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Efeito da adição de fibras híbridas na durabilidade de compósitos cimentícios avançados em ambientes úmidos e agressivos

Effects of fiber hybridization in advanced cementitious composites durability in humid and aggressive environments

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ABSTRACT

Reactive powder concrete (RPC) is one type of advanced cementitious composite that has been increasingly used. Its high mechanical strength enables the application of the composite in bolder structures. Considering its recent history of use, there is a lack of knowledge about this materials durability. Therefore, this paper aimed to evaluate the behavior of hybrid fiber reinforced RPC under accelerated testing, evaluating different hybrid fiber combinations. Composites contained metallic microfibers and polypropylene fibers in the following proportions: 100/0%, 75/25%, 50/50%, and 0/0% (metallic/polypropylene). Accelerated tests were performed (salt spray, carbonation and acid immersion), as well as 3D microtomography analyses, in order to visualize the distribution of fibers throughout the matrix. Results indicated high durability under some aggressive wet environments, with no signs of carbonation, nor damage caused by salt spray, and small damage related to sulfuric acid attack. In addition, it was possible to observe in the 3D microtomography images that fibers are well distributed in the matrix volume, and that there is no transition zone in the material due to the reduced volume void.

Keywords: Reactive powder concrete; Chemical attack; Durability; Deterioration; Fibers hybridization.

1. INTRODUCTION

As investments are made in the development of new technologies and products, such as high and ultra-high performance concrete, careful analysis and determination of the parameters that affect its durability is essential. The goal is to implement improvements to increase the material's ability to resist attack of aggressive agents. The impact caused by chemical reactions in water as well as the weather can affect the original quality of concrete or even reduce its intended service life, hence the need for research into material's durability and construction systems [1,2]. The huge use of conventional concrete stimulates the research for understanding its physical, chemical and mechanical properties throughout its life cycle. Concrete undergoes degradation resulting from environmental effects as well as from the use of the structure. In this scenario, several studies have investigated how the carbonation or chloride ion penetration deteriorate the concrete matrix or accelerate rebar's corrosion process [3].

Conventional concrete properties are affected when exposed to such conditions and, therefore, the material does not meet the requirements for which it was designed, requiring the development of mixtures with higher strength and performance. One of the reasons that conduce conventional concrete to deterioration is the existence of transition zone, between aggregates and cementitious matrix. Considering this, the use of advanced cementitious composite emerges as a material option with better physical and mechanical properties, which increases the structural element's durability.

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Callister Jr. and Rethwisch (2014) [4] define composite materials as a combination of two or more materials that can belong to three classes of basic materials: metals, ceramics and polymers. This material aims to achieve properties not provided by a single material joining the best characteristics of each item of the composition.

These materials consist of advanced compositions, considering that conventional concrete has evolved since its basic composition given the use of superplasticizing additives, reduction of water / cement ratio, pozzolanic additions such as active silica or fly ash, among others. This development aims to reach specific properties of the materials, not achieved with the conventional concretes [5,6,7].

One of those composites is the RPC - reactive powder concrete -, a high performance concrete, widely used with high cement content and ultrathin materials such as silica, quartz powder and sand with particles smaller than 300 µm. Microfibers with approximately 1,2mm of length and 0,2mm of diameter are also included. [8]. This composite doesn´t use coarse aggregate. This material presents higher mechanical properties, with compressive strength around 200 MPa, being able to obtain 800 MPa, with elasticity modulus of 45 GPa [9].

Juanhong; Shaomin; Lin [10] define this special composition as a modern material, considering the possibility to replace concrete with pozzolans to reduce its environmental impact. According to Tutikian et al. (2011) [11], it is used in applications where conventional concrete does not meet design specifications, as in the case of structural elements with large spans, intensive aggressive agent action, need for waterproof structures, and others. RPC has a homogeneous matrix where powders are combined with optimum compaction and reduced voids, being able to withstand more stress and deformation [5,12,13]. In the use of fine short fibers with high deformation modulus the RPC can be able to absorb energy, achieving values similar to the ductility and other isotropic properties of some metal composites.

Additionally, fibers are an important and fundamental constituent as their main function is to strengthen the predominantly fragile cementitious matrix. In addition, confining pressure in the fresh state and high temperature curing can be applied [10], considering that this procedure reduces porosity, enhancing material's properties.

According to Banyhussan et al (2016) [14], the material properties are well known, being the current stage of reduction its microcracks by the use of microfibers. Furthermore, a microstructure with optimal compactness and reduced voids makes the element more resistant to different aggressive agents to which structures are commonly exposed [15]. The RPC without fibers is a material with high mechanical properties but brittle. Thus, as noted by Dawood and Ramli (2011) [16], the introduction of microfibers makes the RPC more resistant and able to withstand stress and deformation. Souza (2014) [17] highlights the expansion of the material, considering the use of microfibers provides greater spans and architectural possibilities.

