



Evaluation of tire rubber surface pre-treatment and silica fume on physical-mechanical behavior and microstructural properties of concrete

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ABSTRACT

The increasing accumulation of tire waste has become a social environmental and public health problem because rubber degradation is extremely difficult to achieve and time consuming. The incorporation of rubber waste in concrete has become a recourse to assist in the disposal of this solid waste. This investigation evaluated the influence of a chemical pretreatment with sodium hydroxide solution (NaOH) on the physical, mechanical, microstructural properties of concretes with two rubber residue contents (15% and 30%) as a natural fine aggregate replacement, and the addition of silica fume (7.5% and 15%) to replace Portland cement. X-ray microtomography and scanning electron microscopy were used to investigate the influence of treatment rubber and silica fume in the microstructure of concretes. The use of rubber in the cement matrix, regardless of the treatment (or lack thereof), decreases the concrete density (lower 10.5%) and compressive strength at 28 d (54%), besides the increase the porosity (18%) than reference concretes. The rubber pre-treatment did not significantly influence the concrete behavior. In contrast, the use of silica fume showed significant compressive strength gains, up to 80% for concretes with 30% of rubber replacement at 28 days. These gains were confirmed by the microstructural analysis and the densification of the interfacial transition zone.

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1. Introduction

Nowadays, approximately 50% of all extracted natural resources are connected to civil construction. The use of some industrial wastes and selected construction residues in concrete would bring technical, economic, and environmental benefits upon replacement of conventional aggregates (Erdem et al., 2018).

Many investigations have been conducted about the use of wastes in cement based materials, like the construction and demolition waste (CDW) (Silva and Andrade, 2017), granite residue (Sharma et al., 2017), siderite residue (Esen and Doğan, 2017), limonite residue (Esen and Doğan, 2018) and water treatment sludge (WTS) (Andrade et al., 2019). At the same time, another

waste investigated today is the use of rubber from tire waste. Tire rubber residues are a by-product of the tire industry, whose global production overcame 2,900,000,000 of tires in 2017 (Raffoul et al., 2017). When the tires are no longer useful, they are dumped in clandestine landfills, which are subject to weathering and water accumulation producing an ideal environment for insect proliferation (Thomas and Chandra Gupta, 2016). When incinerated, tires emit toxic gases, which demands an extremely efficient and expensive treatment, making incineration impracticable (Gesoglu and Güneyisi, 2011).

According to Guo et al. (2017), tire wastes were used for the first time in concrete by Eldin and Senoduci in 1993. The authors concluded that the replacement of coarse aggregates presented worse compressive strength values (85% reduction) compared to the replacement of fine aggregates (65% reduction). Since then, there have been an enhancement of studies on fine aggregate substitution (Si et al., 2017). Thomas and Chandra Gupta (2016) used rubber residues in high performance concrete, where they opted to replace fine aggregates by up to 12.5%. According to the

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authors, high-strength rubber concrete can be used on concrete pavements, floors and roads. According to [Lv et al. \(2015\)](#) the use of rubber particles provides a significant reduction in compressive strength, flexural strength and splitting tensile strength. Based on the experimental results, the modulus of elasticity decreased from 24.1 GPa to 6.3 GPa with the replacement of rubber particles at a high content of 100%. The rubber content should be limited to a maximum of 20–30%, however, the mechanical properties can be improved with higher cement content, lower proportion w/b, adding supplementary cementitious materials such as metakaolin (compressive strength increases by up to 20%) and choosing the surface treatment method to improve the adherence between the rubber and the cement paste ([Strukar et al., 2019](#)). [Gupta et al. \(2014\)](#) observed that concrete modified with rubber fibers decreased compressive strength by increasing the percentage of rubber fibers content for all ratios w/c 0.55, 0.45 and 0.35. For the w/c = 0.35, the reduction in compressive strength at 28 d was 24% considering the content of rubber fiber with a 25%. According to [Tiwari et al. \(2016\)](#), mechanical strength is reduced because the rubber particle has a lower toughness than natural aggregate particles. [Feng et al. \(2019\)](#) stated that the use content of rubber should be less than 30% and the material presented better deformability and good deformation results under dynamic loads. [Shu and Huang \(2014\)](#) reported that the smooth surface makes it difficult to bond the cement paste, and this weak bonding was probably related to the hydrophobic nature of rubber waste. For [Huang et al. \(2013\)](#), there are no chemical links between rubber and cement matrix due to their hydrophobic and hydrophilic nature. According to [Bravo and de Brito \(2012\)](#) water absorption by immersion increased for all natural aggregate replacement rates (5, 10 and 15%) by rubber aggregate and especially as the particle size of the replaced aggregate increased. This trend occurs because concrete with added rubber needs a higher water content to maintain a similar workability in all mixtures and also due to the higher volume of voids between the rubber and the cement paste. Another important factor as the relative density of the tire rubber is significantly lower than conventional aggregates, it considerably reduces the density of concrete ([Roychand et al., 2020](#)). The reduction in compressive strength occurs from the increase of the rubber content inserted into the concrete. This can be mitigated by replacing an appropriate part of the cement with silica fume or by treating the rubber particles with NaOH aqueous solution or immersion in water.

