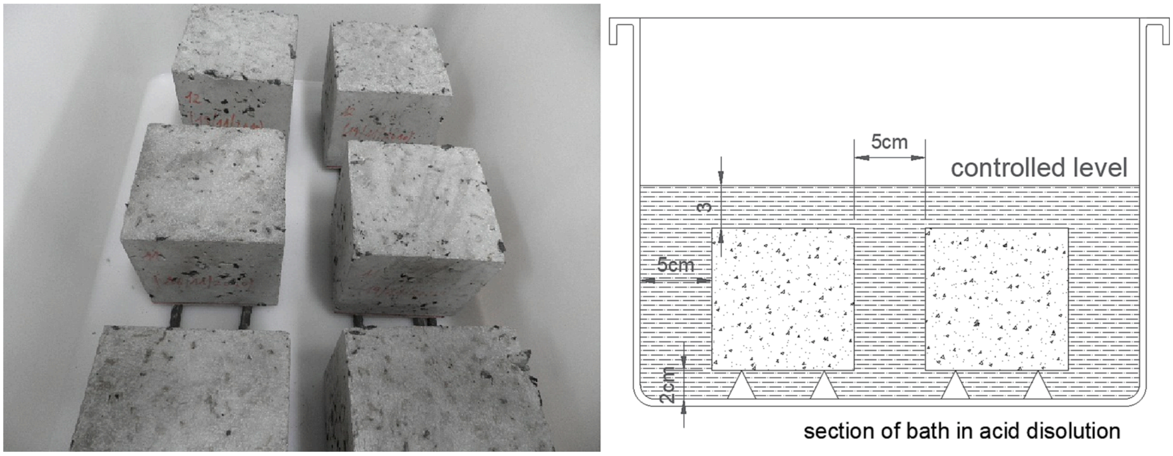

Fig. 3. Disposition of samples during the test with acids indicating the separations between samples.

Fig. 4. Disposition of samples before to be immersed 24 h in water in every cycle, before storage in the freezer.

3. Test results

3.1. Physical and mechanical

Physical and mechanical characterization of the different samples was studied in previous research works [21]. The results are here summarized (Table 1), as they are important in the understanding of the long term behaviour of concrete.

Consistency in all series was dry or very dry, and also presented a reduccion of the bulk density when rubber replaced siliceous aggregate, a 28.1% in samples with 100 %CR and a 31.1% in samples with 100% of FCR. Shrinkage has a noticiable different effect in plain concrete, and RuC with CR or with FCR. The mixtures without rubber aggregate presents a shrinkage of 1.434 mm/m, those with 80% CR aggregate a shrinkage of a 0.728 mm/m, and those with 100% FCR a 0.152 mm/m. The Ultrasonic Modulus (UM) of RuC is 73.5% lower than that of plain concrete, and higher in FCR fiber, to a 85.7%. It can be also noticed that bulk porosity is increased by 27.6% in RuC with CR and 136% with FCR.

Mechanical resistances are also affected with CR and either with FCR. The higher the volume of CR or FCR aggregate, the more noticiable is the mechanical resistance loss. For instance, in RuC mixtures with CR, the most significant reductions in compressive strength, a 86.7% is observed in the mixture with 100% of CR by volume and in those with FCR, with a strength loss of 90.3% when 100% of FCR was added. Bending strength in RuC mixtures is reduced by 60.8% with 100% volume substitution of CR and 64.6 % with 100% volume substitution of FCR (Table 1).

In Figs. 5 and 6, the compression strength (MPa) and strength variation (%) at the end of the different durability tests are presented. In all cases, mixtures with CR presented a final strength, after the different attack processes, lower than that of the RC (Fig. 5). It can be also noticeable that the strength loss after the end of every attack studied is also more pronounced. Altough final strengths are lower when the volume of rubber aggregate is increased,it can be marked that 80 %RC is more stable than 40 %RC and 60 %RC mixtures, as its strenght loss (%) is lower.

In Fig. 6, it can be observed that RuC mixtures with fibers present also a lower compression strength than RC, after the durability tests. The higher the amount of FCR the lower the compression strength. However, the strength loss in 20 %FCR and 40 %FCR is similar to RC, and even in some cases, a gain of strength can be observed after acetic acid attack.

Comparing the strength variation of RuC with or without fibers, it can be observed a more stable behaviour of FCR samples than CR. Specially, in samples with 20% and 40% of CR, the strength loss is clearly lower. After sulfuric acid attack, 20 %FCR mixture presented a strength loss of 24% against a 42% for CR, and 40 %FCR mixture with a strength loss of 18% against 40 %CR mixture with a 56%. In the case of acetic acid attack, the strength gains are 17% for 20 %CR mixture and 32% for 20 %FCR mixture, strength losses of 61% for 40 %CR mixture against a 32% gain for 40 %FCR mixture. After freeze-thawn attack, plain concrete sample reduced its compression strength a 12%, meanwhile 20 %CR mixture presented a strength loss of 38% against 8% for 20 %FCR mixture, and a strength loss of 49% for 40 %CR but a 3% strength gain for 40 %FCR mixture. 60 %FCR mixture also gained stregth (6%), but series with higher FCR volume reduced their resistances.

3.2. Carbonation

Fig. 7 shows the behavior of the different samples with rubber aggregates. A higher carbonation depth can be observed, both medium and maximum, in those samples with FCR. The average depth was increased by 208% and the maximum depth by 153% when FCR is 100% of aggregate. However, in those mixtures with CR, a maximum value for carbonation depth is observed in samples with 60% volume of CR, increasing by 47% the maximum depth and 23% the average depth, which is then progressively reduced in samples with higher volumes, up to a 100% CR. In the case of 80 %vol. CR mixture, a lower carbonation has been recorded than for the plain concrete, with a reduction of 26% for maximum depth and in the case of average depth similar to the plain

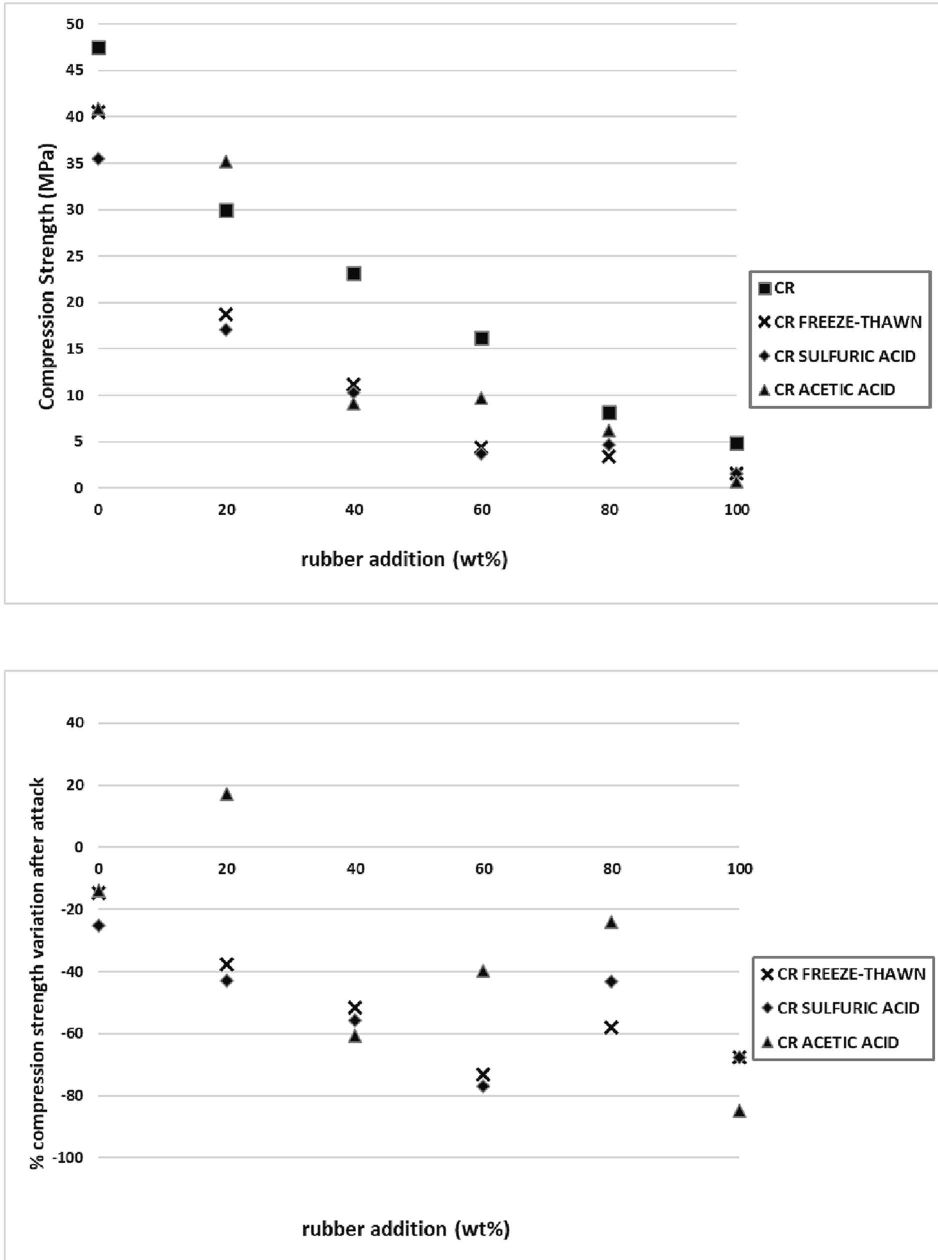

Fig. 5. Compression strength (MPa) and compressive strength variation (%) after the end the different attacks versus the weighted % addition of CR in substitution of stone aggregate.

concrete.

