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ORIGINAL ARTICLE

Chloride ion penetration resistance in concretes produced with recycled fine aggregates and silica fume

Resistência à penetração de cloretos em concretos produzidos com agregado miúdo reciclado e sílica ativa

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Abstract: The use of by-products and recyclable materials in the production of concrete has become an interesting alternative to mitigate environmental impacts, especially those generated by the construction industry, as long as their mechanical and durability properties do not early compromise the service life of the structures. The resistance of concrete to the penetration of harmful agents, such as chloride ions, is an important property since it directly correlates with the performance, integrity, and durability of reinforced concrete structures. This study evaluate four concrete mixes were cast for aggressiveness class III of NBR 6118 [1] produced with 8% of partial replacement of Portland cement with silica fume, resulting of metallurgical production, and with 30% partial replacement of natural fine aggregates by recycled fine aggregate from fresh concrete waste, obtained from the concrete production process in concrete mixer trucks. At 28 days of age, the specimen was submitted to capillarity, mechanical resistance and chloride migration tests, according to the NT BUILD 492 standard [2]. In general, the results indicated that the proposed replacements improved mechanical properties and chloride ion penetration resistance, mainly with the incorporation of silica fume.

Keywords: concrete, chloride ion penetration, fresh concrete waste, recycled fine aggregates, silica fume.

Resumo: A utilização de subprodutos e materiais recicláveis na produção de concreto tornou-se uma alternativa interessante para mitigar os impactos ambientais, principalmente os gerados pela construção civil, desde que suas propriedades mecânicas e de durabilidade não comprometam precocemente a vida útil das estruturas. A resistência do concreto à penetração de agentes nocivos, tais como íons cloreto, é uma propriedade importante, pois se correlaciona diretamente com o desempenho, integridade e durabilidade das estruturas de concreto armado. Este estudo avaliou o desempenho de quatro misturas de concreto moldadas para classe de agressividade III da NBR 6118 [1] produzidas com 8% de substituição parcial de cimento Portland por sílica ativa, resíduo de produção metalúrgica, e com 30% de substituição parcial de agregados miúdos naturais por agregado miúdo reciclado de resíduo de concreto fresco, obtido do processo de produção de concreto em caminhões betoneira. Aos 28 dias de idade, as amostras foram submetidas aos testes capilaridade, resistência mecânica e de migração de cloretos, este, conforme preconiza a norma NT BUILD 492 [2]. De maneira geral, os resultados indicaram que as substituições propostas melhoraram as propriedades mecânicas e a resistência à penetração do íon cloreto, principalmente quando se utilizou a incorporação de sílica ativa.

Palavras-chave: concreto, penetração de íons cloreto, resíduo de concreto fresco por lavagem, agregado miúdo reciclado, sílica ativa.

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1 INTRODUCTION

The most used construction material is concrete, in addition, is the main responsible for the depletion of natural resources that give rise to its main constituent materials, such as aggregates, which are extracted from nature and, therefore, several countries are facing acute shortage of natural aggregates [3]–[5]. Cement production and aggregate processing for producing mortar and concrete are examples of activities with significant environmental impacts [6], [7].

In the last decades, there has been a growing worldwide concern about the high waste generation arising from the production activities of the construction industry sector. Thus, researchers have sought measures to improve this scenario. The development of more environmentally and economically viable production alternatives also considers the scarcity of landfills and their high maintenance costs. In this context, several studies have been developed to enable the reuse of construction waste and industrial byproducts in the production of new materials [8]–[11]. The aim is to reduce the consumption of natural resources, creating more appropriate alternatives for waste disposal [12].

Concrete is a composite material that contains a mixture of aggregates (coarse aggregate and fine aggregate) of different particle sizes, derivatives of the hydration process of cement or cementitious materials, water and occasionally, supplementary cementitious materials and additives [13], [14]. Its performance depends on the particle size distribution of aggregates, water and additive content and type and content of binder [14].

The Portland cement (PC) production, certainly the most important input for concrete, is considered the main responsible for carbon emissions, according to the World Business Council for Sustainable Development (WBCSD), it is estimated that to produce each ton of PC 650 kg of CO_2 are emit [15]–[17]. Portland cement is one of constituent of Portland concrete, largely influencing its physical and chemical properties. It consists of limestone, clay (or other silica inputs), alumina, and iron oxide, and it is produced at clinkering temperature [15], [18]. The production of this material includes chemical combinations, mainly in the solid state, forming: tricalcium silicate (C_3S); dicalcium silicate (C_2S); tricalcium aluminate (C_3A); and tetracalcium aluminate iron (C_4AF), after that, ground clinker [19].

Therefore, researchers have sought to replace Portland cement for other raw materials, such as, silica fume, an industrial by-product [20]–[22]. Silica fume is an ultrafine non-crystalline by-product of silicon processing. This material is a highly reactive pozzolan, dominantly composed of amorphous silicon dioxide with range 15-25 m^2/g of surface area. The use of silica fume as replacement affects fresh and hardened properties of concrete [23]. Silica fume stands out among the industrial byproducts used by the cement industry, being a pozzolan used at 5% to 10% clinker substitution [24]. Silica fume is a type of effective pozzolan due to the ability of this mineral addition to react with hydroxide calcium ($Ca(OH)_2$) generated during cement hydration to form Calcium Silicate Hydrate CSH and thus reduce the volume of large pores and capillaries seen in the cement paste [25], [26]. This by-product can be either incorporated into the cement – partially replacing the clinker – or used as mineral additive in the concrete mixture.

Aggregates are important constituents of concrete, making up at least 75% of the total volume and being uniformly dispersed in the cement paste [18]. Despite the low cost of obtaining it, aggregate extraction affects permanent preservation areas close to cities, and contributes to the silting up of rivers close to water collecting sites. Moreover, several locations already have depleted deposits [19].

Although the Brazilian technical standard NBR 9935 [27] deals with aggregates as generally inert granular materials, however, the properties of aggregates affect concrete characteristics such as apparent specific mass, porosity, granulometric composition, shape, and texture. Furthermore, the mineralogical composition, porosity, and specific surface of aggregates affect the compressive strength, durability, and consumption of cement in concrete [28].

Construction waste (CW) represents a significant volume of solid waste produced in urban areas worldwide. According to the Brazilian Association of Public Cleaning and Special Waste Companies – ABRELPE [29], the collection of construction and demolition waste in Brazil was of the order of 122,012 tons/day in 2018. This highlights the need for guidelines to effectively reduce waste impacts. The National Environment Council – CONAMA, through resolution number 307 [30], establishes guidelines, criteria, and procedures for construction waste management. This resolution defines recycled aggregate as a granular material originating from the processing of construction waste, meeting technical characteristics for application in buildings, infrastructure, sanitary landfills, or other engineering works.