Wu et al (2016) [18] show the incorporation of metal fibers as favorable to the mixture, increasing its tensile strength and reducing crack formation. According to the authors, the parameters of the fibers that interfere in their performance are: format, distribution, way of launching the microfibers in the mixture, shape indices (dimension x height), among others.

In this context, Christ and Tutikian (2013) [19] state the use of fibers increases the tensile strength, reduces initiation and propagation of cracks from shrinkage and increases the impact and fire resistance of mixtures.

Using two types of microfibers can contribute to these aspects as hybridization maximizes the effects of each type of fiber. The combination of steel fibers (0,5%) and polypropylene fibers (<0,2%) is possible to reach a synergy. The hybridization culminates in greater compressive strength [20], benefit the composite's tensile strength, cracks control and ductility increase [21]. Steel fiber are less flexible, which contributes to improving tensile strength. Furthermore, polypropylene fibers, which are more flexible, adds the stress generated immediately after the first openings [22].

Yu; Spiesz; Brouwers [23] found out that, basing on the optimized particle packing and hybrid macro and microfibers, it is possible to produce RPC with low binder amount (about 620 kg/m^3) and low fiber content (volume of 2%). According to the authors, with hybrid fibers composition is possible to improve materials flowability and its mechanical properties. However, the behavior of RPC concerning durability is not well known, though the expectations are positive because of its reduced porosity [24].

Pacheco et al (2016) [25] verified, through SEM, the interface between fibers and the cementitious matrix is not characterized as a fragile zone or accumulation of voids, and no affects the compactness of the material. Stamatina et al (2017) [26] affirm that one of the reasons of hybridity is the improvement of the mechanical and early age properties. It is possible also to use fibers with different functions.

In order to contribute to RPC durability properties knowledge, this study was developed to characterize the hybrid RPC, with two types of microfibers (polypropylene and steel), considering hybridity of fiber function, assessing durability parameters with accelerated tests of carbonation, salt spray and chemical attack by acid (hydrochloric, nitric and sulfuric). Different percentages of two types of fibers were evaluated.

2. EXPERIMENT

The experiment started by materials characterization. For samples' preparation, early age high strength cement was used. The determination of particle size distribution occurred by laser particle size analysis, following the prescriptions set in ISO 13320: 2009 [27], which results are presented in Figure 01. The test showed that the average particle diameter (D50) was 95.76 nm and the average size was 50 µm. Table 01 shows the cement mechanical properties.

Table 1: Cement characterization and mechanical properties

PROPERTIES	VALUE	
Specific density (g/cm3)	3,09	
Specific surface (cm2/g)	4190	
Hardening begin (h:min)	3:05	
Hardening end (h:min)	4:25	
Compressive strength (MPa)	1 day	14,6
	3 days	34,6
	7 days	40,7
	28 days	46,6

The laser particle size analysis made for silica fume was performed following the same procedures employed for cement, yielding the results shown in Figure 01. Table 02 shows chemical composition and physical properties of the silica fume.

Table 2: Silica fume chemical and physical characteristics

CHEMICAL COMPOSITION	CONTENT (%)	PHYSICAL CHARACTERISTICS	CONTENT (%)
Silicon (Si)	88,43	Humidity (%)	2
Sodium (Na)	2,786	Specific density (g/cm3)	2,35
Potassium (K)	0,658	Bulk density	0,375
Aluminum (Al)	0,316	pH	10
Calcium (Ca)	0,286	Specific area (m2/kg)	20.000
Magnesium(Mg)	0,122	State	Solid
Titanium (Ti)	0,091	Medium diameter (µm)	0,004
Iron (Fe)	0,015	Loss on ignition (%)	6

The particle size analysis of quartz powder by laser diffraction presented the results shown in Figure 01. The material's physical and chemical properties are shown in Table 03. The average particle diameter obtained was 91,93nm.

Table 3: Quartz powder physical and chemical characteristics.

Physical Characteristics	Results	Chemical Composition	Content (%)
Humidity (%)	1	Silicon dioxide (SiO_2)	99
Specific density (g/cm^3)	2,8	Aluminum oxide (Al_2O_3)	0,25
Bulk density	1,35	Iron Oxide (Fe_2O_3)	0,05
pH	11	Titanium Dioxide (TiO_2)	0,03
State	Solid		
Loss on ignition (%)	0,6		

Fly ash is not a common component of RPC, but it is already proven that it can be used and improve composite's mechanical properties [28], creating an Environmentally-friendly class of RPC [29]. The fly ash used in this research comes from a steam generating unit of a paper manufacturer. Its main components obtained by X-ray fluorescence testing, is shown in Table 04. Figure 01 shows the ash particle size distribution. The average particle diameter obtained was 63,92nm.