3.3. Acid attack

Acid attack has been assessed through three different tests: weight loss, ultrasonic speed velocity and final strength after acid exposure. Two different acid dilutions have been used to assess the durability of the samples: sulfuric acid and acetic acid. SEM image analysis has been also performed to study the Interfacial Transition Zone (ITZ) of rubber aggregates and the cement matrix after acid exposure as it plays an important role in the mechanical resistance [55].

3.3.1. Sulfuric attack

In Fig. 8 is plotted the weight variation of the different samples with CR exposed to sulfuric acid. A reduction of mass in every sample can be observed, especially in those with low amount of CR (20–40 wt%). It can be noticed that the reference concrete (0 wt% CR), shows the higher mass reduction, up to a 9% after 180 cycles. In the case of RuC, it can be also observed that mixtures with 60 wt% to 100 wt% CR, show a stabilization of mass reduction after 100 cycles. Additionally, it can be also remarkable that they show the lowest mass loss after the tests (180 cycles), a of 5.5% for 60 wt% CR mixture, 3.75% for 80 wt%CR mixture and 3.60% for 100 wt% CR mixture respectively.

Likewise, the weight evolution of the different RuC mixtures with FCR during 180 cycles of sulfuric acid attack have been assessed in Fig. 9. After the process, the samples present a mass loss that follows a similar trend than those with CR. Concrete with FCR as aggregate instead of siliceous aggregate, obtained a better durability under sulfuric acid attack. The values of reference concrete were reduced from a 9% of weight loss to a 2.2% in RuC with 80 wt%FCR mixture, 2.1 with 100 wt %FCR mixture and 1.6 with 60 wt%FCR mixture. A stabilization of the process can be also observed in the 60 wt% FCR mixture after 150 cycles.

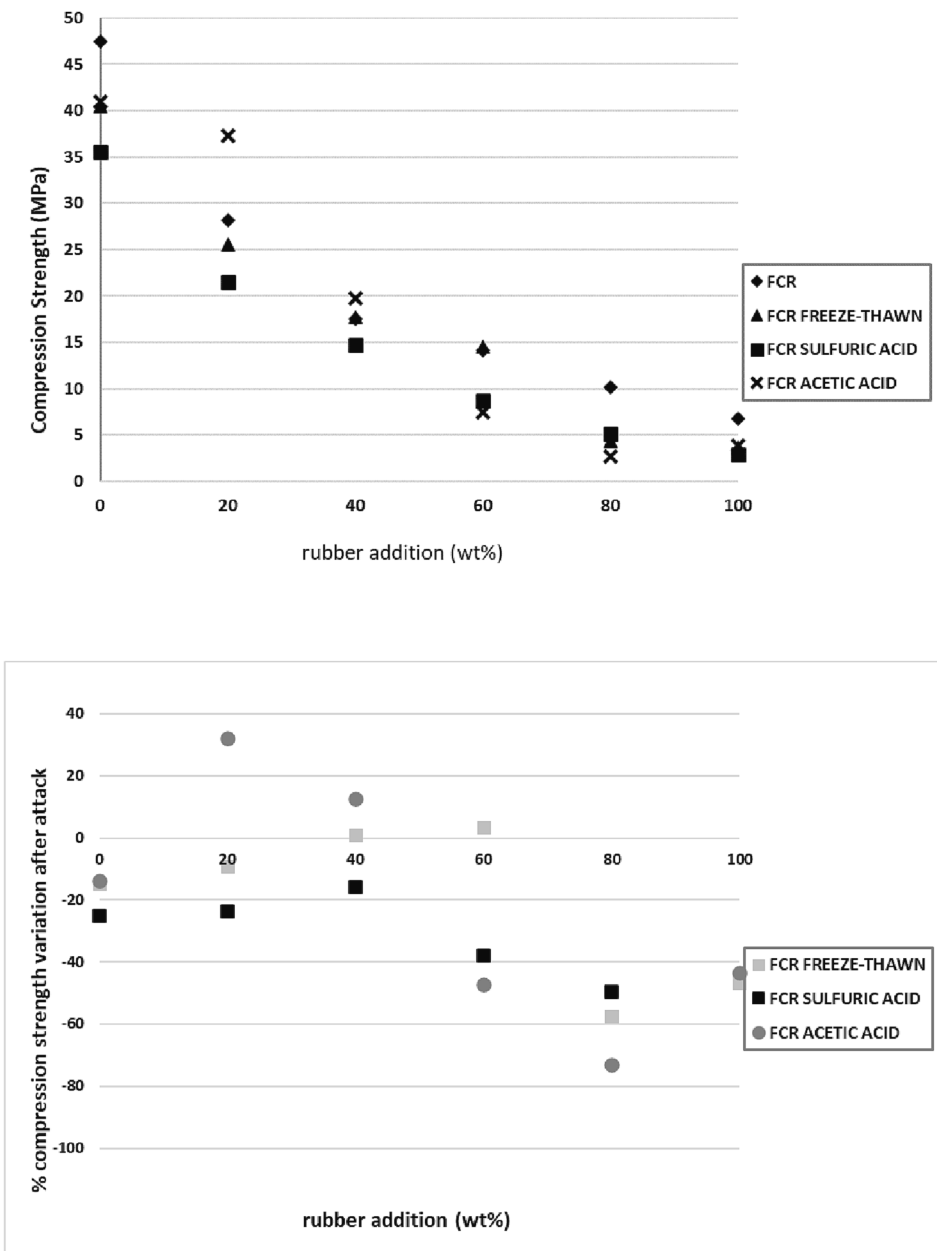

Fig. 6. Compression strength (MPa) and compressive strength variation (%) after the end the different attacks versus the weighted % addition of FCR in substitution of stone aggregate.

As it was afore mentioned, the sulfuric attack has been also assessed using UPV measures of concrete samples summarized and plotted in Figs. 10 and 10. Fig. 10 shows an US speed gain in some of the samples when exposed to sulfuric acid in the first few cycles of the attack. However, every mixture presented a loss of UPV at the end of the test. It is more remarkable in those with high CR weighted percent of aggregate. It can be observed that plain concrete only presented a 5% slower UPV after 180 cycles, unlike with weight loss measurement. However, RuC with CR addition suffered a reduction of the UPV after the process of test, reaching up to 48.5% in 80 wt% FCR mixture.

In Fig. 11, the RuC mixtures with FCR show a similar trend than those without fibers, but the reduction of UPV is lower than for mixtures with CR. In the initial cycles (up to 40 cycles in some cases), an increase of UPV can be noticed. Every RuC with FCR also showed a reduction of UPV at the end of the test. The mixture with the highest UPV reduction, 16.5%, is the one with a 60% volume of FCR, while the one with a 80% volume of FCR, only presented an UPV reduction of 9%. The mixtures with 20 %FCR and 40 %FCR had a slightly higher UPV at the end of the test than plain concrete. In general terms, UPV reduction in concrete samples is less remarkable when rubber with fibers are also added under sulfuric acid attack conditions.

In Fig. 12 SEM images of the samples before attack are shown. In Fig. 12a, we can observe a sample with FCR aggregate before sulfuric acid exposure. In this image, steel fiber and fabric fibers are wrapped in the cement matrix after fracture. Fig. 12b shows an aggregate of CR prior to attack in the vicinity of rubber-cement interface or ITZ, showing full contact but not chemical bonding or adhesion.

Fig. 13 allows to analyze samples with CR aggregate after sulfuric acid exposure. On one hand, we can observe that the ITZ of CR aggregate is not bond to the matrix, which also shows many secondary ettringite needles surrounding the contact area of the matrix with rubber aggregate. Although these new crystals are developed in the cement matrix, the matrix is disrupted in separated flakes without continuity. This can be also observed in RuC with FCR (Fig. 14), where the matrix after

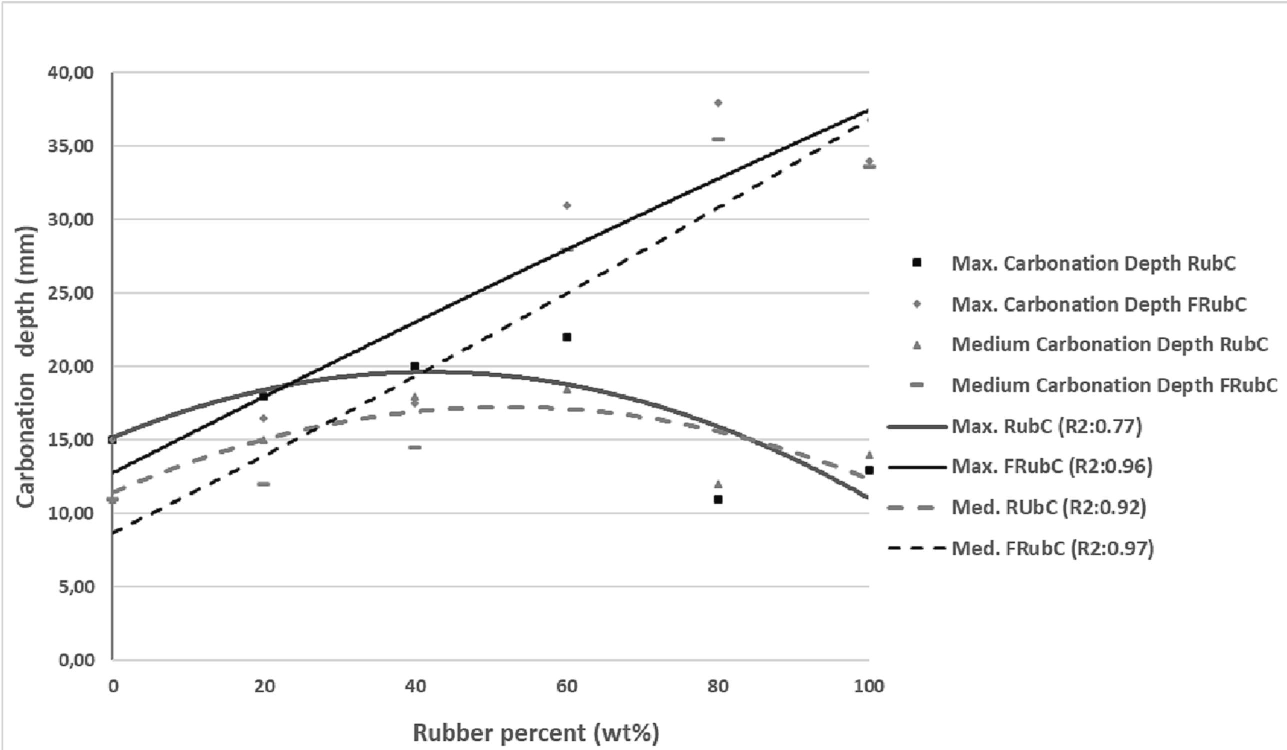

Fig. 7. Carbonation depth versus rubber weight percent (wt%) of RuC with CR and FCR aggregate instead of stone aggregate.