Among the well-known construction waste, those produced from concrete plants stand out. They can be obtained in three ways: (i) from the cleaning of facilities and technological control laboratories, corresponding to hardened concrete waste; (ii) from the washing of concrete mixer trucks, carried out to prevent the concrete from adhering to the sides of the mixing bowl, which would impair homogenization efficiency; and (iii) from fresh concrete returned from job sites due to nonconformity or excess. In cases (ii) and (iii), the residual portion obtained is usually deposited in a set of settling tanks [7]–[10], [31].

In Brazil, recent research indicates that approximately 3% of the volume of concrete produced by ready mix concrete plants retuned to the plant, and about 55% of this volume of concrete is due to the adhered concrete from the drum of the concrete mixer [32], [33].

The material sedimented in the tanks is discarded in controlled landfills or can be reused through beneficiation processes such as comminution, where the particles are reduced to dimensions similar to those of aggregates, enabling their use in concrete production.

In volumetric fraction, the compositions of fresh concrete waste obtained by washing concrete mixer trucks are normally 70% or more of aggregates (which may be recoverable) and 30% or less of potentially unrecoverable paste, which is a combination of cement materials, water, partially hydrated cement or pozzolanic reaction products, a limited amount of fine aggregates and a relatively insignificant amount of unreacted additive [34]. The composition of the waste generated from the washing of fresh concrete have a strongly alkaline and calcium-rich character. This waste consists mainly of hydrated cement particles and aggregates [35]. Recent research evaluated the 20% replacement of natural fine aggregates for recycled fine aggregates obtained by crushing fresh concrete waste, that was recycled from washing operations, and demonstrate an average compressive strength was almost the same than conventional concrete, with relative difference approximately 1% [33]. Study that presented the dimensioning of concrete mixtures determined from the resistance classes and using partial replacement of natural aggregate by recycled aggregate, reveled improved mechanical properties, at levels above 19% of replacement, even with an increase of 10% to 20% in the w/c ratio [36].

Moreover, for replacement levels less than 20%, the benefits include compressive strength gain, less water absorption, and reduced chloride ion penetration. However, concrete workability decreases at replacement levels above 30% due to high water absorption of residue, higher water/cement ratio, resulting in loss of mechanical strength and higher chloride ion penetration [37], [38]. Researchers demonstrates that one of those solution to overcome the negative effects on concrete properties due to partial the partial replacement of the natural aggregates with the recycled ones, is the use of silica fume, a pozzolanic material [39], [40]. Contents below 10% addition of cement weight by silica fume is adequate to enhance the mechanical properties in concretes with recycled aggregate and can present a significantly increased the strength parameters and slightly decreased the permeability parameters, where the 28 days compressive strength, splitting tensile strength and the flexural tensile strength increased by 100%, 20% and 20.3%, respectively [40].

Therefore, recycled fine aggregates are a promising alternative for the production of more sustainable concrete, with satisfactory performance as long as durability and mix proportioning requirements are well specified [41].

Currently, the Brazilian standard NBR 7211 [42] only covers the reuse of aggregates recovered from fresh concrete waste (item 1.6), recommending the use of up to 5% and suggesting the proper granulometric characterization of the aggregate.

Durability correlates with the behavior of materials when subject to different actions, whether of physical, chemical, biological, or structural origin. Among such actions, corrosion of the reinforcement, which can be induced by the attack of chloride ions, is a major mechanism of degradation, reducing the service life of reinforced concrete structures exposed to aggressive environments [43]. A change in the passive film takes place when the deleterious agents reach the steel surface, leaving it prone to corrosion. Chloride ions can be found in concrete in different ways, such as: (i) chemically combined (chloroaluminates); (ii) physically adsorbed on the surface of capillary pores; and (iii) free, in the concrete pore solution [44]. Therefore, the use of materials that provide reactions that reduce the availability of free chloride ions in the concrete, or that form a less porous cementitious matrix and prevent the penetration of this harmful agent into the reinforcement, is essential for the development of a durable concrete.

This study evaluates partial replacement of Portland cement for silica fume and partial replacement of natural fine aggregates for recycled fine aggregates obtained from the processing of fresh concrete waste. The main focus is on the mechanical properties and durability of concrete, especially with regard to chloride ion penetration resistance.

2 MATERIALS AND EXPERIMENTAL PROGRAM

2.1 Materials

To carry out this study, 116 cylindrical concrete specimens (10 cm x 20 cm) were produced, which were used in accordance with the requirements recommended by the standards described in this experimental program.

The materials used for concrete production were Portland cement of high initial strength (type CPV-ARI) (binder), silica fume (pozzolan), washed natural sand of medium particle size (natural fine aggregate), fresh concrete waste from washing (recycled fine aggregate), No. 1 crushed stone (coarse aggregate), tap water, and superplasticizer MC-PowerFlow 1180 as additive. The technical specifications of the materials are presented below.

2.1.1 Binder

Concrete production in this study took place with the use of Portland cement of high initial strength (type CPV-ARI), according to the requirements of NBR 16697 [45]. The physical and chemical characterization of the material are shown in Table 1.

Table 1. Characterization of the Portland cement (CPV-ARI)

Property analyzed		Test method	Results	NBR 16697 limits [45]
		Physical characterization		
Fineness – Blaine Method (cm^2/g)		NBR 16372 [46]	4597	≥ 3000
Setting time		NBR NM 65 [47]	131	≥ 60
Compressive strength (MPa)	1 day	NBR 7215 [48]	27	≥ 14
	3 days		41.4	≥ 24
	7 days		46.2	≥ 34
	28 days		51.4	-
		Chemical characterization		
MP 1000º C		NBR NM 18 [49]	5.63	≤ 6.5
SiO_2		NBR 14656 [50]	18.14	-
Insoluble residue		NBR NM 15 [51]	0.95	≤ 3.5
Al_2O_3		NBR 14656 [50]	4.90	-
Fe_2O_3			2.99	-
CaO			64.58	-
MgO			0.73	-
SO_3			2.53	≤ 4.5
K_2O			0.78	-
CO_2		NBR NM 20 [52]	4.87	≤ 5.5
28 days C_3A (theoretical)		Bogue equation	7.57	-

Source: Test report provided by the manufacturer (2018).

2.1.2 Supplementary cementitious materials

Silica fume was used as pozzolanic material, being incorporated into the mixtures as a partial replacement of Portland cement. This material was obtained according to the requirements of NBR 13956-1 [53]. Its chemical and physical characterization are shown in Table 2.

Table 2. Characterization of the silica fume used

Definition	Unit	Limit	Analysis
	Chemical characterization		
SiO_2	%	min. 85.0	96.30
Moisture	%	max. 3.0	0.19
Loss on ignition	%	max. 6.0	1.96
Na_2O alkali equivalent	%	max. 1.5	0.55
	Physical characterization		
45 µm sieve residue	%	max. 10.0	< 10
Apparent density	kg/m^3	min. 350	478
Solid content – aqueous dispersion			
Pozzolanic activity index	%	min. 105	> 105
Specific mass	g/cm^3	-	2.18
Specific area (Blaine)	m^2/g	-	19380

Source: Test report provided by the technical adviser of the manufacturer (2018).