The silica fume is an alternative to improve the bond between the rubber waste and cement matrix, filling the interfacial transition zone (ITZ) due to their pozzolanic and filler characteristics ([Onuaguluchi and Panesar, 2014](#)). The study of [Aslani et al. \(2018\)](#) replaced the fine aggregate by rubber aggregates with a content of 10% where they reached a compressive strength higher than 30 MPa, considering w/b = 0.45, and used 40% cement, 32.5% fly ash and 22.5% granulated blast furnace slag and 7.5% silica fume. [Esen \(2010\)](#) investigated the temperature effect in concretes with 20% fly ash, whose results showed that no significant decrease was observed in concretes with temperatures up to 400 °C. [Pelisser et al. \(2011\)](#) replaced fine aggregates (10% in weight) by tire rubber and used an additional 15% of silica fume in cement replacement. The concretes were made with conventional rubber and modified alkaline activated rubber (NaOH). A microscopic analysis showed that, with the silica fume, the adhesion rubber/cement interface is improved. Consequently, the porosity was minimized, the concrete strength improved, and the permeability was lowered. [Gupta et al. \(2014\)](#) evaluated a concrete with 10% of fly ash in cement replacement and 25% replacement of natural fine aggregate with untreated rubber residue. The microscopic analysis showed that there was still a separation between the rubber particles and the cement matrix. The interfacial transition zone (ITZ) between the

rubber particles and the cement matrix has micro scale spaces and the width of the ITZ is affected by the addition of rubber. The bond can be improved by physical or chemical modification of the surface of the rubber particles ([Xu et al., 2020](#)).

Another way used in order to improve the mechanical properties of concretes with rubber waste is the use of pre-treatment techniques aiming the increase the bonding between rubber and cement paste. Many procedures were investigated, as a simple wash with water ([Aslani et al., 2018](#)) and exposure to ultraviolet rays (UV) ([Shanmugaraj et al., 2006](#)), but the most common approach is the use of some alkali-activator, like sodium hydroxide (NaOH) solution ([Elchalakani, 2015](#)). According to [Medina et al. \(2018\)](#), increased roughness produces a larger contact surface between the paste and the rubber aggregates. An infrared analysis showed that zinc stearate can be eliminated from the NaOH-treated rubber surface, causing modify in rubber surface chemistry, which explains the better adhesion between the treated rubber and the matrix cement. Dense cement hydration products are believed to be generated around the rubber particle and contribute to a better bond between them ([Chou et al., 2007](#)). [Youssef et al. \(2016\)](#) evaluated the ideal treatment period for the particles, whose results presented that immersion in NaOH solution by 30 min was sufficient to advance the performance of rubber concrete, and longer periods did not result in significant variations. This same pre-treatment time was observed by [Kashani et al. \(2017\)](#), where in their investigation the treatment was performed around 30–40 min. The authors verified that the samples containing rubber treated with NaOH showed a significant improvement in compressive strength in relation to the untreated samples. The treated samples have a compressive strength about 40% higher compared to the samples containing untreated rubber. The main objective of the pre-treatment is to improve the interface between the rubber and the cement paste. The compressive strength of concrete samples with rubber treated with NaOH solution increases by 23.4% compared to the results of samples with untreated rubber ([Guo et al., 2017](#)). Concrete mixtures with rubber aggregate treated with NaOH have a slightly higher compressive strength than mixtures containing rubber aggregate. The reference sample showed a brittle rupture (failure) while the concrete with rubber showed gradual failure instead of sudden collapse, from the rupture analysis associated with the increased fragility of concrete according to the increased use of rubber particles as fine aggregate ([Si et al., 2018](#)).