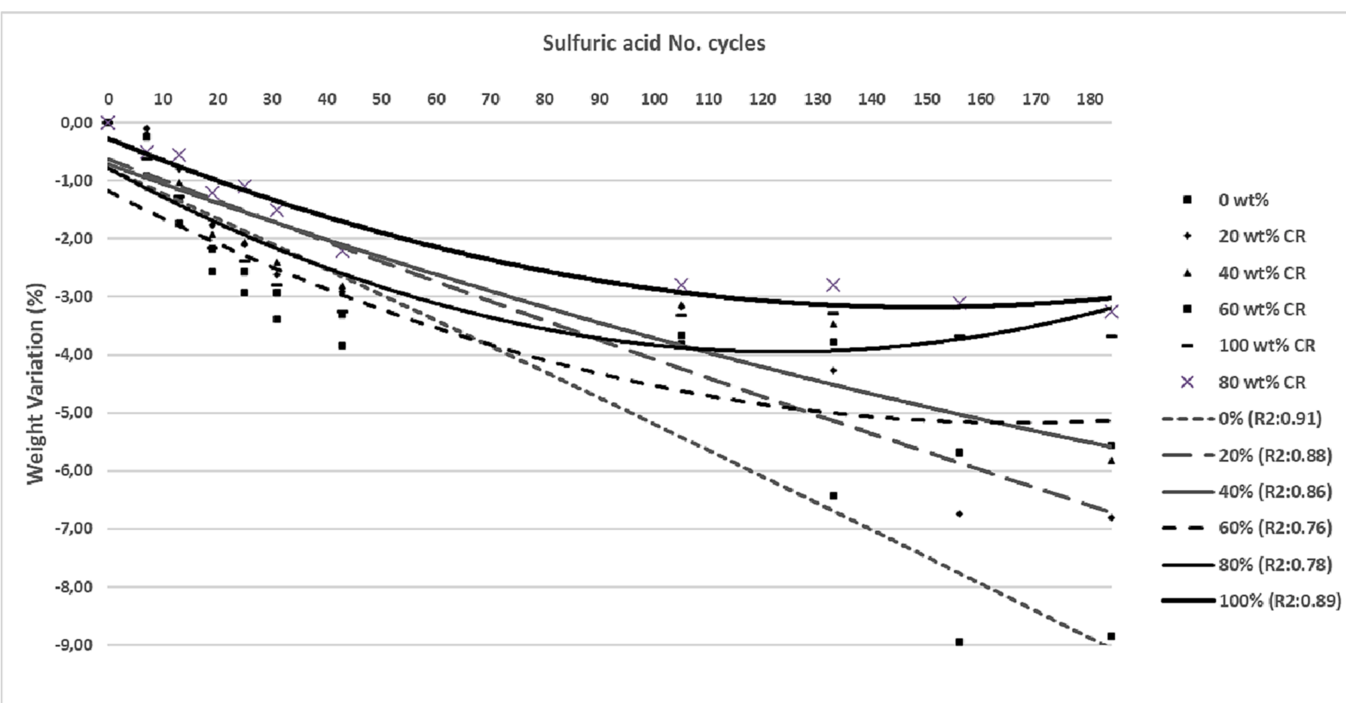

Fig. 8. Weight variation versus sulfuric acid cycles of RuC with CR aggregate.

sulfuric acid exposure shows a disrupted flaky distribution around the fiber–matrix interface. On the other hand, damages in rubber aggregate or fiber are not noticeable.

3.3.2. Acetic attack

Acetic attack of RuC with CR and FCR has been studied in an accelerated process to study its long-term degradation. In Fig. 15, weight variation of RuC samples with CR during the exposure to acetic acid is stated. It can be noted that in the early cycles, the samples presented an augment of weight but at the latest cycles of the test, their weight is finally reduced. Reference concrete shows the lowest initial variation but at the end of the test its weight was reduced by 2.3%. Concrete

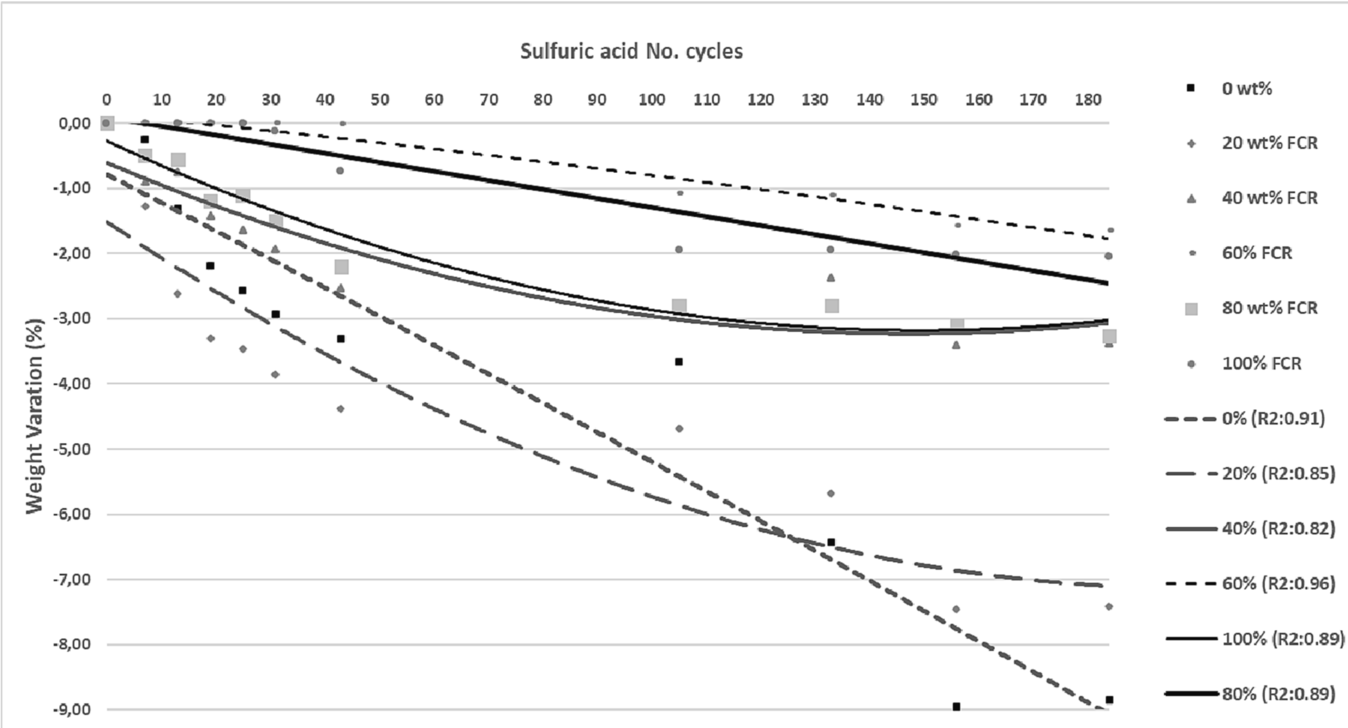

Fig. 9. Weight variation versus sulfuric acid cycles of RuC with FCR aggregate.

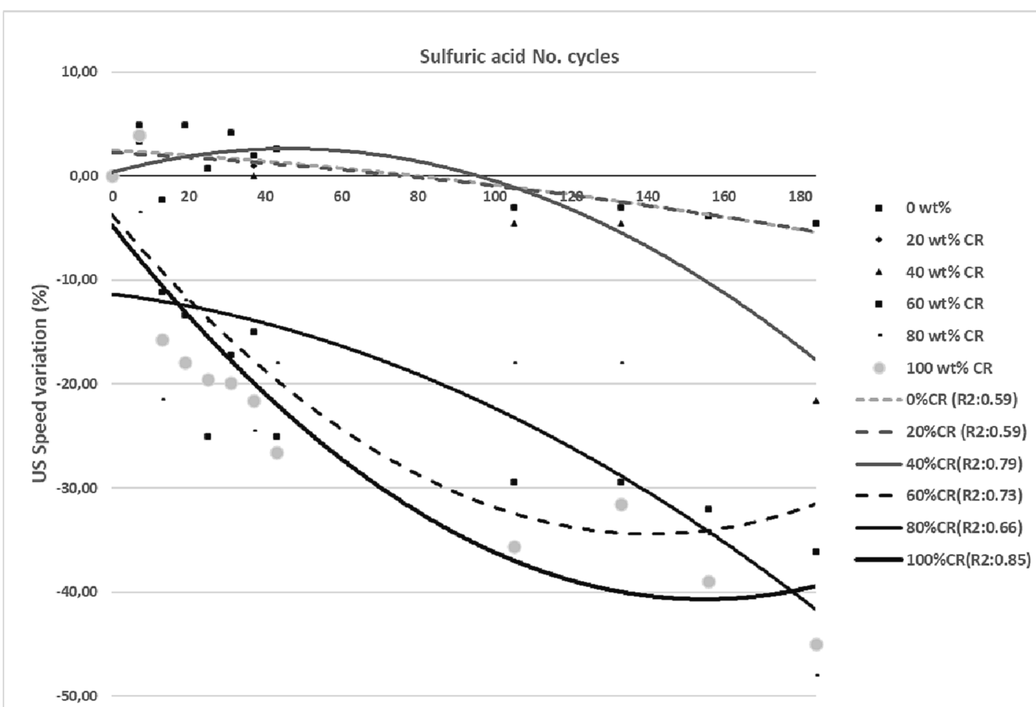

Fig. 10. US variation versus sulfuric acid cycles of RuC with CR aggregate.

mixtures with 20–60% volume of CR aggregate presented similar final weight loss, but higher rises in weight at the first stages than reference concrete. However, RuC with 80 %CR and 100 %CR aggregate volume presented only a 1.1% and 0.9% weight reduction respectively. Weight variation of RuC samples with FCR in acetic acid immersion can be observed in Fig. 16. Weight in RuC with FCR are affected under acetic acid attack, with initial rises in mixtures with 80% FCR up to 4.7% and 60 %FCR up to 3.2%, but final losses down to 19.2% for 60% FCR mixture and down to 12.5% for 100% FCR mixture.