2.1.3 Fine aggregate

2.1.3.1 Natural fine aggregate

The fine aggregate used is of natural origin. Table 3 presents the main physical characteristics of the material.

Table 3. Physical characterization of the natural fine aggregate used

Property analyzed	Test method	Results
Specific mass (g/cm^3)	NBR NM 52 [54]	2.62
Loose unit mass (g/cm^3)	NBR NM 45 [55]	1.43
Compacted unit mass (g/cm^3)		1.68
Powdery material content (%)	NBR NM 31 [56]	1.61

The granulometric distribution of the natural fine aggregate was obtained according to the recommendations of NBR 7211 [42] and is shown in Figure 1.

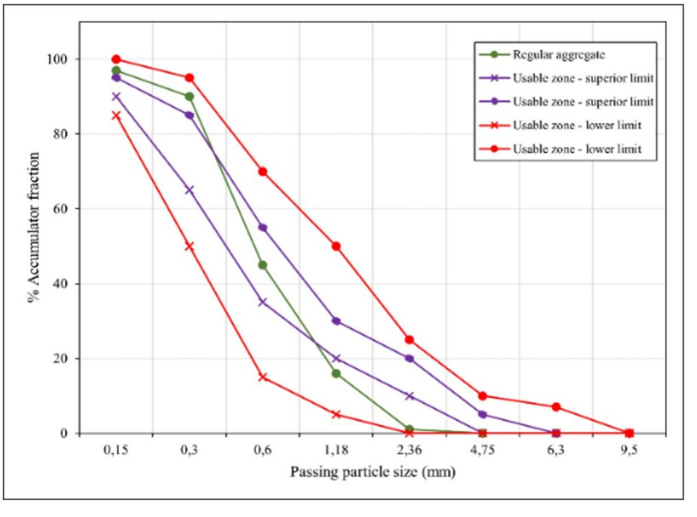

Figure 1. Granulometric curve of the natural fine aggregate

2.1.3.2 Recycled fine aggregate

The recycled fine aggregate used was obtained through the processing of fresh concrete waste sedimented in settling tanks after the washing of concrete mixer trucks of a concrete.

This residual material is from setting tanks consists, predominantly, of concretes produced in concrete plant, with a compressive strength resistance class from 25 to 35 MPa. When returning to the plant, concrete mixer trucks usually unload any fresh concrete leftovers. Then, their mixing bowls are washed internally with water under the rotation of the concrete mixer, which removes the concrete adhered to these structures. Finally, all material (water and fresh concrete waste) is discharged into a set of settling tanks. The wastewater obtained from the decantation is reused for washing elements and installations of the concrete plant. To carry out this study, the waste sedimented at the bottom of the first tank - basically composed of residual aggregate of the original concrete and cement paste - was collected, transported, and discharged into a mining area attached to the concrete plant.

As a strategy for processing the fresh concrete waste obtained, was opted for comminution with the aid of a crushing set (model Asteca) equipped with a jaw crusher with 1” (25.4 mm) and 7/8” (22.23 mm) mesh opening and a cone crusher with 1/2” (12.7 mm) and 1/4” (6.35 mm) mesh opening. The mining company attached to the concrete plant carried out this process. Thus, was obtained a granular material with dimensions similar to those of fine aggregates. Figure 2 shows the process for obtaining this material.

Figure 2. Stages of obtaining and processing the recycled aggregate. (a) Settling tanks; (b) Material discharge area; (c) Crushing; (d) recycled fine aggregate.

The physical characterization and granulometric curve of the recycled fine aggregate, obtained according to the recommendations of NBR 7211 [42], are shown in Table 4 and Figure 3, respectively.

Table 4. Physical characterization of the recycled fine aggregate.

Property analyzed	Test method	Result
Specific mass (g/cm^3)	NBR NM 52 [54]	2.25
Loose unit mass (g/cm^3)	NBR NM 45 [55]	1.08
Compacted unit mass (g/cm^3)		1.15
Powdery material content (%)	NBR NM 31 [56]	26.45

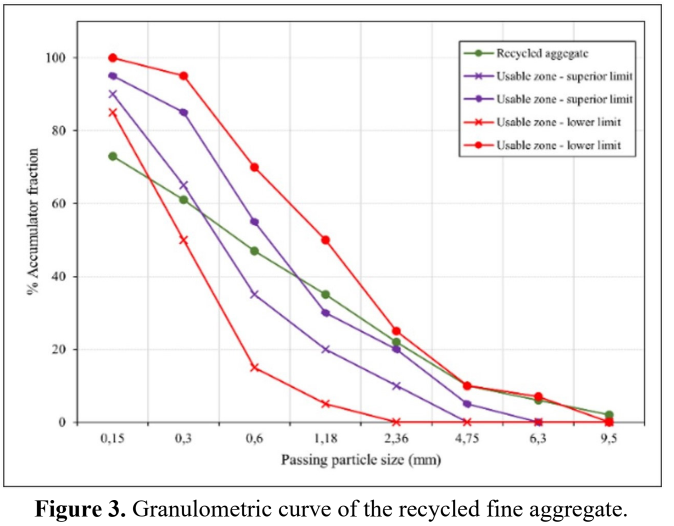

Figure 3. Granulometric curve of the recycled fine aggregate.

Finally, the Figure 4 shows the aspects of the natural fine aggregates and recycled fine aggregates of fresh concrete waste, as well as the aspect of the aggregates with a content of 30% of replacement of fine natural aggregates by recycled fine aggregates.

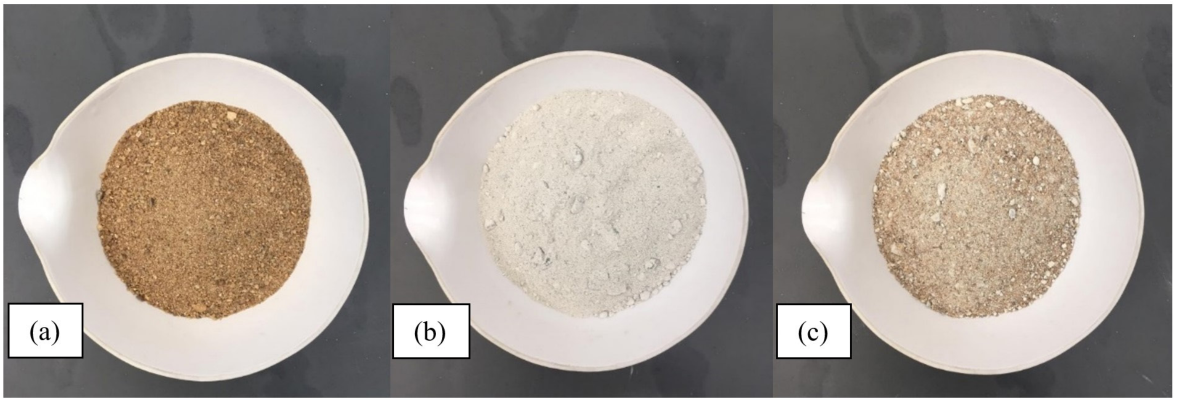

Figure 4. Aspect of the materials used as fine aggregates. a) natural fine aggregate; b) recycled fine aggregate; c) fine aggregate with 30% of replacement of fine natural aggregates by recycled fine aggregates

2.1.4 Coarse aggregate

The coarse aggregate comes from the crushing and processing of gneiss by a mining company located in southeastern Brazil. The physical characterization and granulometric distribution were obtained following the recommendations of NBR 7211 [42], are shown in Table 5 and Figure 5, respectively.