However, some facts are controversial between different authors regarding the pre-treatment effect. [Tian et al. \(2011\)](#) verified that a 24h pre-treatment of rubber resulted in a lower compressive strength compared to the untreated rubber concrete. [Turatsinze et al. \(2007\)](#) did not observe significant changes with treatment use. For [Li et al. \(2004\)](#), the treatment for 30 min did not result in a statistical difference in the values found, i.e., the results of the untreated rubber concrete were the same. Besides, pre-treatments are also expensive and can only be justified if concrete performance is significantly improved ([Raffoul et al., 2017](#)). According to the authors, prewashing rubber with water and pre-coating with silica fume did not improve concrete performance for substitution levels of up to 100% natural aggregate with rubber. [Záleská et al. \(2019\)](#) performed the pretreatment and left the rubber in the (NaOH) and after the rubber particles were washed with water and naturally dried. Even with treatment, the strength of the studied concretes containing rubber-based aggregate was reduced. [Tiwari et al. \(2016\)](#) found that most of the research using rubber waste in concrete was directed at the evaluation of mechanical properties. Thus, a detailed study evaluating the impact of other factors (microstructure, porosity and elastic modulus) in the replacement of rubber waste in concrete is necessary.

Most researchers suggest that rubber treated with NaOH solution increases the mechanical properties of rubberized concrete. However, other researchers reported that the mechanical properties of rubberized concrete treated with NaOH solution do not change or decrease. The different sizes of rubber particles, the origin of the rubber, the concentration of the solution and the processing time may have contributed to the contradictory results (Li et al., 2019). Considering that the literature in this subject is quite contradictory, there is a need for further investigation. The main objective of the present study is to investigate the effectiveness of chemical pre-treatment of tire rubber and the use of silica fume on the physical-mechanical through the use of a statistical validation. From this, the properties of the concrete with the treated rubber were evaluated: bulk density, porosity, water absorption by immersion, compressive strength, splitting tensile strength, elastic modulus, X-Ray microtomography and scanning electron microscopy.

2. Experimental program

2.1. Materials characterization

Was used the early-age Portland cement CP V (similar to the ASTM Type III cement) manufactured in Brazil as the binder material, with a specific gravity of 2.94 g/cm³, density of 1.02 g/cm³, and compressive strength 28 d of 54.1 MPa. Silica fume was also used as binder. It presented a specific gravity of 3.01 g/cm³ and density of 0.85 g/cm³. Fig. 1 shows the X-ray diffraction (XRD) of the used silica fume and cement.

The composition of both cement and silica fume are shown in Table 1 obtained through a semi-quantitative X-ray fluorescence (XRF) analysis performed on the PANalytical Axios mAX equipment.

The utilized rubber residue was originated from the tire recapping process in a company located in Brazil. During this process, the magnetic separation of the steel wires and the separation of the rubber particles occur through an exhaustion process, with the disposal of metallic elements. The rubber particles presented a specific gravity of 1.13 g/cm³, fineness modulus of 2.4, and length variations between 0.8 mm and 3.11 mm, as shown in Fig. 2.

A chemical pre-treatment was applied to rubber particles. It was based on a method proposed by several authors, including Si et al. (2017), Guo et al. (2017), and Pelisser et al. (2011). In these studies, the best results were obtained by employing a 1 M NaOH solution

Table 1

Chemical composition of binders used.

	SiO ₂	Al ₂ O ₃	CaO	MgO	K ₂ O	SO ₃	Na ₂ O	Fe ₂ SO ₃
Cement	1.26	3.13	77.02	6.55	1.26	5.28	0.17	4.47
Silica fume	49.89	3.01	18.16	8.13	10.86	1.8	2.29	3.49



Fig. 2. Rubber waste.

for approximately 40 min. Afterwards, all material was washed in abundant water to remove any residue of the solution. The rubber particles were left on a clean, smooth surface for drying at 27 °C. After that, they were kept in a protected container until incorporated into the concrete.

Fig. 3 shows the surface appearance of untreated rubber particles (Fig. 3 (a)), treated particles (Fig. 3 (b)), and fine aggregates (Fig. 3 (c)) through scanning electron microscopy (SEM) images. The untreated rubber presented rougher texture and the rubber treated in the NaOH solution had a smoother surface texture.

Wang et al. (2017) reported a similar tendency compared to the results obtained in the present work. They verified the presence of deposited loose particles on the untreated rubber surface, and attributed the weak bonding between cement hydration products to these results. When performing a NaOH treatment, Tian et al. (2011) observed the erosion effect of this acid solution on particles, which presented improved adherence with cement particles. The authors report that most effective additive in this case is zinc stearate, which your removal makes the rubber surface rougher, resulting in an improved adherence to the paste.

The main elements in the chemical composition of the rubber waste are shown in Table 2. They were obtained by a semi-quantitative test using energy-dispersive spectroscopy (EDS) coupled with SEM. Rubble samples were analyzed without treatment (A) and with treatment (B).