Figs. 17 and 18 represent the variation (%) of UPV in RuC samples under acetic acid attack. Similar to the weight variation, UPV is increased at the beginning of the acid attack but decreased at the end of the test. It can be highlighted that the UPV is more stable for measures in reference concrete with slight variations (under 3%) than on RuC, either

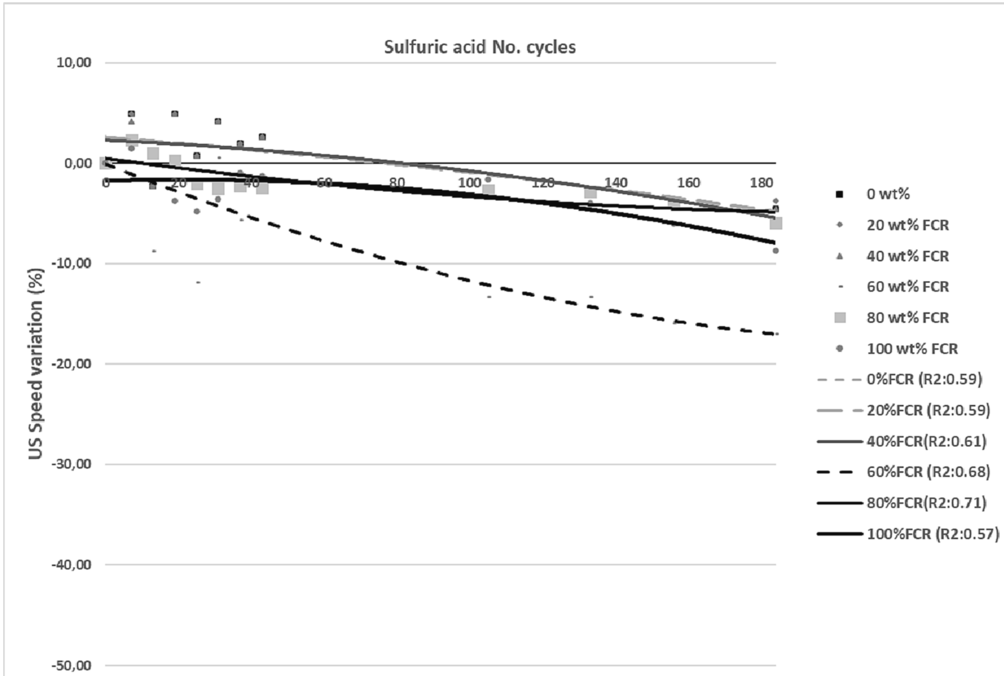

Fig. 11. US variation versus sulfuric acid cycles of RuC with CR aggregate.

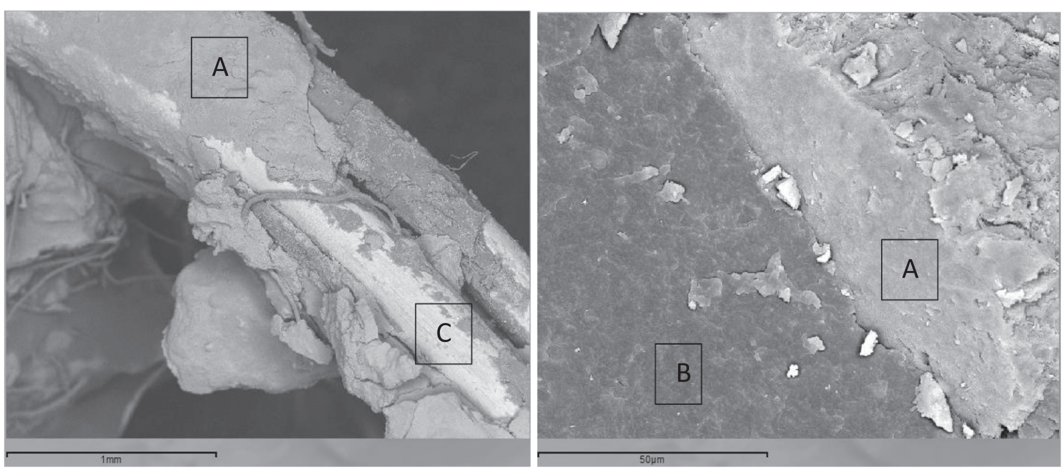

Fig. 12. Images of the sample before acid exposure. 8a (left) shows FCR and 8b shows CR surrounded by the cement matrix. (A. cement matrix, B. rubber, C steel fiber).

with CR or with FCR. In Fig. 17 it can be observed that the higher amount of CR, the lowest UPV. 80 %CR mixture showed the slowest UPV, with a reduction of a 38% after the immersion in acetic acid solution. In the case of FCR mixtures (Fig. 18), UPV is also reduced, showing the sample from the 60 %FCR mixture a UPV 58% slower than at the beginning of the test.

In Fig. 19, SEM images of RuC with CR aggregate after immersion in acetic acid are presented. It can be noticed that the matrix presents some voids and many flaky crystals in a disrupted flaky surface. Rubber aggregates do not show damages or erosion after the tests. However, ITZ between rubber aggregate and cement matrix present no adhesion, as the rubber face is hydrophobic and does not allow the formation of hydraulic cementitious compounds on the outer layer of rubber. In Fig. 20, the product found in the ITZ of rubber and cement is analyzed by X-ray diffraction (XRD). In this analysis, it has been found that the main product corresponds to crystals of Calcium Acetate Hydrate ($C_4H_6CaO_4 \cdot (H_2O)0.5/(CaC_4H_6O_4) \cdot (H_2O)0.5$).

3.4. Freeze-thaw attack

Freeze-thaw attack up to 84 cycles have been studied in RuC samples with CR and FCR. Figs. 21 and 22 depict the variation of dry weight of the different mixtures. It can be observed that the samples presented a tipping point in their behavior. It can be noticed an initial increase of weight, after 49 cycles, but subsequently a weight reduction starts. However, samples did not present a reduction of weight compared to the initial values. It can be observed similar weight loss, after the initial weight gains, in plain concrete as in those RuC mixtures with rubber aggregates up to a 60% volume substitution. However, samples with 80 and 100% of CR presented a stabilization of the mass after the initial weight increase. Fig. 22 shows a similar behavior for RuC samples with FCR, but with a slightly lower volume variation than those with CR.

In Fig. 23, UPV evolution in mixtures under freeze–thaw attack is depicted. We can observe a reduction of UPV with the use of CR. We also can observe here the detail of the UPV increase in samples with CR

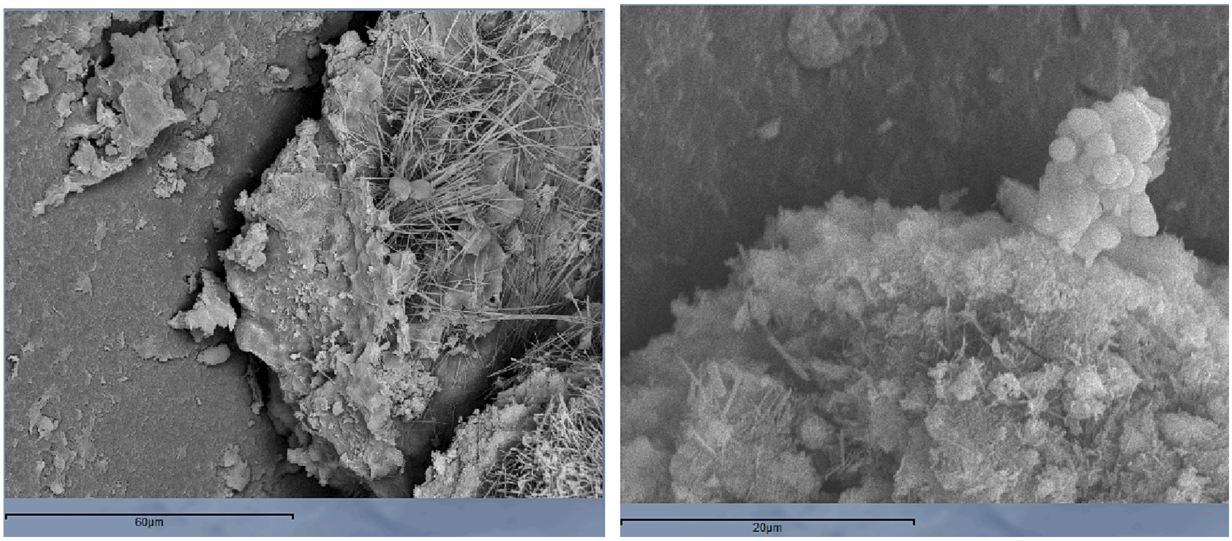

Fig. 13. Sample with CR aggregate after sulfuric acid exposure at different scales.