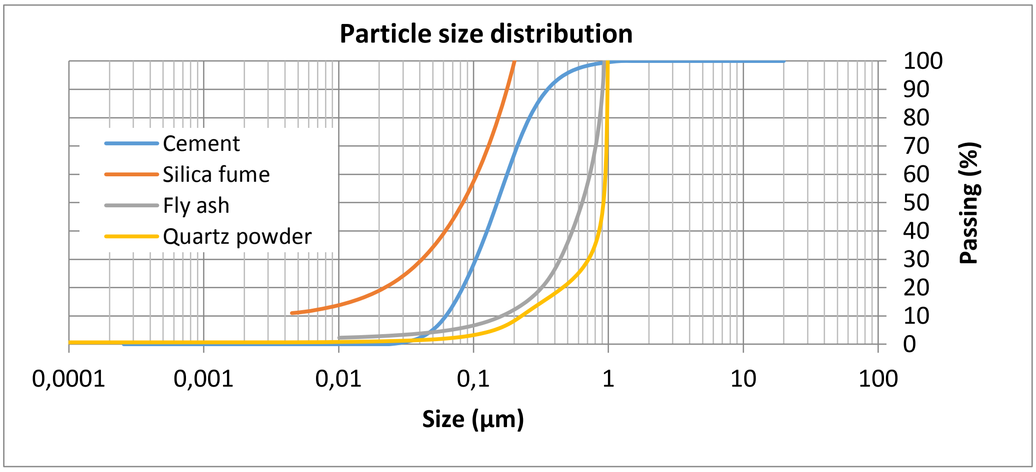

Figure 1: Cement, silica fume, fly ash and quartz powder particle size distribution – laser granulometry.

Table 4: Fly ash main components

CHEMICAL COMPOSITION	CONTENT (%)
Silicon (Si)	69,3
Aluminum (Al)	26,1
Iron (Fe)	1,8
Potassium (K)	1,4
Calcium (Ca)	0,9
Sodium (Na)	0,3
Magnesium(Mg)	0,05

Two types of sand were used throughout the preparation of the RPC mixes studied: fine sand, usually used for mortar coating finish, and regular sand. The particle size distribution of both types of sand was determined according to procedure by AASHTO T 27: 2015 [30]. The results obtained are shown in Figure 02. The fineness modulus for fine sand and regular sand was 1.64 and 2.83, respectively. Table 05 shows the properties of the superplasticizer and the viscosity modifier as informed by their manufacturers.

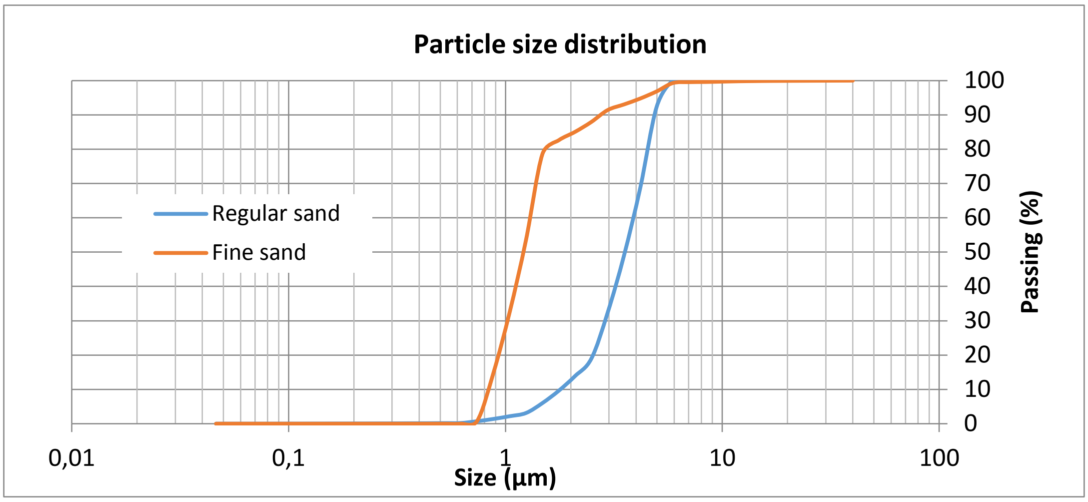

Figure 2: Sand particle size distribution

Table 5: Chemical additives characteristics

PROPRIETIES	SUPERPLASTICIZER ADDITIVE	VISCOSITY MODIFIER ADDITIVE
Physical state	Liquid	Liquid
Color and odor	Turbid yellow and characteristic odor	Transparent and characteristic odor
Specific density (g/cm³)	1,10 +/- 0,02g/cm³	1 +/- 0,02g/cm³
pH (pure product)	5,5 +/- 1,0	9,0 a 10,5
Fire reaction	-	Not inflamed and non-explosive
Volatiles (% by weigth)	51%	-
Water solubility	Completely soluble in water	-

Microfibers were characterized for their geometric properties. The metal microfiber had 13 mm in width, 0.11 mm diameter and form factor of 0.01. For the polypropylene microfibers these values were 6 mm, 0.0012 mm and 0.0002, respectively.