Table 5. Physical characterization of the coarse aggregate

Properties	Test method	Results
Specific mass (g/cm^3) – specific mass of the dry aggregate (g/cm^3)	NBR NM 53 [57]	2.67
Specific mass of the saturated aggregate in dry surface (g/cm^3)		2.64
Apparent specific mass (g/cm^3)		2.64
Water absorption (%)	NBR NM 45 [55]	0.45
Compacted unit mass (g/cm^3)		1.45
Loose unit mass (%)		1.32

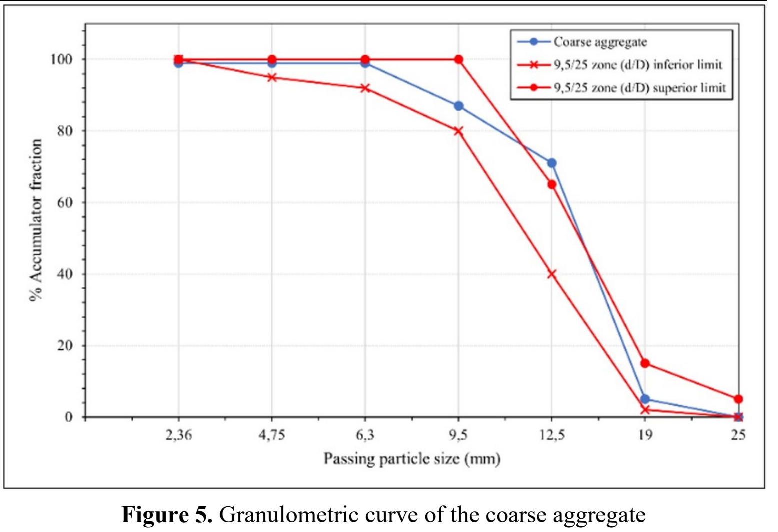

Figure 5. Granulometric curve of the coarse aggregate

2.1.5 Additive

The use of the superplasticizer MC-PowerFlow 1180 as additive was necessary to provide similar workability conditions between the concretes produced for maintain the same water/cement ratio (w/c = 0.55) in all proposed mixtures.

2.1.6 Water

The water used for concrete production comes from the local public supply network and meets the conditions required by the NBR 15900-1 standard [58].

2.2 Experimental program

The concrete mixtures should meet the Brazilian standard NBR 6118 [1]. This class considers exposure to high environmental aggressiveness and a high risk of structure deterioration, as occurs in marine and industrial environments.

Therefore, the water/cement ratio was set at 0.55, and the concrete strength class was set as C30 (fck ≥ 30 MPa). In addition, concretes were dosed in such a way that their consistencies were kept within an interval of (12 ± 1) cm when evaluated by means of the Cone trunk abatement test according to NBR NM 67 [59].

Four different concrete mixtures were proposed, namely: reference mixture (RC); mixture with partial replacement (8%) by mass of Portland cement (type CPV–ARI) with silica fume (SFC); concrete mixture with partial replacement (30%) by mass of natural fine aggregate with recycled aggregate (RAC); and concrete mixture with partial replacement (8%) by mass of Portland cement (type CPV–ARI) with silica fume and with partial replacement (30%) by mass of natural fine aggregate with recycled aggregate (SRAC). Table 6 shows the composition of the four concrete mixtures produced.

Table 6. Concrete mixtures produced.

Concrete mixture	W/C	Cement (kg/m^3)	Silica fume (kg/m^3)	Fine aggregate (kg/m^3)	Recycled fine aggregate (kg/m^3)	Coarse aggregate (kg/m^3)	% Superplasticizer addition in cement mass	Water (liters)
RC	0.55	340	0	834	0	935	0.09	187
SFC	0.55	312.8	27.2	834	0	935	0.44	187
RAC	0.55	340	0	583.8	250.2	935	0.42	187
SRAC	0.55	312.8	27.2	583.8	250.2	935	0.82	187

Specimens were molded according to the specifications of Brazilian standard NBR 5738 [60]. All concrete specimens were demolded after a period of 24 hours of casting, identified and cured in water until the tests ages.

2.2.1 Tests

2.2.1.1 Chemical characterization of recycled fine aggregate

With the aim of carrying out the Chemical characterization of the recycled fine aggregates used in this research, samples of this material were submitted to X-ray Fluorescence and X-ray Diffraction tests.

2.2.1.2 Axial compressive strength

Concrete specimens were submitted to the axial compressive strength test at the ages of 28, 63, and 91 days after casting, in accordance with the procedure recommended by Brazilian standard NBR 5739 [61].

2.2.1.3 Static compressive elasticity modulus

The static compressive elasticity modulus of the concretes was evaluated at 28 days old. Five specimens of each mixtures were removed from the saturated cure and tested according to the Brazilian standard NBR 8522 [62].

2.2.1.4 Capillary water absorption

The capillary water absorption was assessed as suggested by the Brazilian standard NBR 9779 standard [63], when they were 28 days old of specimens.

2.2.1.5 Chloride ion penetration resistance

The chloride ion penetration resistance of the produced concretes was assessed when they were 28, 63, and 91 days old. The assessment was based on the migration coefficients of chloride ions obtained through the accelerated migration test, in nonstationary state, as prescribed by the Finnish standard NT BUILD 492 [2].

The samples were previously prepared and then conveniently placed on plastic supports, slightly inclined, inside the two test chambers containing, each of them, a cathodic solution of sodium (NaCl) dissolved in 10 liters of water, as shown in the schematic apparatus proposed by the standard, as shown in Figure 6.

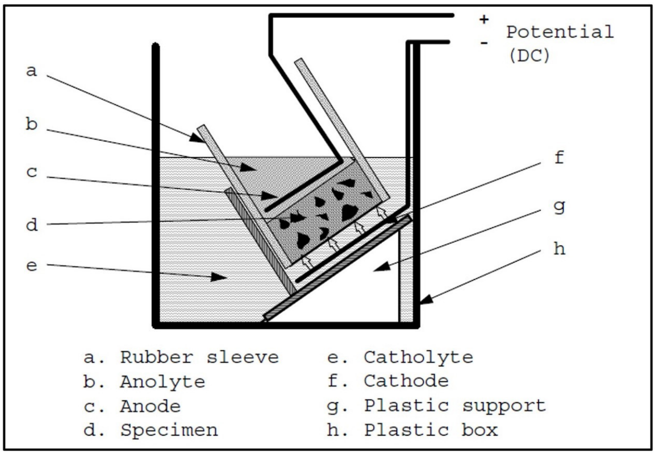

Figure 6. Arrangement of the migration set-up. Source: Nordtest [2]

Subsequently, electrodes were connected in order to interconnect the rods and metal plates of the test apparatus to a switch box which, in turn, was connected to a power supply responsible for generating an electrical potential difference between the cathode and the anode, starting the test. After the period predetermined by the standard, the samples were removed from the solution and sectioned, so that it was possible to spray a silver nitrate solution on the newly sectioned internal surfaces in order to obtain evidence of the penetration of chlorides in the samples, identified by a clearly visible white precipitate, which were measured, directly on the surface of the sample, from the center at intervals of 10 mm to obtain seven values and calculate the chloride migration coefficients.