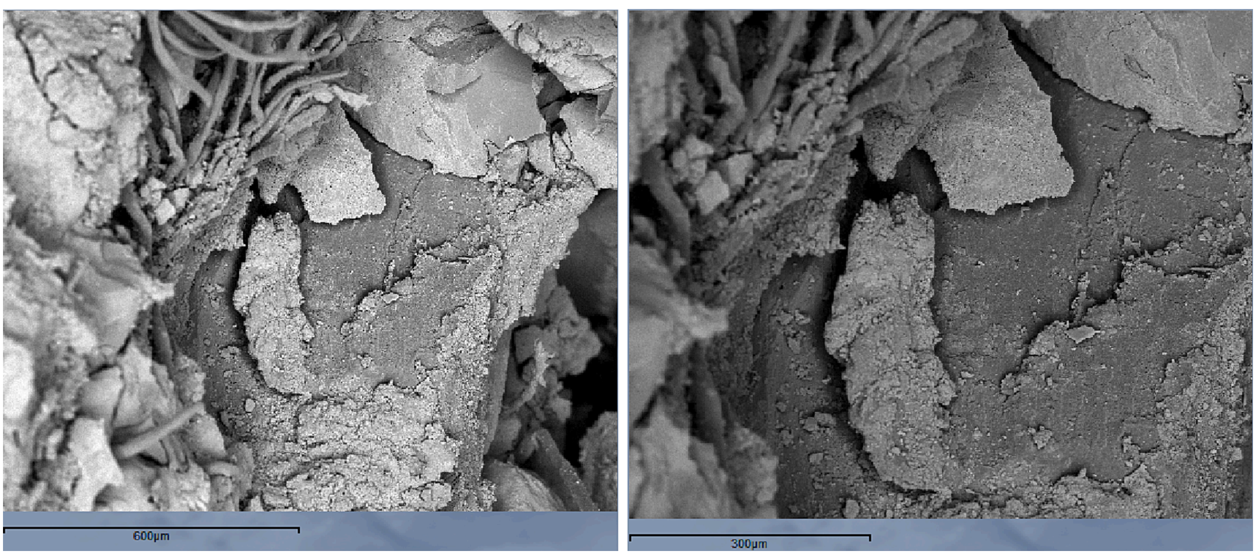

Fig. 14. Sample of FCR after sulfuric acid exposure at different scales.

aggregates of up to 40% in volume in the first cycles, but only before 10 cycles. Plain concrete showed a reduction of velocity of a 7% after been tested. In Fig. 23, we can observe than 20 %CR showed the more stable behavior with only a 3% reduction of UPV. Nonetheless, the higher variation can be found in 100 %CR mixture, with a reduction of velocity of 54%.

Fig. 24 also presents the UPV variation in samples with FCR aggregates. To some extent, it can be also observed that UPV is reduced from a 7% in plain concrete to a 3% when FCR added reaches 40% of volume substitution, but then, 100% FCR mixture showed a UPV 60% slower after it has been tested.

4. Analysis and discussion.

As reported in previous studies, plain concrete has higher density and mechanical resistance than RuC, and moreover lower porosity [21]. These properties are of great importance in a cementitious material to assess the lifespan of the composite [56]. Density is related also to compression strength and bending strength, but also with durability [56,57]. Moreover, porosity is related to density but, in terms of durability, it must be analyzed as it is also linked to permeability of both gases and liquid, as porous media connects the inner and the outer areas of concrete [56]. As it can be observed, concrete with CR or FCR as aggregate shows similar bulk density, but RuC with FCR has higher bulk porosity than with CR (Table 1), and therefore is more permeable and easily attacked [27,28,35,37,57]. It has been also reported by several authors that CR as an aggregate increases the air entrapped [24], but we can also observe a dryer consistency in samples with FCR aggregate (Table 1) which hinders casting and increases the presence of internal voids [30]. It has been also reported the reduction of workability with fibers caused by a balling-up effect [21].

The influence of permeability in durability, associated to transport of aggressive agents, in gaseous or liquid form, has been reported in the literature [20,56]. However, although RuC mixtures are more porous, carbonation is reduced with the use of high volumes of crumb rubber. Besides, when FCR is added, carbonation presents higher depths. Reduction of carbonation depths in samples with high volume of CR (80/100 %), could be pointed as the result of the impermeability of rubber, which reduces the pass of CO_2 through the mass, although cementitious matrix is the main access for the agents to the inner

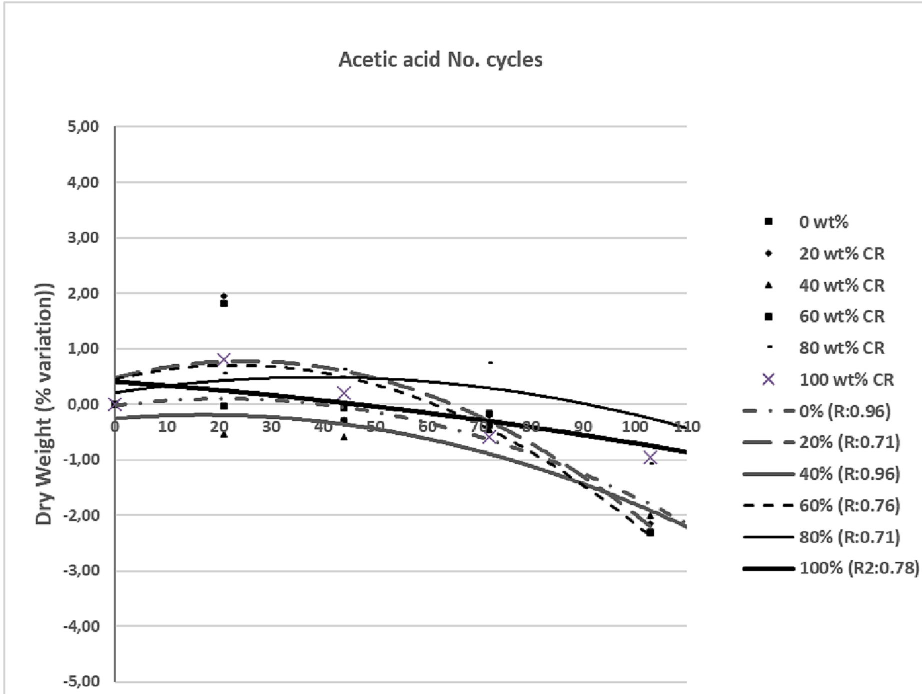

Fig. 15. Weight variation versus acetic acid cycles of RuC with CR aggregate.

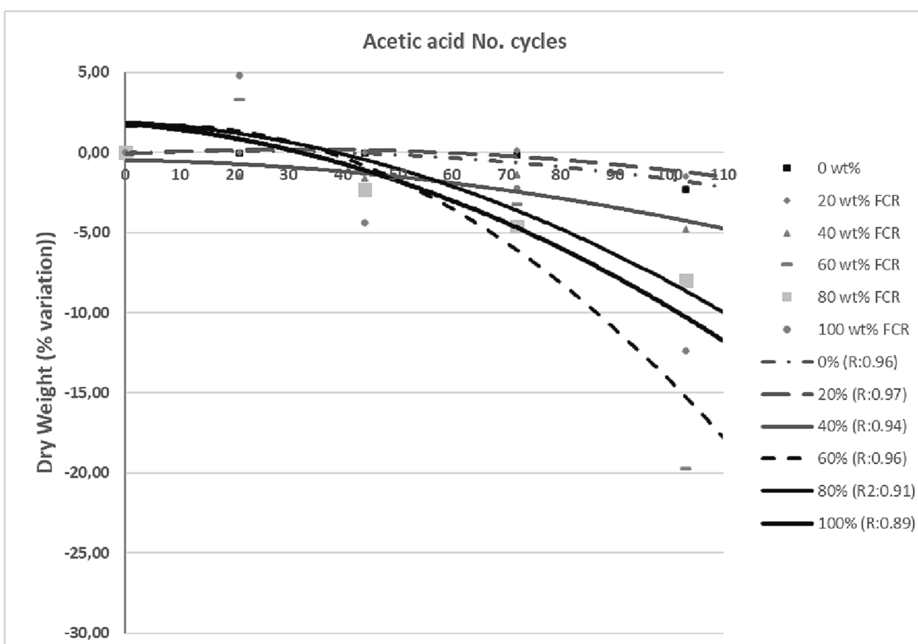

Fig. 16. Weight variation versus acetic acid cycles of RuC with FCR aggregate.

microstructure. Further and more in-depth studies can be carried out to assess the reduction of the porosity of the matrix, which was not observed in this study. In the case of mixtures with FCR, although rubber is also present in the mass, the porosity is much higher, from a 11% for 100 %CR mixture to a 21% for 100 %FCR mixture. This higher porosity, typical of pervious concrete [56], permits the entrance of air with CO_2 and the formation of Calcite through the combination with Portlandite and therefore a reduction in pH occurs [20]. It must be noticed that it could be worsened whether superplastizicers are used [38], although water/cement relation must be under 0.5 to enhance the carbonation resistance [16]. To mitigate carbonation [58] used crumb rubber in a mixed-grade proportion, instead of singly sized rubber aggregates to reduce porosity [58]. Pozzolanic additions [54] or rubber aggregate pretreatment [25] can be used to reduce carbonation depths of the RuC mixtures studied here [27,46].