The composition of mixtures was determined using the modified Andreassen's packing model [31]. For the durability tests, samples were prepared by applying 20 MPa pressure in the fresh state and heat curing in the first 24 hours, at 90 °C. This pressure was applied through a universal testing machine, with load increase velocity of 0,45MPa/sec.

After this stage, the samples were kept in an environment with controlled temperature of 23±2 °C, submerged in water until the date of testing. Table 06 shows the mix used, as well as the variations of use of both microfibers, metal and polypropylene. A volume content of 3% of microfibers was adopted for the mixture, corresponding to 235 kg/m3 of fiber. Thus, it was possible to analyze the RPC durability with a hybrid fiber mixture.

The nomenclature used in this study refers to WF for non-fiber samples; 100 (SF) for samples using only metal microfibers; 75/25 for samples with 75% use of metal microfibers and 25% polypropylene microfiber; and 50/50 for samples using 50% of each type of microfiber.

The samples used for accelerated carbonation testing were prismatic with dimensions 4x4x16 cm. In this procedure, the carbonation depth was determined by spraying a phenolphthalein indicator on 28 and 56 days. The carbonation chamber was adjusted to a CO_2 concentration of 5%. As for the reaction of the chemical indicator on the concrete, the color pink or fuchsia indicates high pH; if it remains colorless, it means that the pH decreased due to carbonation [32]. As the pH decreases (from approximately 13 to 9,5) the alkalinity of concrete also drops, resulting in reduced protection for the structure, making it more prone to attack by external agents.

Table 6: Concrete mix composition and fibers use percentages

Materials	WF		100/0		75/25		50/50	
Cement	1	0,44	1	0,44	1	0,44	1	0,44
Silica fume		0,37		0,37		0,37		0,37
Fly ash		0,19		0,19		0,19		0,19
Fine sand	2,39	1,02	2,39	1,02	2,39	1,02	2,39	1,02
Regular sand		0,86		0,86		0,86		0,86
Quartz		0,51		0,51		0,51		0,51
Water/binder relation	0,22							
Superplasticizer additive	0,03							
Viscosity modifier additive	0,01							
Steel fiber	0		100		75		50	
Polypropylene fiber	0		0		25		50	

The conditions of a marine environment were simulated, considering that its aggressiveness is due to the presence of salts, which act as electrolytes resulting in electrochemical corrosion. The test was carried out according to the specifications of ASTM B-117:2011 [33], with cubic samples of 50 mm side suspended inside the chamber. For the system operation, a cycle with moisture saturation was defined with sodium chloride (NaCl) concentration of 5% for 500 hours. Visual checking and mass determination of samples were performed every 100 hours.

Sulfuric acid (H2SO4), nitric acid (HNO3) and hydrochloric acid (HCl) were used to perform the chemical attack tests on concrete. Twelve prismatic specimens (4x4x16 cm) were molded for each mixture. Nine of these specimens were exposed to acid attack, three for each acid, and three were used as reference. Testing was performed according to ASTM C452:2010; ASTM C1012:2013 standards [34, 35]. Monteiro et al (2000) [36] highlight the importance of checking the flexural and compressive strength of samples in order to identify potential mechanical damages caused by immersion in acid, which was done in this study. The attack cycles lasted 14 days, including immersion, drying and mass and dimension verification. Each solution had three times the volume of samples and 5% concentration of acid. This experimental program had five cycles of immersion and drying. After the last cycle, samples were subjected to flexural tensile strength and compressive strength tests.

The 3D microtomography test was performed in one of the materials prepared with the main purpose of identifying the different constituent components and observe their arrangement along the generated geometry. A small 22 mm diameter and 30 mm length cylindrical sample was extracted from the original prismatic specimens.

3. RESULTS AND DISCUSSION

The carbonation of samples subjected to accelerated carbonation testing was verified by spraying phenolphthalein indicator. It is shown in Figure 03 the verifications on 28 and 56 days. In both cases, the RPC samples did not show signs of carbonation, not even in the peripheral zone. This is highly possible due to RPC high compactness and low number of voids or capillary pores, which results in increased protection against degrading agents. The color of the chemical indicator was darker in the mixes with fiber, possibly indicating a path for penetration of aggressive agents.

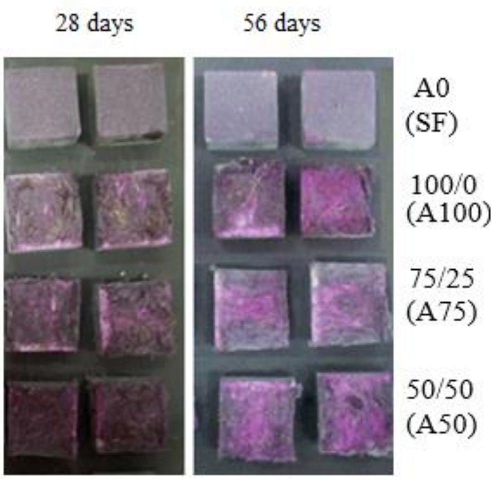

Figure 3: Depth of carbonation analysis at 28 and 56 days.