A classification of chloride penetration resistance in concrete at 28 days was proposed by Gjørv [64] based on the chloride ion migration coefficients obtained in the tests, as shown in Table 7.

Table 7. Chloride ion migration resistance.

Chloride ion migration coefficient at 28 days (D_{28} x $10^{-12}m^2/s$)	Chloride penetration resistance
$D \geq 15$	Low
$10 < D < 15$	Moderate
$5 \leq D \leq 10$	High
$2.5 < D \leq 5$	Very high
$D < 2.5$	Extremely high

Source: Adapted from Nilsson et al. *apud* Gjørv [64].

3 RESULTS AND DISCUSSIONS

The statistical analysis of the results was performed by analysis of variance (ANOVA) to assess the importance of one or more factors by comparing the means of response variables at different factor levels.

3.1 Chemical characterization of recycled fine aggregate

The chemical composition of the fresh concrete waste was determined by X-ray fluorescence (XRF) test, with the aid of a spectrometer (PANalytical, Zetium model) equipped with an X-ray tube with a power of 3 kW. For this, the material was quartered, and a sample was selected and ground until 100% passed through the #200 sieve (75µm). The average levels of the main oxides for the recycled fine aggregate are shown in Table 8.

Table 8. Chemical composition of the fresh concrete waste obtained by X-ray fluorescence

Chemical species	Content
% Loss to fire – MP 950 ºC	6.24
% Silicon dioxide – SiO_2	61.70
% Aluminum oxide – Al_2O_3	13.14
% Iron oxide – Fe_2O_3	2.47
% Calcium oxide – CaO	9.49
% Magnesium oxide – MgO	0.96
% Sulfuric anhydride – SiO_2	0.38
% Sodium oxide – Na_2O	3.65
% Potassium oxide – K_2O	2.03

Source: Test report provided by the technical adviser of the manufacturer (2020).

Silica (SiO_2) is the main constituent oxide of the fresh concrete waste under study, accounting for 61.70% of its content. The origin of this material correlates mainly with the natural aggregates of concrete and mortar. Alumina (Al_2O_3) and calcium oxide (CaO) are the other most representative oxides, corresponding to a content of 13.14% and 9.49%, respectively, being essential components of Portland cement. Such situation is specific for the samples under study since the mineralogy of aggregates may vary depending on both the extraction region and the concrete molding site.

The investigation of the crystalline phases of the fresh concrete waste, was carried out by means of powder polycrystalline X-ray diffraction (XDR), on a Bruker equipment, model D8 Advance, using Cu Kα radiation. The XRD patterns were collected in the 2θ range of *5–90*°, using a scan velocity of $0.6° s^{-1}$. The analysis method is based on the comparison of the values of interplanar distances and peak intensities in the diffractograms of the analyzed samples, using the standard PDF-2 database of the ICDD (International Centre for Diffraction Data®). The identification of the crystalline phases of fresh concrete waste were performed using the number files: 2-466 (Glauconite) ((K, Na) (Fe^{+3}, Al, Mg)$_2$ (Si, Al)$_4$O$_{10}$(OH)$_2$), 12-583 (Lizardite) ((Mg, Al)$_3$(Si, Al)$_2$O$_5$(OH)$_4$), 41-1480 (Albite) ((Na, Ca) Al(Si, Al)$_3$O$_8$), 72-1114 (Microcline) ($KAlSi_3O_8$), 81-2027 (Calcite) ($CaCO_3$), 2-969 (Portlandite) ($CaO.H_2O$), 2-731 (Hillebrandite) ($Ca_2SiO_4.H_2O$), 2-392 (Calcium Aluminum Oxide) ($Ca_3Al_{10}O_{18}$), 9-327 (Rankinite) ($Ca_3Si_2O_7$), and 4610-45 (Quartz) (SiO_2) were attributed to the crystalline phases present in the washing residue.

It was observed that the phases identified is the X-ray Diffraction test came from the aggregates used to produce the original concrete, residual cement paste and anhydrous cement [37], [65]. The diffractogram obtained is shown in Figure 7.

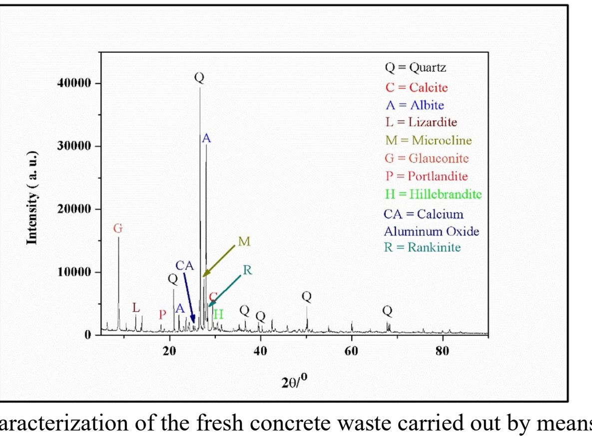

Figure 7. Mineralogical characterization of the fresh concrete waste carried out by means of X-ray diffractometry

The results observed are consistent with the results observed in the identification of oxides by X-ray fluorescence analysis. The high levels of silicon and aluminum oxides identified through the X-ray fluorescence teste are justified by the presence of albite, lizardite, microcline and glauconite. In addition, the predominance of silica is also due to the presence of quartz, hillebrandite and rankinite. The aluminum oxide content identified is also related to the presence of calcium aluminate. The calcium oxide content identified is related to the presence of calcite, albite, portlandite, hillebrandite, calcium aluminate and rankinite. The other oxides found are present on some of the minerals that constitute the aggregates used to produce the original concrete.

3.2 Mechanical strength

The mechanical characterization of the concretes was carried out through the tests of axial compressive strength and static compressive elasticity modulus, the Table 9 presents the results of the mechanical tests, which are presented graphically in Figure 8 for the tests of axial compressive strength and elasticity modulus, respectively.

Table 9. Results of mechanical strength tests.