The assessment of acid attack in concrete was performed by measuring the weight of the specimens and recording ultrasonic velocity during the durability tests. From the results obtained of samples subjected to sulfuric acid attack (Figs. 8-11), it can be noticed that through the measurement of weight loss during the attack, the formation of

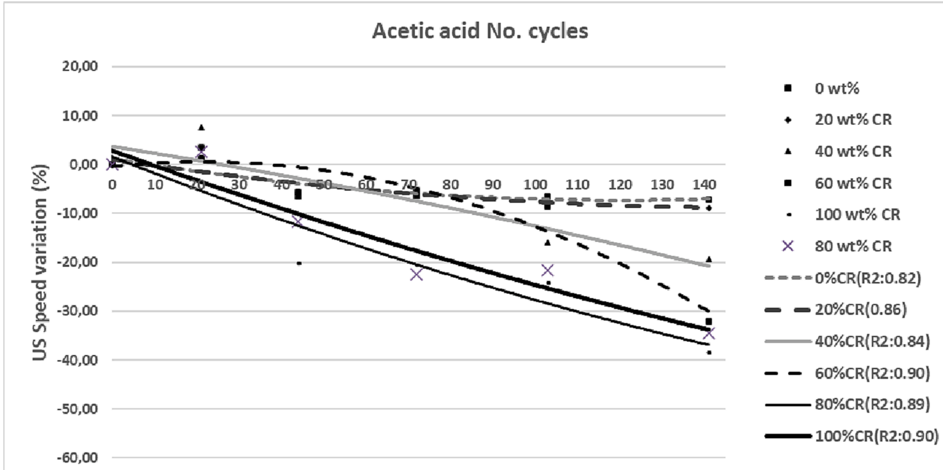

Fig. 17. UPV variation versus acetic acid cycles of RuC with CR aggregate.

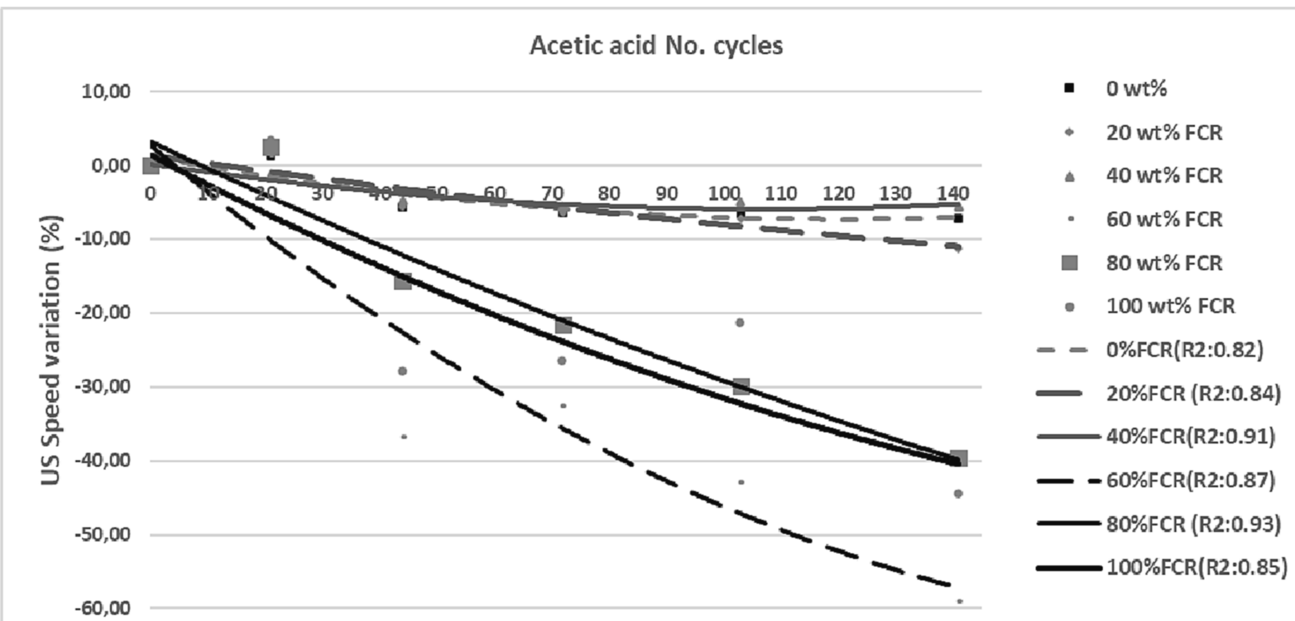

Fig. 18. UPV variation versus acetic acid cycles of RuC with FCR aggregate.

ettringite cannot be detected during the early cycles of the process (first stage), except for UPV test. In the initial stages, acid sulphate combined with portlandite creates calcium sulphate and later ettringite (equations 2–4) [34]. Calcium sulphates and ettringite crystals can fill the voids of the cementitious matrix, which eases the wave travel and raises the velocity [57], showing an increase of UPV in the first cycles. However, it is remarkable from UPV test and also but more slightly from weight variation measurements, that sulfuric attack in RuC as in RC [20,56,57] presents a second period or phase in which the ettringite begins to crack and disaggregate concrete mass (Figs. 10-11). Later on, ettringite in the presence of H_2SO_4 can be also modified as monosulphate which is more dilutable and can be transported out of the sample [20].

In this stage, ettringite is also able to press and push out part the mass of the hardened concrete in its outer face. As it has been observed by SEM analysis, the internal mass is disrupted by the growth of ettringite crystals (Figs. 12-14), what reduces the UPV. Other authors also observed a decalcification of C-S-H through SEM analysis environments [63]. RuC ITZ is less dense than cement matrix and may present cracking in the boundary with cement matrix, what also reduces the resistivity to aggressive environments [63]. Moreover, the growth of ettringite in the ITZ microcracks increase their size, on the other hand it has been also observed that the hydrophobicity of CR aggregates and un-reactivity with sulphuric acid reduces acid diffusion in RuC [28]. Moreover, the mass loss also increases the porosity and, therefore, the wave that cannot be easily transported in the internal mass and UPV is further reduced. From the results, UPV testing seem to be more sensible as a technique to evaluate sulfuric attack in RuC, as the trends are more similar comparing the figures of percentage variation of compressive strength, UPV and weight loss. Therefore, by assessing the relation between non-destructive methods used and the reduction of compression strength, we can remark that, for instance, in 60 %CR mixture the strength is a 73% reduced after the attack, while a 32% reduction of UPV and a 5.5% of weight loss was recorded. Weight variation is only perceptible when the mass is disaggregated from the face of the sample (mainly in advance attack) or transported during drying and wetting cycles [57], being the UPV measurement more accurate. However, establishing a direct correlation between UPV measurements and final strength should be deeply studied.

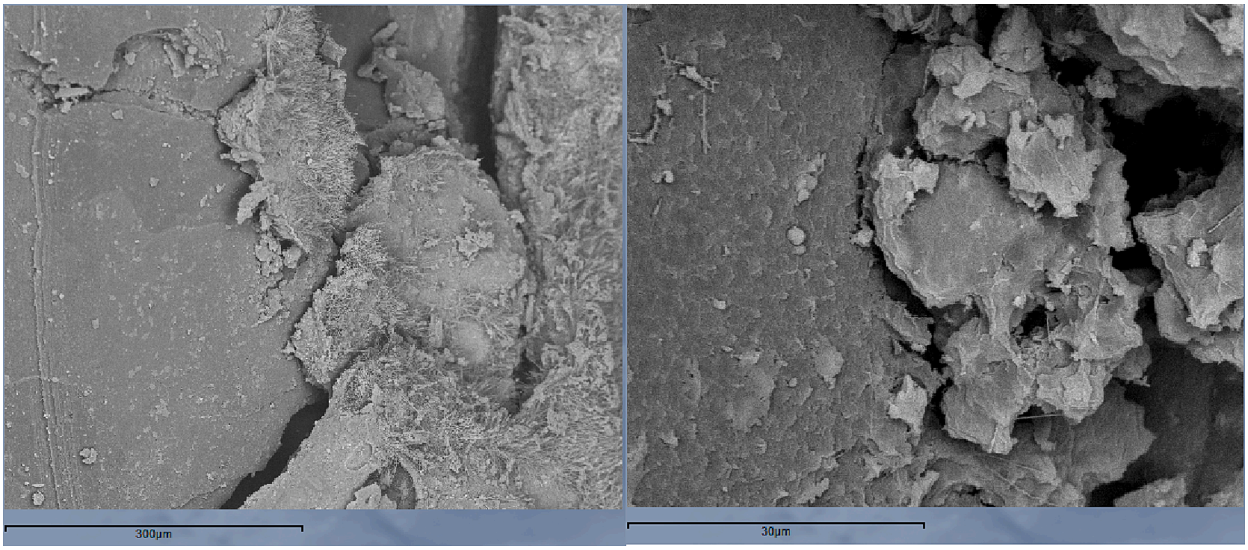

Fig. 19. SEM images of CR aggregate after acetic acid immersion, matrix present some voids and many pointed crystals.