There is a significant difference between the elements of the analyzed rubbers. Some elements such as Cu, Al, Na, and Mg were identified for only one sample (A or B). Moreover, the elements of sample A were different from those identified by Gupta et al. (2016). There is a significant percentage difference in the specific peaks, especially for carbon (C). It can be concluded that the strength of carbon-containing concrete is lower compared to the reference because carbon is a soft material. Regards to the number of elements identified by Gupta et al. (2016) for untreated rubber is more similar to the percentage observed for treated rubber (A). The differences between the identified element peaks and the percentage of each element can be attributed to the rubber particle production process.

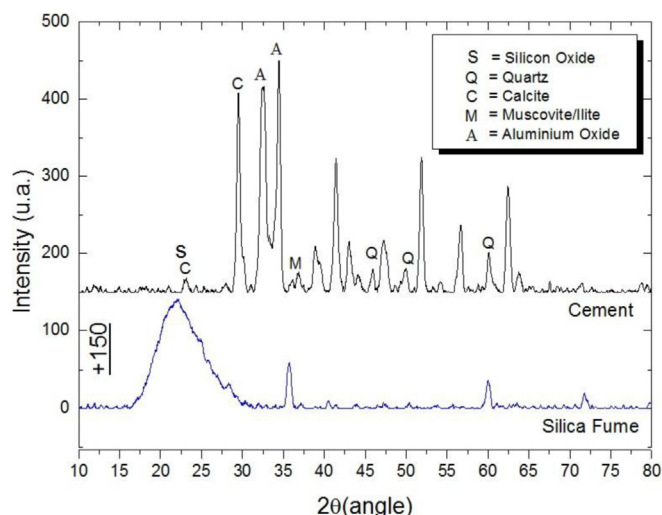


Fig. 1. XRD of cement and silica fume.