Concrete	Cone trunk abatement (cm)	Elasticity modulus (GPa)	Axial compressive strength (MPa)		
		28 days	28 days	63 days	91 days
RC	11	29 ± 1	37.4 ± 0.3	38.54 ± 0.6	39.64 ± 0.6
SFC	11.5	27.60 ± 1	43.9 ± 1	48.43 ± 1	50.73 ± 0.7
RAC	12	29.8 ± 1	44.56 ± 1.71	46.20 ± 1.06	48.47 ± 0.6
SRAC	13	26.80 ± 1	45.43 ± 0.8	47.27 ± 1.1	50.20 ± 0.5

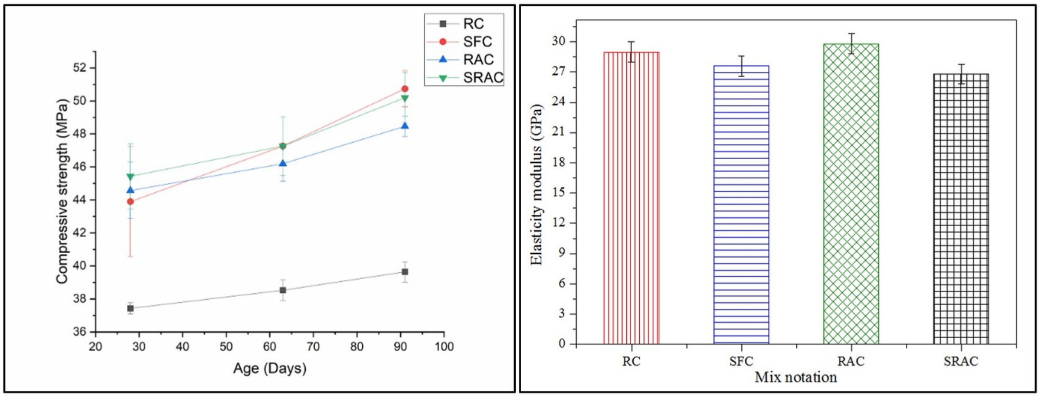

Figure 8. Results of axial compressive strength and elasticity modulus.

The results show that the reference mixture (RC) had the lowest compressive strength, at all ages, in comparison to the other concretes produced. This fact suggests that the incorporation of both silica fume and recycled fine aggregate improved this property. At the most advanced age analyzed (91 days), the SFC mixture, with partial replacement of Portland cement with silica fume (8%), and the SRAC mixture, with 8% silica fume and 30% recycled fine aggregate, showed a resistance 28% greater than the reference mixture. Still at this age, the RAC mixture, with partial replacement of natural aggregate with recycled aggregate (30%), showed an increase of 22% in compressive strength in comparison with RC.

As previously observed in the literature [40], [25], the concretes produced with silica fume showed higher values of axial compressive strength in the different ages analyzed. These improvements stem both from pozzolanic reactions, with the formation of more resistant hydration products, and from refinement of the concrete microstructure [20]. Likewise, the incorporation of the recycled fine aggregate implied an increase in the degree of packaging of the cementitious matrix, providing conditions for increasing compressive strength.

Moreover, all the concretes produced, with a water/cement ratio of 0.55, showed compressive strength greater than 30 MPa. This confirms the possibility of placing them in the environmental aggressiveness class type III of NBR 6118 [1].

Table 10 presents the analysis of variance (ANOVA) of the statistical model applied to the results of compressive strength.

Table 10. Analysis of variance applied to the results of compressive strength.

Variable	Degrees of freedom	Sum of squares	Sum of mean squares	F-value	p-value
Mixture	3	606.6	202.21	43.66	< 0.001
Age (days)	2	53.6	26.79	5.78	0.008
Mixture/Age (days) interaction	6	178.4	29.73	6.41	< 0.001
Residues	24	111.1	4.63	-	-

All factors were significant at 5% and 1% probability levels for compressive strength.

The statistics analysis built with the variables Mixture and Age (days) explains 88.30% of the total variability. The variable Mixture is the factor that explains most of the in the data, accounting for 63.88% of the total variability in compressive strength, followed by the Mixture-Age (days) interaction, with 18.78% of the total variability. The variable Age explains 5.64% of the total variability in compressive strength.

The interactions between the factors Mixture and Age (days) were significant at 5% and at 1% probability levels.

The assumptions of the statistical analysis were met, and the residues follow a normal distribution. The graph of the behavior of the average compressive strength by Mixture and Age (days) (Figure 8) shows a substantial distance between the averages of the RC mixture in comparison to the other concretes. This concrete had the worst results for average compressive strength at all ages.

The behavior of the average compressive strength agrees with the significance of the interaction in the model. Mixtures SFC (8% silica fume) and SRAC (8% silica fume and 30% recycled fine aggregates) had similar performance, with lower compressive strength at 28 days and greater results at 63 days in comparison to the RAC mixture. These mixtures remained with the best performance at 91 days. Since the F value of the analysis of variance was significant, the Tukey test for multiple comparisons was performed to determine differences between treatment averages. The comparisons showed no significant differences in compressive strength between these two mixtures.

3.3 Capillary water absorption

The evaluation of capillary water absorption consisted of measuring the amount of water absorbed, per unit area, after 3, 6, 24, 48, and 72 hours of exposure. The results are shown in Table 11 and graphically in Figure 9.

Table 11. Results of capillary water absorption at different exposure periods.

Concrete mixture	Capillary absorption (g/cm^2)				
	After 3 hours	After 6 hours	After 24 hours	After 48 hours	After 72 hours
RC	0.16 ± 0.06	0.22 ± 0.17	0.45 ± 0.17	0.65 ± 0.22	0.80 ± 0.26
SFC	0.11 ± 0.17	0.16 ± 0.07	0.31 ± 0.10	0.41 ± 0.12	0.48 ± 0.12
RAC	0.20 ± 0.05	0.27 ± 0.06	0.55 ± 0.12	0.76 ± 0.15	0.91 ± 0.17
SRAC	0.15 ± 0.02	0.21 ± 0.04	0.43 ± 0.05	0.58 ± 0.04	0.68 ± 0.04

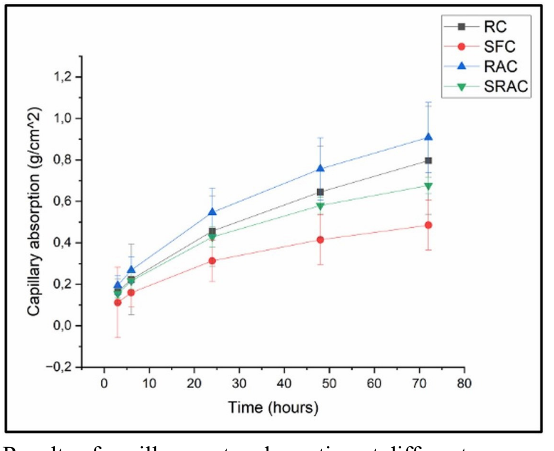

Figure 9. Results of capillary water absorption at different exposure periods.