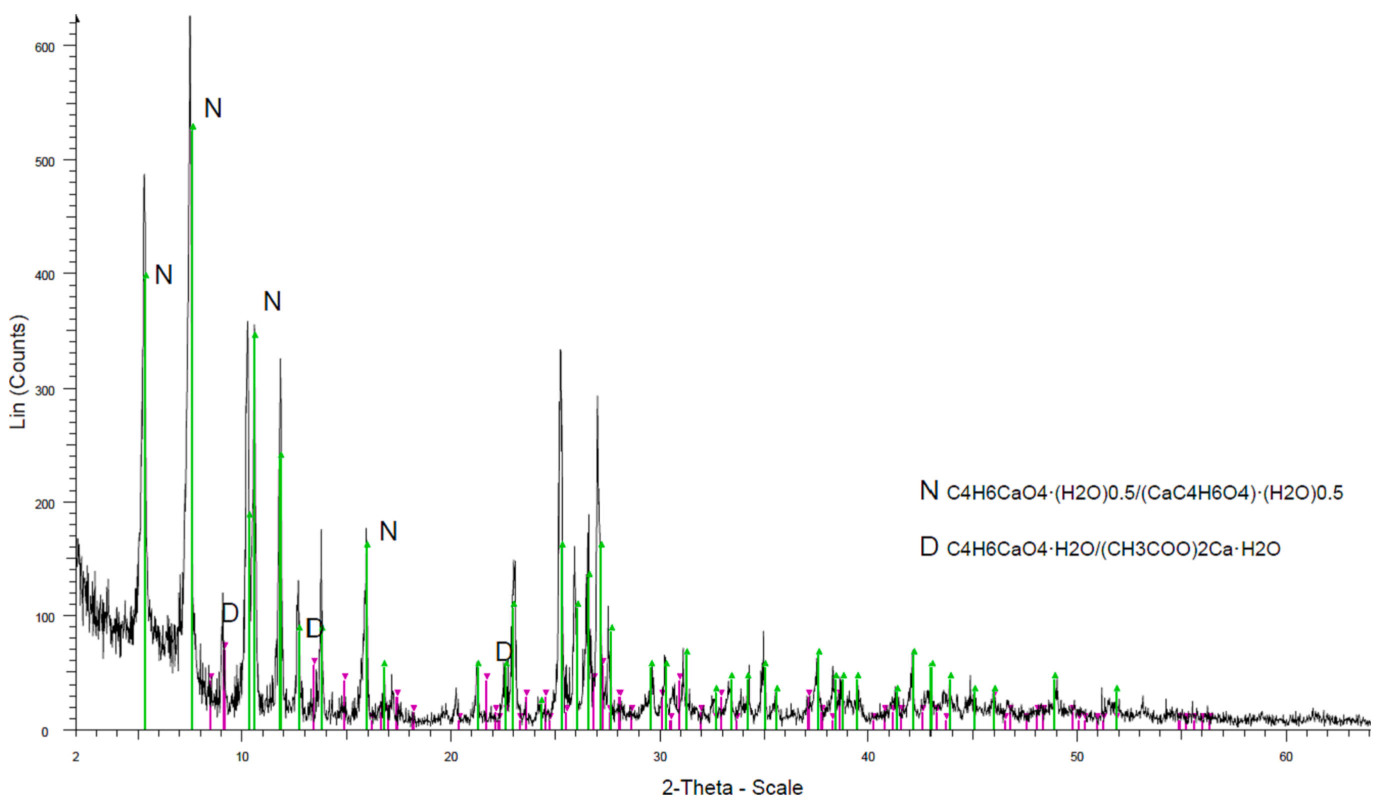

Fig. 20. XRD pattern of crystals obtained in the vicinity of CR after acetic acid attack.

It is also noteworthy that RuC samples with an aggregate volume over 80% of CR or FCR aggregates do not lose as much weight as mixtures with lower CR volume. It can be caused as CR aggregates are deformable, providing extra room to release internal stresses caused by the expansion of gypsum what reduces the cracking growth, and they are hydrophobic slowing the deterioration process loop [28]. In addition, mixtures with FCR also present less weight loss as fiber may binder the mass and reduces cracking [18,21], mass loss and disaggregation on the outer face of the samples [26]. Despite, it is remarkable that the majority of RuC samples here studied showed more strength losses than RC, contrary to other studies with volumes of rubber usually around 5% [30,54], or 20% [44]. The higher volume of CR in concrete mixtures increased the strength loss at the end of the attack, but in the case of mixtures with FCR it is only appreciable in the case of 80/100% volume. In terms of strength loss, RC showed 25% less strength after the acid exposure, and RuC samples with of 20 %CR and 80 %CR aggregate volume presented losses around 40%, but RuC with 20 %FCR and 40 % FCR aggregates only 23% and 16% respectively (Figs. 5-6). Although their final resistances continue to be lesser than reference concrete, the FCR increases its final strength thanks to its cohesion to the cementitious mass, even after the attack (Fig. 14).

Acetic acid attack on RuC showed two stages during the damage that

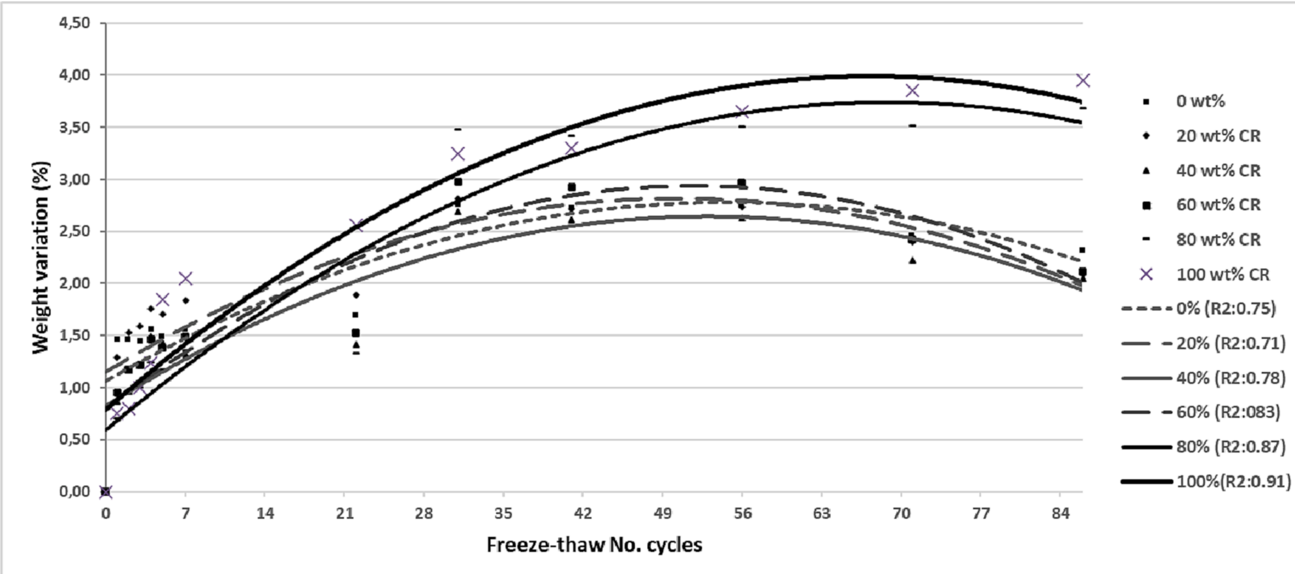

Fig. 21. Weight variation versus freeze–thaw cycles of RuC with CR aggregate.

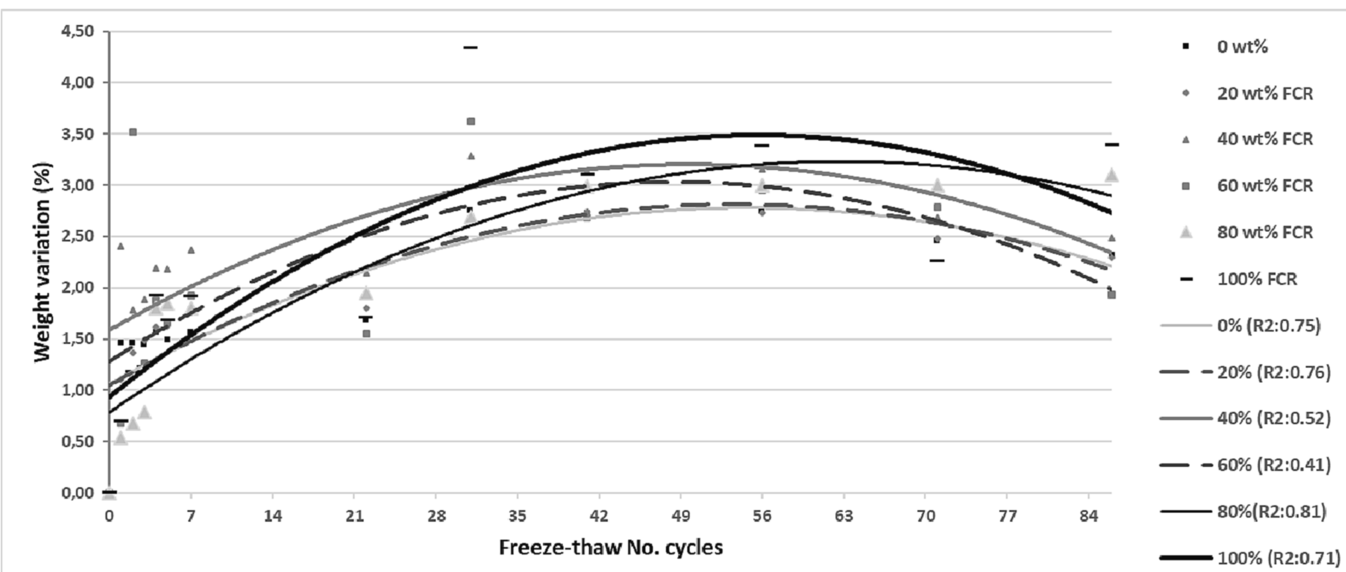

Fig. 22. Weight variation versus freeze–thaw cycles of RuC with FCR aggregate.

has been observed by weight and UPV measurements, showing that both methods can be used to evaluate the acid corrosion. In the first stage, both weight and UPV are increased but reduced in the second stage. On one hand, the formation of calcium acetate hydrate ($C_4H_6CaO_4 \cdot (H_2O)$ 0.5/($CaC_4H_6O_4$)$\cdot$(H_2O)0.5), confirmed by XRD analysis (Fig. 20), can fill the microstructure in the first stages of attack. On the other hand, usually portlandite is found in ITZ as several authors reported [55], and when portlandite and acetic acid are presented and salts are formed, aggregates (both siliceous or rubber) can be separated from the cementitious matrix when acetic acid attack occurs. Linoshka Soto-Perez et al. observed that calcium salts are highly soluble in water, reducing in advanced stages of the attack the mass of the samples [59]. Comparing the weight loss in samples with CR or FCR, we can observe that RuC with FCR concrete is more affected because FCR mixtures are more porous (Table 1). This is due to the fact that acetic acid can enter the mass more easily in samples with FCR than those with CR aggregates, but also to transport the calcium acetate hydrate formed outside the sample in the outer layer. Besides, attending to the reduction of UPV, FCR mixtures present lower reduction. This may occur, since UPV variation indicates more accurately than the weight loss, the internal cracking or disruption in the microstructure, which in the samples with FCR is lower since the fibers can reduce cracking by seeding the cracks [13,21]. Compression strength variations in samples with FCR after acetic acid attack, presented in general terms better values than those with CR, highlighting the importance of the fibers as they bind the internal mass of concrete.