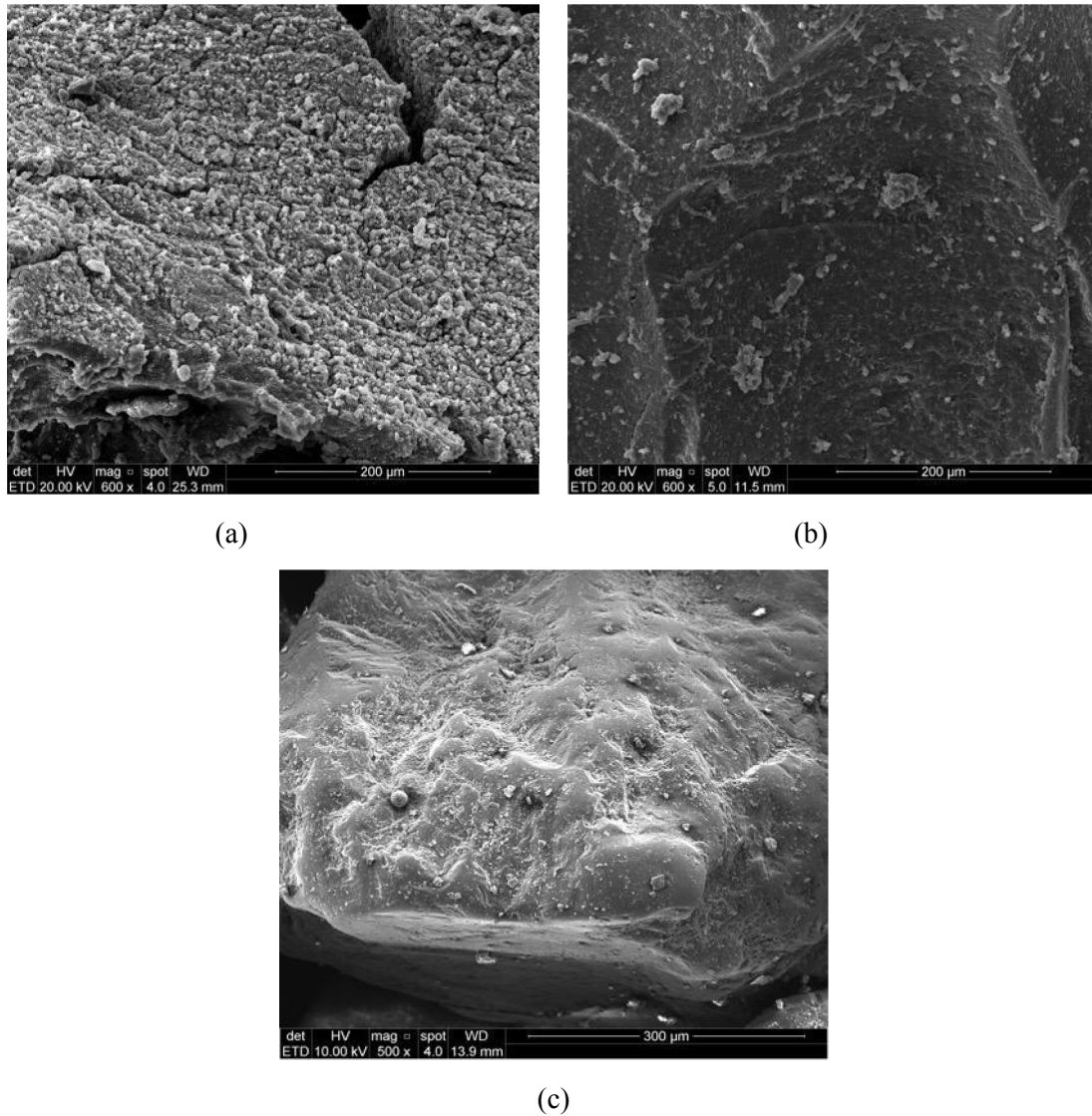


Fig. 3. SEM images of rubber (a) without treatment, (b) with treatment, and (c) fine aggregate (magnification = 500 ×).

Table 2

Chemical composition of the rubber without (A) and with (B) treatment (in %).

	C	Fe	Zn	O	Cu	Si	Al	S	Ca	Na	Mg
A	44.30	16.42	15.04	6.54	3.58	1.32	0.95	0.48	0.39	—	—
B	60.14	0.27	4.04	5.36	—	0.21	—	1.70	0.26	0.44	0.08
Gupta et al. (2016)	87.51	—	1.76	9.23	—	0.20	0.08	1.08	—	—	0.14

Table 3

Materials characterization.

Property	Natural sand	Brazilian Standard	Basaltic Stone	Rubber	Brazilian Standard
Specific gravity (g/cm^3)	2.63	NBR NM 52 ABNT (2009b)	2.64	1.13	NBR NM 53 ABNT (2009a)
Fineness module	1.69	NBR NM 248 ABNT (2003)	6.06	2.4	NBR NM 248 ABNT (2003)
Maximum size (mm)	4.8		19	3.11	

Sand from the Guaíba Lake (State of Rio Grande do Sul) was used as fine natural aggregate, and crushed stone of basaltic origin was used as coarse aggregate. Their characterization is presented in Table 3, and the particle size distribution of the materials is presented in Fig. 4.

2.2. Experimental design

Natural fine aggregates were replaced by rubber at 15% and 30% (volume). The rubber with and without the NaOH treatment was analyzed in order to investigate the influence of the treatment on

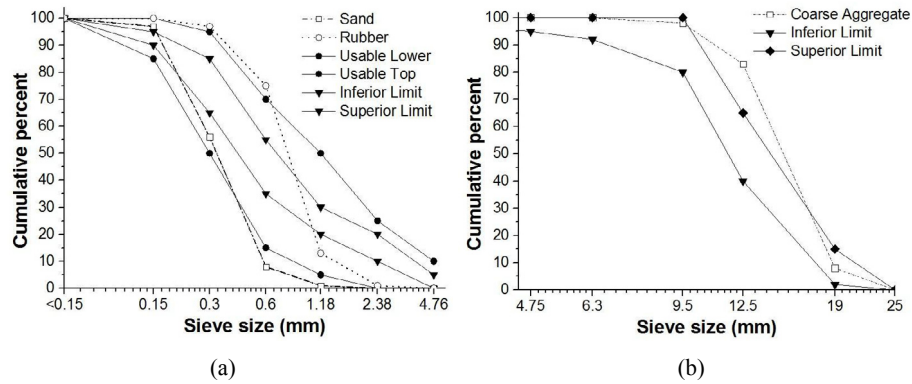


Fig. 4. Particle size distribution of (a) rubber and sand and (b) coarse aggregate.

the particles, whose similar studies were performed by other researchers. Kashani et al. (2017) used 20% of replacement, while Si et al. (2017) used 15–25% NaOH treated rubber. As previously studied by Pelisser et al. (2011) the authors used 15% silica fume and Gupta et al. (2016) opted for the use of 5 and 10%. In this study, the silica fume was used to replace the cement mass at 7.5% and 15% of the weight of the binder. A constant water/binder ratio of 0.5 was chosen for all concretes, based on similar studies carried by Shorbi and Karbalaie (2011) and Youssf et al. (2016).

Analysis of variance (ANOVA) was performed and from the F test it was determined if the analyzed parameters had a statistically significant effect on the reference concretes and with the use of rubber and silica fume in physical-mechanical properties at a confidence level of 95%.

2.3. Concrete production

The IPT/EPUSP (Helene and Terzian, 1992) method was used for mix proportioning all concrete mixtures. The workability of all

concretes was fixed at 90 ± 10 mm. To achieve the desired consistency, a polycarboxylate based superplasticizer additive was employed in some mixtures. The molding and curing of the specimens were performed according to the Brazilian standard NBR 5738 (ABNT, 2016).