Similarly, Juanhong; Shaomin; Lin (2009) [9] proposed to perform this same analysis in samples of RPC, one of which contained only cement as a binder, and in the other fly ash was used. Due to the exposure to the accelerated carbonation chamber, for both compositions, the authors verified that the depth of damage was zero, that is, the test did not present any damage to the specimens analyzed.

After periods of exposure to salt spray, samples were subjected to compressive strength test. It was possible to check the effects of fiber hybridization in mixture's performance. Results are shown in Table 07.

Table 7: Compressive strength values and percentage loses

CYCLES	1° CYCLE – SALT SPRAY 100H		2° CYCLE – SALT SPRAY 100H		3° CYCLE – SALT SPRAY 100H		4° CYCLE – SALT SPRAY 100H		5° CYCLE – SALT SPRAY 100H	
Sample	fc (MPa)	Average reduct. (%)	fc (MPa)	Average reduct. (%)	fc (MPa)	Average reduct. (%)	fc (MPa)	Average reduct (%)	fc (MPa)	Average reduct (%)
WF	73,4	0,46	166,3	0,5	101,2	0,4	106,8	0,29	80,3	0,42
100/0	143,6	0,53	178,8	0,55	173,2	0,26	175,5	0,3	203,3	0,3
75/25	137,9	0,5	226,9	0,61	153,6	0,28	228,3	0,32	139,5	0,23
50/50	111,4	0,57	153,4	0,72	136,4	0,28	127,4	0,23	138,4	0,36

No linear relationship was observed between compressive strength and the progression of exposure cycles to salt spray. As for mass reduction of samples, it was more intense in the first stages of exposure, and final mass reductions were less than 1%.

It was also noted that the specimens were affected along the direction of the cut, in dry condition. Metal microfibers were exposed to corrosion because of the effects of the chamber, explaining why it was possible to notice that the most affected samples were those with 100% of microfiber use. Fibers were affected only in the unprotected area (cut) as internal fibers did not show signs of corrosion. Internal fiber protection is explained by the material impermeability, preventing the ingress of external deterioration agents.

Shaheen; Shrive (2006) [37] verified the action of ice and melt cycles in RPC mixture with the addition of carbon fibers, realizing that the use of microfibers in the composite provided greater resistance to cracking and durability, as showed in this study for salt spray cycles. Carbon fibers, however, presents high cost.

By the verification of the diffusion coefficient of chloride ions by calculation, Juanhong; Shaomin; Lin (2009) [9] found reduced diffusion, expected by the reduced presence of voids in the RPC compositions, in agreement with the results obtained in this study. Chuang; Huang (2013) [38] evaluated, through accelerated tests, the use of pozzolans could be a way to increase the resistance to diffusion of chlorides, agreeing with this study, since fly ash was used in the composition of the concrete.

Chemical attack results were grouped according the acid immersion. Figure 04 shows the mass loss values along the five cycles for samples subjected to the action hydrochloride acid. Mass reduction was observed for all samples in every cycle, though small and visually imperceptible. The samples with the lowest mass reduction values were those with a 50/50 mix, indicating that fiber hybridization is feasible and positive. The specimen with no fiber showed the highest mass reduction, proving indicating that fibers contribute to durability by hydrochloric acid attack. Figure 05 shows the results for nitric acid attack.