Durability of RuC in freeze–thaw attack improves in samples with FCR, but not in samples with CR, in contrast to the literature studied [23,50,60]. It can be caused by the high amount of rubber aggregate added and its size [27]. Xifeng Gao reported an improvement of RuC when rubber aggregates do not exceed a 25 kg/m3 and a range of rubber size between 0.1 and 0.5 mm [64]. Li, Y.; et al. 2022 also set a maximum of 10% CR content to reduce water permeability of RuC, above this percent a more permeable concrete was observed. Luo, G 2021 improved the compressive strength loss through the combination of

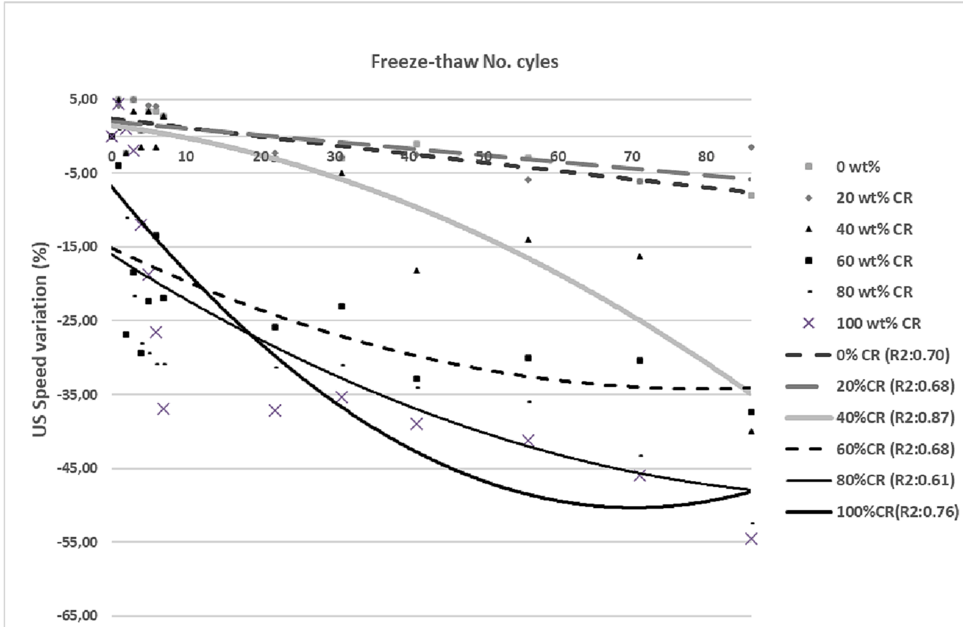

Fig. 23. UPV variation versus freeze–thaw cycles of RuC with CR aggregate.

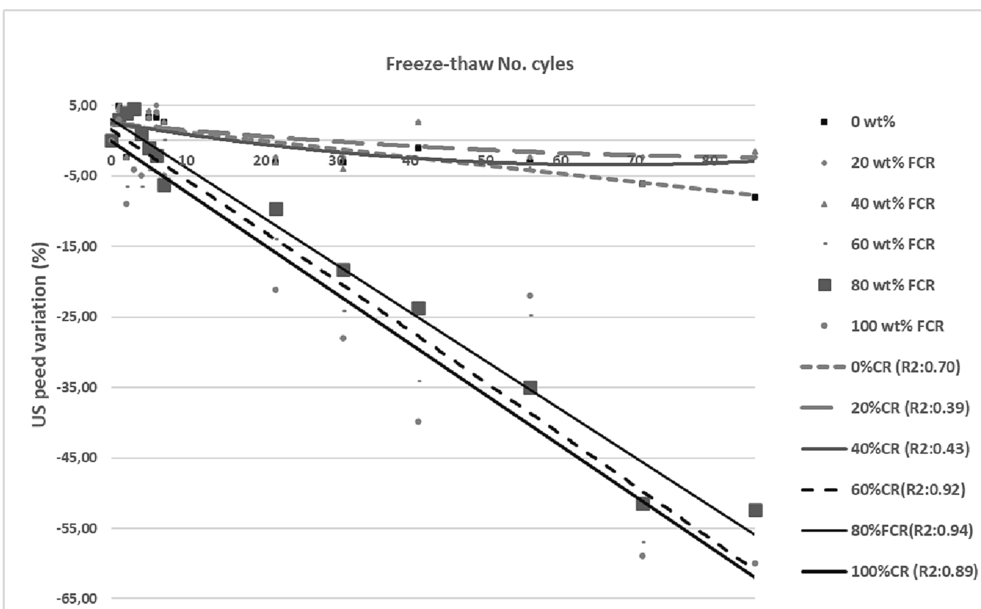

Fig. 24. UPV variation versus freeze–thaw cycles of RuC with FCR aggregate.

rubber powder, from a 18.5% strength loss to a 5% with a 12% of silica fume, and also in pervious rubberized concrete [35]. Moreover, in this research work, it can be caused by the high volume of CR and FCR and the dry consistency of the mixtures here studied, which have a larger permeability than those from the literature, facilitating the entry of more water [26]. The dry consistency concrete here studied presented a weight increase during the test in all samples, but also UPV as non-hydrated cement phases re-hydrated in the immersion part of the tests.

RuC with FCR aggregates showed improved compression strength, as previously reported by other authors with industrial fiber used in self-compacting RuC [30,61], recycled-tire steel fiber in conventional RuC [Peifenf Su] and macro polypropylene fibers [60]. The fibers are able to absorb also energy when the ice pushes the internal mass, as they can work in tension and reduce the disaggregation of the mass and the strength loss. Furthermore, FCR in volumes between 20 and 60% the strength loss is lower than for plain concrete and in those with 40–60% FCR present gains of strength also because some of the non-hydrated cement phases have re-hydrated during the process.

Further research to enhance the durability of RuC with CR and FCR by reducing the permeability of RuC can be done. Pre-compressed rubber can reduce air voids and therefore permeability and absorption by immersion pof RuC, increasing its resistance to sulphate attack and chloride ion penetration [60]. Attending to the literature, to improve durability of RuC with FCR, more attention must be paid to the ITZ improvement, through the use of air-detraining admixtures that reduce the amount of air trapped [33] or pozzolanic fillers (fly ash, metakaolin or Silica Fume) which reduce ITZ width [39], permeability of the matrix and fill voids showing a more homogeneous microstructure and less cracking in the boundary between hydratation products and aggregates [26,35,37,39,40,62]. It can be more efficiently improved when combined with a previous treatment of rubber, such as water cleaning (to eliminate dust from recycling and store process) and later a bath in acid dissolution to guarantee a better bond between rubber and cementitious matrix (obtaining a more rough and less hydrophobic rubber surface). KMnO4 can be also used as rubber treatment, obtaining a layer of Mg on the rubber surface that enhances roughness and hydrophilicity of rubber and improves the workability as edges of rubber are smoother [28]. Also cement treatment modifies surface and it has been proved to be effective as hydrophilicity enhancer of rubber aggregate, leading to better particle packing of concrete [28]. Latex or precoating Cr with synthetic resin and styrene–butadienetype [27], also can improve rubber and matrix adhesion [19].

The study here presented was focused in dry consistency concrete to be used in precast items, for example, but also it can be studied the influence of FCR in self-compacting concrete to reduce the air entrapment and therefore increasing the durability of concrete.

Future research works may be focused on the improvement of the workability of the mixtures, as final porosity can be reduced as in self-compacting RuC [33]. On the other hand, durability of RuC with FCR can be also studied in dry consistency concretes with CR and FCR at high substitution volumes by the aforementioned pre-treatment types of these rubber aggregates through the improvement of ITZ [23].

5. Conclusions

Durability of rubberized concrete (RuC) with rubber aggregates and steel and plastic fibers coated with revalorized rubber crumb from waste tires in aggressive environments commonly found in residential, civil and industrial uses has been assessed.

On one hand, rubber aggregates increase the porosity of the mixtures, since consistency is dry, their workability is reduced with CR but specially with FCR, and moreover, air is entrapped during casting. On the other hand, crumb rubber aggregates here studied did not show any damage after the test, although their ITZ with cementitious matrix is affected.

- Carbonation is reduced in RuC with CR in volumes substitutions of 80–100%, as it acts as a barrier to gases. However, carbonation in RuC mixtures with permeability over 20%, such as in mixtures with 80–100% of FCR, increases carbonation.
- Acid attack on RuC here studied, both acetic and sulfuric environment, presents a first stage of filling of the internal voids of RuC, and a second stage with a weight loss and UPV reduction. This second stage occurs as the salt crystals can be diluted and externally transported and/or matrix is disrupted. This results in a strength loss, augmented with the reduction of the ITZ bond between aggregate and matrix, as the new compounds have been observed in the vicinity of the rubber aggregates creating debonding and layering of the matrix. Combined analysis of UPV and SEM micrographs show that the debonding and reduced transmission of ultrasonic pulse are related to the strength loss and deterioration of the samples here studied.
- FCR instead of CR, used in RuC with rubber aggregate volume 80–100% subjected to sulfuric acid corrosion, reduces the strength reduction which increases the stability of the sample after attack. In acetic acid attack, RuC samples with FCR in show lower resistance loss than those with CR after testing, as the fibers in FCR saw cracking and maintain matrix bond with the aggregate.
- RuC in freeze–thaw cycles with FCR in volumes from 20 to 60% present not only lower resistance reductions after being tested, but also strength gains, demonstrating also a better behavior than crumb rubber without fiber, as fiber is able to absorb energy when ice increases in volume.
- UPV measurements are a more accurate and sensible method to estimate RuC lifespan than weight loss, although further studies should be conducted.

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

No data was used for the research described in the article.

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