Due to the difference between the specific mass of the fine aggregate and the residue, it was necessary to adjust the amount of material to be used for the production of concrete using Equation (1).

$$M_{RUBBER} = M_{NA} \times \frac{\gamma_{RUBBER}}{\gamma_{NA}} \quad (1)$$

Where M_{RUBBER} is the mass of the rubber aggregate (kg), M_{NA} is the mass of the natural aggregate (NA) (kg), γ_{RUBBER} is the specific mass of the rubber aggregate (kg/dm^3), and γ_{NA} is the specific mass of the natural aggregate (kg/dm^3). The mixture proportions of the concretes are presented in Table 4.

Table 4
Mixture proportions used in investigation.

Mixture	Rubber (%)	Silica fume (%)	Cement (kg/m^3)	Silica fume (kg/m^3)	Aggregates (kg/m^3)			Additive (%)	Slump (mm)
					Fine	Rubber	Coarse		
REF	0	0	379	0	825	0	1068	0	95
R15SF0	15	0	356	0	702	54	1068	0.085	85
R30SF0	30	0	337	0	578	107	1068	0	80
R0SF7.5	0	7.5	379	28.43	825	0	1068	0.081	100
R0SF15	0	15	379	56.86	825	0	1068	0.155	80
R15SF7.5	15	7.5	356	26.7	695	53	1068	0.035	80
R30SF7.5	30	7.5	337	25.28	578	106	1068	0.108	100
R15SF15	15	15	356	53.4	702	53	1068	0.208	95
R30SF15	30	15	337	50.56	578	106	1068	0.124	85
RN15SF0	15	0	356	0	705	53	1072	0	85
RN30SF0	30	0	337	0	583	107	1072	0	80

Table 5
Characterization test used in concretes.

Properties	Brazilian Standard	Specimens and dimensions	Age
Bulk density	NBR 9779 (ABNT, 2012)	3 Cylinders (100 × 200 mm)	28 days
Porosity			
Water absorption by immersion			
Compressive strength	NBR 5739 (ABNT, 2018)	Cylinders (100 × 200 mm)	7, 28, 63 days
Splitting tensile strength	NBR 7222 (ABNT, 2011)	Cylinders (100 × 200 mm)	28, 63 days
Elastic modulus	NBR 8522 (ABNT, 2017)	3 Cylinders (100 × 200 mm)	28, 63 days
X-Ray microtomography	Specific procedure, not standardized	Cubes ($a = 2.5$ mm)	28 days
Scanning electron microscopy	Specific procedure, not standardized	Pieces (3–6 mm)	28 days

2.4. Tests performed

The concrete was cast using cylindrical specimens (10×15 cm) for each mix design, according to the NBR 5738 (ABNT, 2016). After 24 h, the specimens were demolded and submerged in water until they reached their age for physical-mechanical tests (Table 5). To evaluate the microstructure of the concretes at 28 d, the densification effect in the interfacial transition zone (ITZ) of the samples were analyzed, using samples from middle of the test specimen.

SEM analyses were performed at 28 d, mainly to verify the ITZ of the concrete samples in the Inspect F50 – FEI, at 0.3–30 kV and resolution point of 1.2 mm. For that identification, a method based on previous study was employed. Pham et al. (2018) obtained images where featured the microstructure the concrete that contains ITZ, rubber and the cementitious matrix. The microstructural analysis was performed using dispersive energy spectroscopy (DES, Oxford Instruments), with the instrument connected to SEM.

The X-ray microtomography was performed according to the method proposed by Pelisser et al. (2011), which analyzes the microstructure of the concrete with rubber and silica fume at 28 d. All concrete samples were analyzed by the SkyScan 1173 – Bruker scanner. The equipment it has a resolution of $10 \mu\text{m}$, the samples were 2.5×2.5 mm, the volume reconstruction of the obtained data was performed through software, and the data was exported as a 32-bit file. The volume was calculated based on the average of each plan.

3. Results and discussion

3.1. Physical properties

The results of concrete absorption, porosity, and specific mass tests are presented in Fig. 5 for concretes without silica fume (a), with 7.5% of silica fume, (b) and with 10% of silica fume (c). The porosity of the sample with 15% rubber replacement increased by 18.32% compared to the reference. However, it remained constant for the 30% rubber replacement. Moreover, it is possible to relate the minimization of the concrete's density with the increase of rubber content. In this case, a decrease of approximately 10.46% in relation to the reference was obtained for the replacements of 15% and 30%.