The results of capillary water absorption tests at the age of 28 days revealed that the partial replacement of Portland cement with silica fume decreased capillary water absorption in the produced concrete (SFC and SRAC) in comparison to the RC and RAC, without the presence of silica fume in the mixture. This fact suggests that this pozzolanic addition refined the pores and formed a denser cementitious matrix, as verified in other studies [21], [22], [24], [25].

According to Molin [26], silica fume has high reactivity and a large specific surface, generating fast reactions. In addition to the pozzolanic effect, silica fume has a filler effect, providing an internal reorganization of the voids. Therefore, such characteristics can provide a considerable reduction in the size of the pores and, eventually, their obstruction, preventing the passage of fluids. Considering the specimens analyzed, the partial replacement of natural fine aggregate by recycled fine aggregate increased capillary water absorption, with the RAC mixture presenting the highest results of water absorption. These results indicate that the incorporation of the recycled fine aggregate formed a concrete with more permeability, that is, with a less refined pore structure.

Table 12 presents the analysis of variance (ANOVA) of the statistical model applied to the results of capillary water absorption.

Table 12. Analysis of variance applied to the results of capillary water absorption.

Variable	Degrees of freedom	Sum of squares	Sum of mean squares	F-value	p-value
Mixture	3	0.89	0.29	13.20	<0.001
Time (hours)	4	5.53	1.38	61.48	<0.001
Residues	112	2.52	0.02	-	-

All factors were significant at 5% and 1% probability levels for capillary water absorption. The model built with the variables Mixture and Time (hours) explains 71.83% of the total variability in capillarity absorption. The variable Time (hours) is the factor that explains most of the variability in the data, accounting for 61.86% of the total variability, while the variable Mixture explains 9.96% of the total variability. Both have a very low p-value, close to zero. The interaction between the factors Mixture and Time (hours) was not statistically significant. The assumptions of the statistical analysis were met, and the residues follow a normal distribution.

The graph presented in Figure 9 shows the behavior of the average capillary water absorption by Mixture and Time (hours). The variable Time (hours) proved to be significant and followed the same pattern (trend of capillary water absorption increase over time), except for the SRAC mixture. There was a more marked increase in capillary absorption between the periods of 6 and 24 hours in all mixtures. The SFC mixture performed better at all periods, followed by the SRAC, RC, and RAC mixtures. The SFC mixture, with partial replacement of Portland cement with silica fume (8%), presented average results significantly different from the others, as also evaluated by the Tukey test. The RAC mixture, with partial replacement of natural fine aggregates with recycled aggregates (30%), presented average results significantly different from the SRAC mixture, also produced with recycled aggregates but with partial replacement of Portland cement with silica fume (8%). However, its average results did not differ significantly from the results of the RC mixture.

3.4 Chloride ion penetration resistance

Table 13 and Figure 10 show the penetration depths and chloride ion migration coefficients of concretes aged 28, 63, and 91 days.

Table 13. Results of penetration depths and chloride ion migration coefficients.

Concrete mixture	Average penetration depth at 28 days	Migration coefficient at 28 days	Average penetration depth at 63 days	Migration coefficient at 63 days	Average penetration depth at 91 days	Migration coefficient at 91 days
	(mm)	D_{63}(x 10^{-12}/s)	(mm)	D_{63}(x 10^{-12}/s)	(mm)	D_{91}(x 10^{-12}/s)
RC	30 ± 1	22 ± 1	34 ± 1	19.5 ± 0.6	32 ± 1	18.5 ± 0.7
SFC	12.7 ± 0.8	4.3 ± 0.2	16.4 ± 0.7	4.6 ± 0.6	13.9 ± 0.8	3.8 ± 0.5
RAC	30 ± 2	16.9 ± 0.9	26 ± 1	14.9 ± 0.3	26.1 ± 1.1	15.1 ± 0.7
SRAC	15.6 ± 0.5	5.5 ± 0.2	13.8 ± 0.6	4.7 ± 0.7	13.1 ± 0.7	4.5 ± 0.6

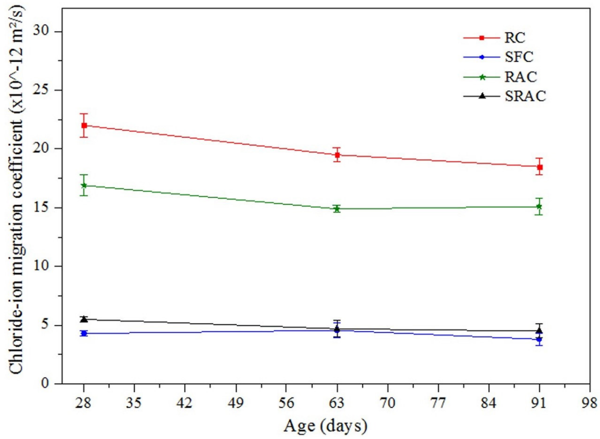

Figure 10. Graph of average chloride ion penetration coefficient.

The chloride ion penetration resistance evidenced in the cross sections of the samples after spraying of a silver nitrate solution, as shown in Figure 11.

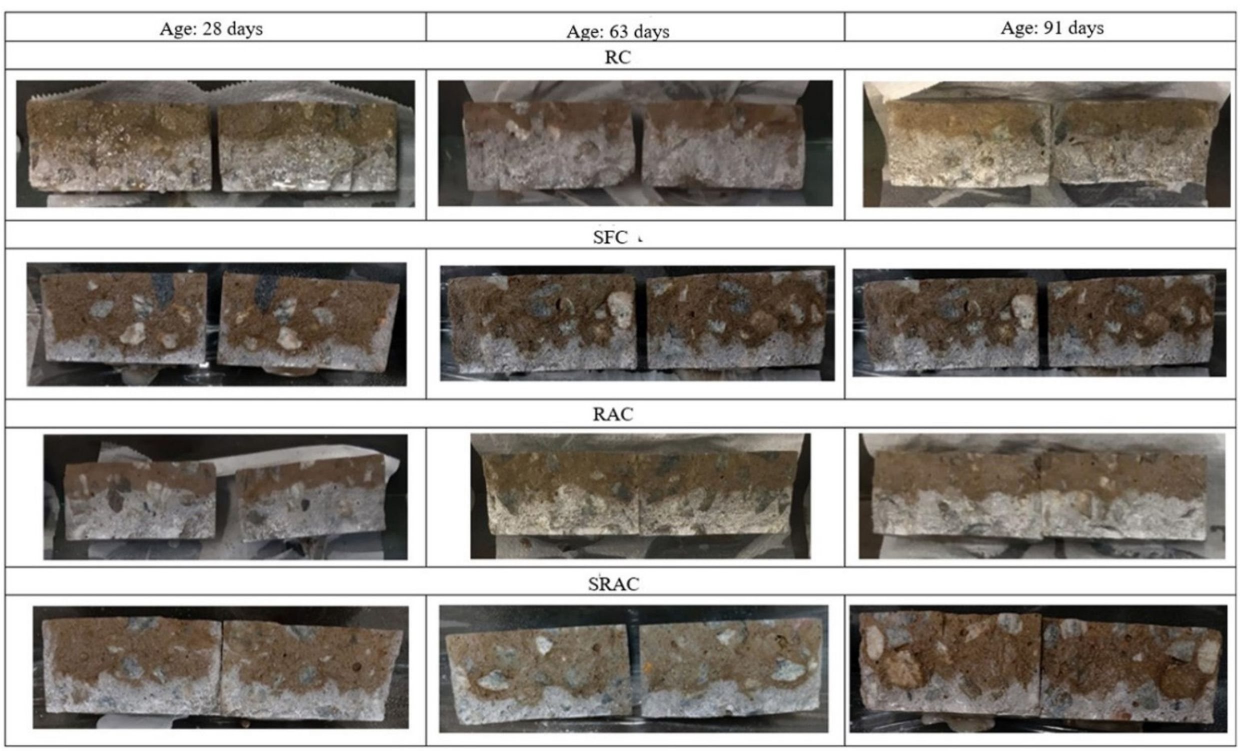

Figure 11. Evidence of chloride ion penetration in concrete samples at different ages.