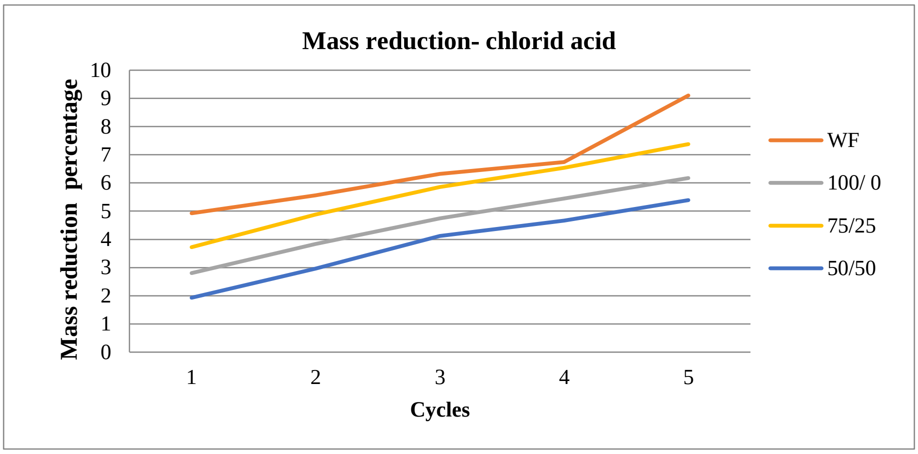

Figure 4: Mass reduction after chloride acid exposure

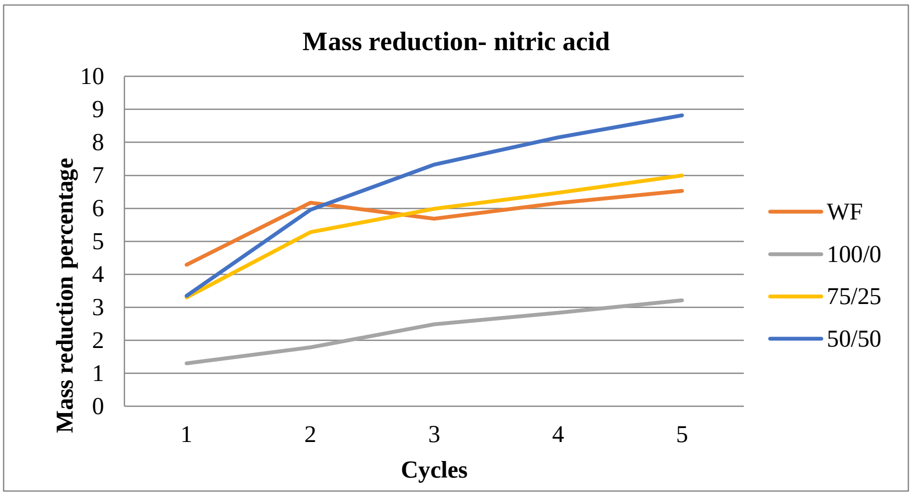

Figure 5: Mass reduction after nitric acid exposure

Similarly to what occurred with hydrochloric acid, all samples subjected to nitric acid showed mass loss and the 100% metal microfiber mixture had the best behavior. The sample with no fibers had mass loss similar to the 50/50 sample and lower than the 75/75 sample. Figure 06 shows mass loss by sulfuric acid attack.

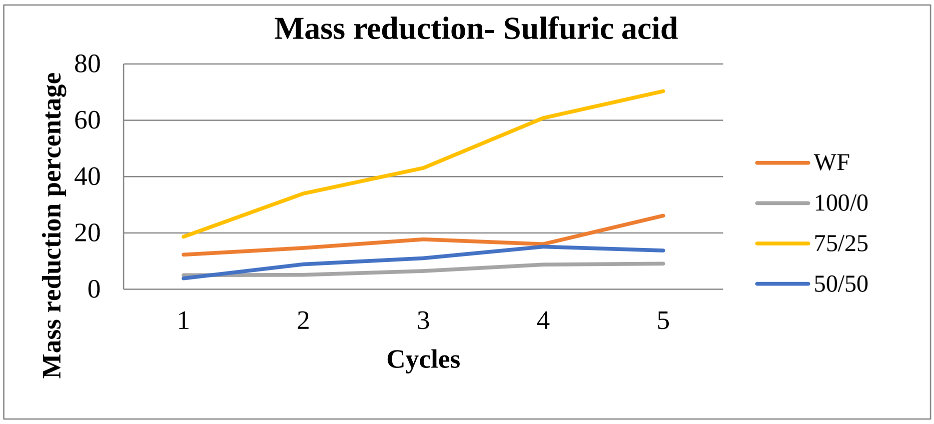

Figure 6: Mass reduction after sulfuric acid exposure.

In this stage, samples showed higher mass reduction than in the other acid attack tests, as shown in Figures 4, 5 and 6. It was similar for all mixes, but the 100% steel microfiber mix had the lowest mass loss. It was also possible to check, through visual inspection, that only after sulfuric attack the samples suffered expansion.

Following the chemical attack tests, tests for compressive strength and tensile strength in flexure were performed. Figure 07 shows the values for compressive strength. It's worth to highlight that samples indicated as reference samples are related to those which were not exposed to acid attack