For concretes molded with 7.5% silica fume (Fig. 5 (b)), the porosity was 27.84% lower compared the no-rubber concrete with and without silica fume (Fig. 5 (a)). Such porosity remains constant at 0–15% rubber contents. However, for 30% rubber, there was an increase of 28.39% on the concrete with the same silica fume percentage. On this case, the density also showed an inversely proportional behavior to absorption and porosity. By using 30% of rubber, these values decreased by 7.08% compared to ROSF7.5.

The initial porosity of the concretes with 15% of silica fume was 5.89% (Fig. 5 (c)), which is 37.60% lower compared to the reference value (Fig. 5 (a)). With a 30% rubber addition, the porosity increases 49.74% compared to the reference, and the density decreased almost constantly according to the increase in the replacement levels, unlike the results presented in Fig. 5(a) and (b). For 30% rubber addition, the density decreases approximately 10.04% compared to ROSF15.

It can be observed that the results are in agreement with those obtained by others investigations. Thomas and Chandra Gupta (2016) pointed out that porosity increases with the addition of rubber and, consequently, the concrete absorbs more water. Concretes with 20% of crumb rubber presented an increase of 70% in water penetration. This behavior also was verified by Pelisser et al. (2011), that found an increase in the compressive strength by

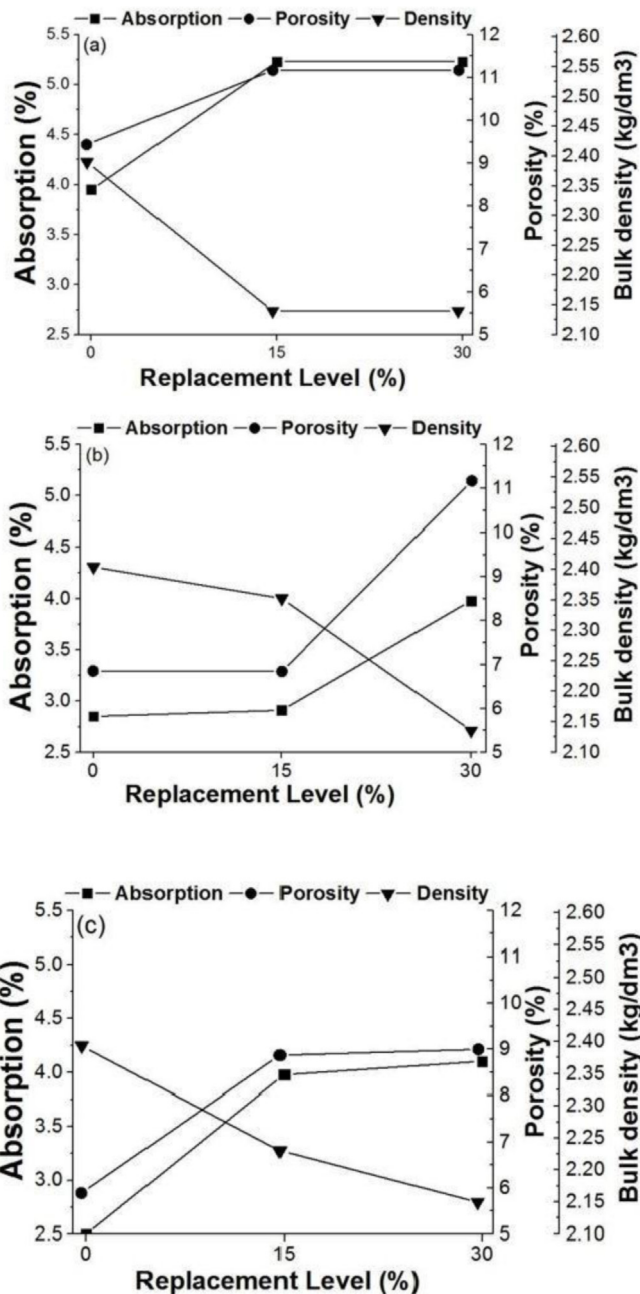


Fig. 5. Absorption, porosity, and density for concretes: (a) without silica fume, (b) with 7.5% silica fume, and (c) with 15% silica fume.

61.7% for a w/c ratio equal to 0.40, considering concretes with 15% of silica fume. This occurs mainly due to the effect of chemical treatment combined with silica fume fills the voids. The concrete is denser and less porous, which contributes to better adhesion and reduced weaknesses, recovering a significant part of their strength.