The results show that partial replacement of Portland cement with silica fume (SCF and SRAC mixtures) directly reduced chloride ion migration coefficients at all ages in comparison to the mixtures without pozzolanic addition (RC and RAC). The SCF and SRAC mixtures accounted for reductions of up to 78% and 68% in comparison to the reference mixture (RC) and the mixture with partial replacement of natural fine aggregates with recycled fine aggregates (RAC), respectively. The lower penetration of chlorides, determined by the reduction of migration coefficients, indicates a lower permeability in these mixtures in relation to the others. These results are consistent with those seen previously with the decrease in capillary water absorption.

Furthermore, the use of recycled fine aggregates reduced chloride ion migration coefficients at all ages in comparison to the use of the reference mixture. This fact suggests that the replacement of natural aggregates with recycled aggregates (30%), combined or not with the partial replacement of Portland cement with silica fume, was effective in improving this property. However, for SFC and SRAC mixtures, both including the partial replacement of Portland cement with silica fume, the partial use of recycled aggregates in the mixture did not improve chloride penetration resistance. Thus, although both incorporations have been shown to be potentially beneficial for the reduction of chloride penetrability, the use of silica fume has led to more significant reductions (up to 80%) in relation to the partial replacement of natural fine aggregates with recycled fine aggregates (up to 23%). These findings suggest that the pozzolanic addition acted chemically and physically in reducing concrete porosity, forming hydration products capable of refining the cementitious matrix.

As shown in Table 7, the concretes produced were classified according to chloride penetration resistance at the age of 28 days. In this context, the partial replacement of Portland cement with silica fume led to concretes with *very high* chloride ion penetration resistance when only natural fine aggregate was used (SFC), and concretes with *high* chloride ion penetration resistance when recycled fine aggregate was incorporated., SRAC. In the mixtures without pozzolanic material, the concretes produced showed *low* chloride ion penetration resistance.

Regarding the statistical analysis, Table 14 presents the analysis of variance (ANOVA) for the adjusted statistical model. All factors were significant at 5% and 1% probability levels for chloride ion migration coefficients.

Table 14. Analysis of variance applied to the average chloride migration coefficients.

Variable	Degress of freedom	Sum of squares	Sum of mean squares	F-value	p-value
Mixture	3	3317	1105.7	1674.68	<0.001
Age (days)	2	36	17.8	27.01	<0.001
Mixture/Age (days) interaction	6	20	3.4	5.16	<0.001
Residues	60	40	0.7	-	-

The test model built with the variables Mixture and Age (days) explains 98.84% of the total variability in the diffusion coefficient. The variable Mixture is the factor that explains most of the variability in the data, accounting for 97.19% of the total variability, followed by Age (days), accounting for 1.05% of the total variability. The interaction between Mixture and Age (days) explains 0.60% of the total variability.

The interactions between Mixture and Age (days) were significant at 5% and 1% probability levels. The assumptions of the models were met, and the residues follow a normal distribution. The graph shown in Figure 8 shows the behavior of the average diffusion coefficients by Mixture and Age (days). The statistical analysis of the results confirmed the trend of higher chloride ion penetration resistance with the use of concrete produced with partial replacement of Portland cement with silica fume (SFC and SRAC). The use of silica fume alone did not differ statistically from its use in association with the partial replacement of natural aggregates with recycled aggregates for concrete production. Furthermore, when fixing the factor Age (days), the chloride ion migration coefficients in the RC and RAC mixtures were statistically different from those of mixtures produced with cement plus silica fume. These mixtures were also statistically different from each other, with the reference concrete (RC) performing worst.

The average of the migration coefficients was shown to decrease with increasing ages for the SRAC and RC mixtures. For the RAC mixture, this variable decreased considerably between the ages of 28 and 63 days and increased very little between the ages of 63 and 91 days. On the other hand, the SFC mixture shows a slight increase of this variable between the ages of 28 and 63 days, followed by a slight decrease between the ages of 63 and 91 days. This corroborates the statistical significance of the interactions. The superior behavior of the mineral-added concretes in relation to the others can be explained, basically, by two reasons: the first is related to the filler effect and the second is related to the pozzolanic reactions. In both cases, there is a densification of the cement paste, with pore refinement and reduction of the interconnection of these pores, making the transport of chlorides difficult.

4 CONCLUSIONS

In this study, was investigated the use of industrial by-products and construction waste - in particular, fresh concrete waste from washing operations - as alternative materials in the production of concrete, proving to be a viable alternative in the design of future concrete mixtures more ecological, in this case with better mechanical properties and greater durability compared to concrete without the use of the proposed materials.

The increase in the specific surface area of the materials, obtained by partially replacing Portland cement with silica fume and natural fine aggregate with recycled fine aggregate, may have contributed to obtaining concretes with less porosity and permeability, resulting in a more refined cement matrix with improvement of the analyzed properties.

In addition, it can be concluded that partial replacement of Portland cement with silica fume significantly improved the properties tested in comparison to partial replacement of natural fine aggregate with recycled fine aggregate. This is because, as mentioned in the literature review, in addition to the pore filling (filler effect) provided by the recycled fine aggregate, the silica fume has pozzolanic activity, leading to greater reactivity and forming more resistant and morphologically denser hydration products. Even so, the incorporation of recycled fine aggregates proved to be potentially viable since it increased, although slightly, mechanical strength and chloride ion penetration resistance with the use of a more sustainable mixture.

The use of silica fume in the proposed content (8%) proved to be effective in achieving significant beneficial effects in all the properties analyzed. For example, it increased compressive strength by up to 28% and reduced chloride penetrability by up to 79%. It is noteworthy that, according to the classification proposed in Table 7, this addition led to the production of concretes with *very high* chloride ion penetration resistance.

Finally, it is suggested that future studies produce mixtures with incorporation of recycled fine aggregates at other replacement levels, as well as new tests aimed at characterizing the materials and specification of possible parameters more suitable for use. With this, the mechanical properties and durability of concrete can be improved.

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