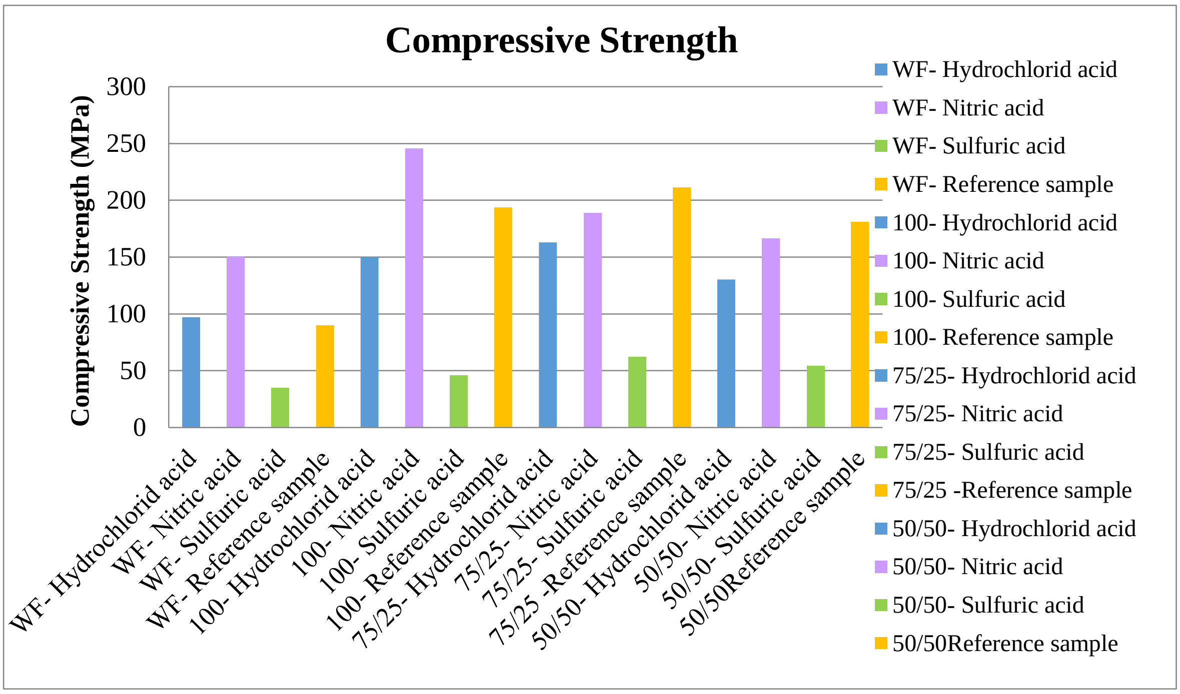

Figure 7: Compressive strength after samples exposition to acids.

The compressive strength test showed that samples suffered the most damage by sulfuric acid, whereas the nitric acid samples with no fibers and with 100% metal fibers had increased strength values as compared with the reference mixtures. Caggiano et al (2017) [39] found out that the use of microfibers slightly influences the compressive strength of composites, opposing to the results obtained and showed at Figure 7. Observing the values obtained by reference samples, the use of hybrid fibers (75/25) more than doubled the compressive strength. The values for tensile strength in flexure are shown in Figure 08.

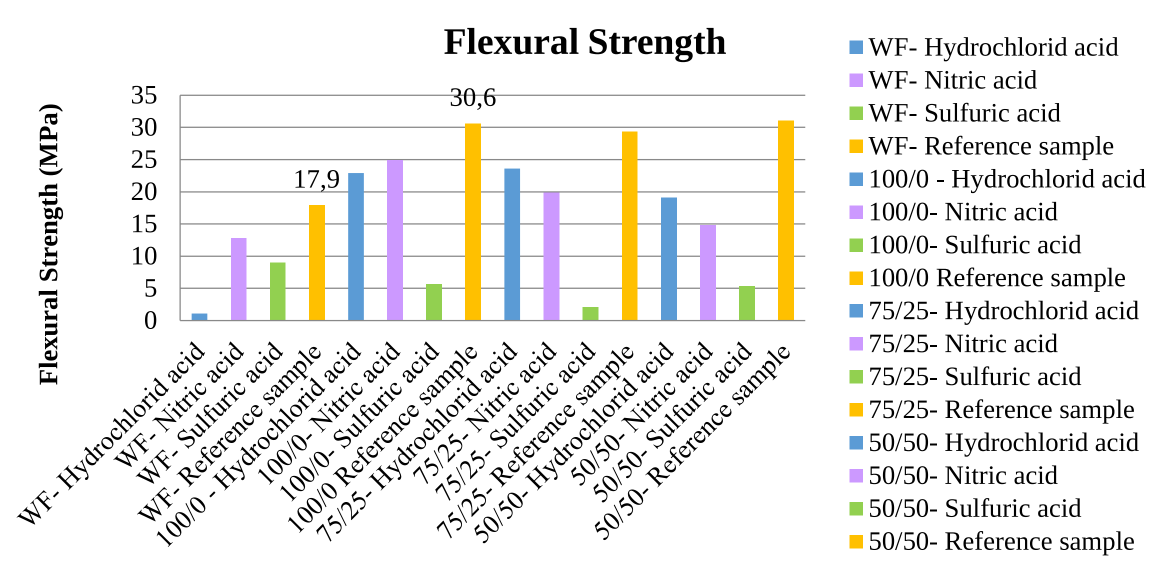

Figure 8: Flexural strength after samples exposition to acids.

The results showed that the damage caused to conventional concrete were very similar for the three acids. Also, sulfuric acid was the acid that showed the greatest potential for concrete degradation. The concrete samples not reinforced with fibers (NF) were more severely damaged by hydrochloric acid and, when compared with the sample not exposed to chemical attack, the average value is 57% lower. It is also possible to identify that the 100% steel fiber samples were more damaged by sulfuric acid attack. These tests showed increased specimens volume and surface deterioration, resulting in the formation of voids and affecting material's strength. In this mix, the damage caused by hydrochloric and nitric acids did not have a significant impact on the strength values of the samples. As for the samples above, the 75/25 and 50/50 mixes were more severely damaged by sulfuric acid, not showing extreme losses when attacked by the other acids.

When analyzing only the reference sample, to verify the effect of the fibers on the mechanical properties, it is highlighted that the interaction between the two types of microfibers was favorable. The greatest compressive strength occurred in the 75/25 proportion, and the higher tensile strength in the 100/0 and 50/50 samples. In a study with similar analysis, the authors Yu, Spiesz and Brouwers (2015) [23] realized that the best mechanical behavior occurred with hybrid mixture containing two sizes of steel fibers.

Figure 09 shows the sample with material differentiation obtained by microtomography analysis. The materials differ in density.

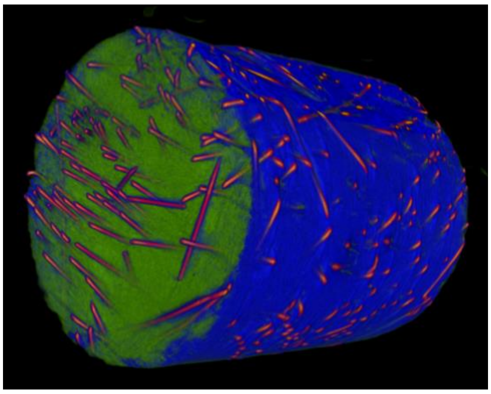

Figure 9: Microtomography 3D images of hybrid fibers samples.

The mostly green area represents the RPC cementitious matrix and the steel fibers can be seen by the reddish particles distributed along the sample. Because of their low density value, polymeric fibers (polypropylene) could not be identified by the equipment. In order to determine metal fibers distribution, imaging highlighting only the filaments was used. Figure 10 shows the high fiber concentration in the mixture for the small specimen removed.

An important finding concerns the distribution, concentration and orientation of fibers. The image on the right of Figure 10 refers to a single section of the sample. The generation of a fiber mesh, preferably homogeneous and random, with no flaws or inadequate fiber agglomeration, enhances the overall ductility of the composite and helps define the properties with isotropic tendencies. An interesting finding refers to the presence of fibers in that the polymeric filaments did not cause segregation of the mixture nor affected the distribution of metal fibers.

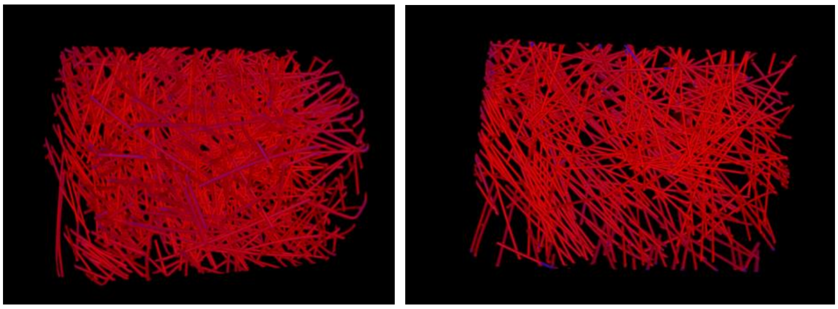

Figure 10: Metal fibers distribution

4. CONCLUSION

The accelerated carbonation test results were very similar for all different samples, where the phenomenon did not occur as verified on days 28 and 56 due to the low number of voids and high compactness. The reinforced RPC with metallic microfibers and polypropylene fibers samples showed high resistance against chemical attacks;

The results obtained with the salt spray test indicates RPC's resistance against attacks in the salt environment to which it was subjected, with reduced loss of mass values and variation of volume. No linear relationships with the values obtained for compressive strength or mechanical losses caused by the accelerated tests was identified. Fibers were damaged only in their cut area as there was no ingress of degrading agents in the areas protected by coating;

The results for chemical attack by acid immersion were considerably different, showing that hybrid fibers reinforced RPC samples were more resistant (except the 75/25 mix immersed in sulfuric acid). As observed, the immersion acid with the highest degradation potential was sulfuric acid. Therefore The reinforced RPC with metallic microfibers and polypropylene fibers samples satisfactorily met the durability expectation showing small signs of damage in the accelerated tests, suggesting reduced penetration of degrading agents.

The reinforced RPC with metallic microfibers and polypropylene fibers has a large potential to resist to some common chemical deterioration agents. This material seems able to be applied even in several conditions. The fiber hybridization improved the mechanical properties. It is possible to use those composites with greater performance than conventional concretes. The exposure to high temperatures was not assessed in this study

5. AKNOWLEDGMENTS

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