3.2. Mechanical properties

3.2.1. Compressive strength (f_c)

The effect of treatment in the compressive strength is shown in Fig. 6. It can be seen that a higher substitution content minimizes the compressive strength compared to the reference concrete, regardless of the treatment performed on the particles. It can also

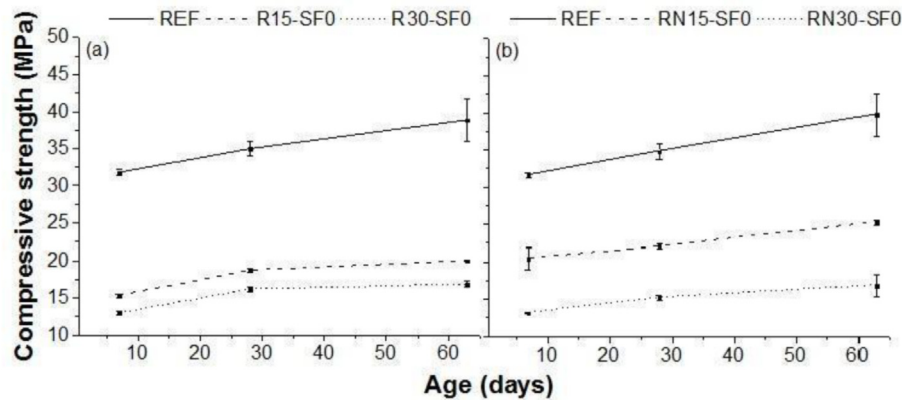


Fig. 6. Compressive strength for concretes with (a) treated and (b) untreated rubber.

be observed that the concrete treated with rubber presents lower resistance compared to the untreated rubber concrete.

RN15SF0 concrete showed compressive strength 51.6% lower than reference concrete at 28 d. In case of concrete with treated rubber, the compressive strength of concrete R15SF0 was 41.3% lower when compared to REF concrete. RN15SF0 presented compressive strength 17.47% higher when compared to R15SF0. Albano et al. (2005) observed a compressive strength with 10% untreated rubber approximately 61.7% lower than reference concrete. When compared to treated rubber concrete with NaOH, the reduction was 59.8%. Based on results obtained by the authors, treated rubber concrete the damage suffered was 4.5% greater when compared to untreated rubber concrete.

The obtained results are controversial in the literature. Some researchers mention that the treatment does not significantly influence the physical-mechanical properties of concretes Li et al. (2004) pointed out that NaOH treatment do not present effect for larger sized tire chips. Tian et al. (2011) showed that use of acidic or alkaline solutions do not present a significant performance in rubberized concretes for roads. Otherwise, some research showed that the treatment improves these properties, Balaha et al. (2007) showed that concretes treated with polyvinyl acetate (PVA), silica fume and NaOH solution presented an average decrease of 16% in compressive strength, but was less than concretes with untreated rubber, that presented a decrease of 27% considering a reference concrete with $w/c = 0.4$ and cement content equal to 400 kg/m^3 . According to Mohammadi et al. (2016), 20% and 30% treated rubber with NaOH was produced concrete in different treatment period (20 min, 24 h and 7 d) to different water/binder ratio (0.40 and 0.45). The authors observed that the best result were 24 h treated concrete for all w/b ratios. The concretes presented average compressive strength of approximately 25% higher when compared to untreated rubber concrete. In an experiment carried out by Turatsinze et al. (2007) was verified that the incorporation of

rubber aggregates (20% and 30% of total volume) results in a significant decrease in tensile and compressive strengths.

Deshpande et al. (2014) state that, under a load, the higher tenacity of the rubber in relation to natural aggregates produces a discontinuity in the matrix, decreasing its resistance. He et al. (2016) reports that the zinc-stearate diffuses to the rubber surface, leaving it with low free energy, which leads to a weak adhesion to the paste. According to Li et al. (2004), the particles size presents influence in these properties because the performance of the rubber chip composites (bigger than 76–13 mm) is lower than that of concretes with smaller rubber particles (4.75–0.425 mm). In accordance with this author, these results can be attributed to the larger surface contact between rubber and mortar.

The ANOVA results related to the compressive strength are presented in Table 6. It can be observed that main parameters investigated can influence the compressive strength of concretes. Nevertheless, the rubber replacement presents a higher influence (according to the magnitude of the F-test indicator) than the treatments.

Fig. 7 shows that the increase in the silica fume content results in higher compressive strength of concretes. As previously reported, the incorporation of rubber leads to a significant decrease in strength. However, with the use of silica fume this effect tends to be minimized. Gupta et al. (2016) found similar results, whose concretes with 25% of rubber fiber presented an increase of 33%, 27% and 25% for w/c ratios of 0.35, 0.45 and 0.55, with the replacement of 10% of cement by silica fume. Onuaguluchi (2015) reported that the increase of compressive strength in concrete with different levels of rubber (5%, 10% and 15%) with 10% of silica fume was equal to 104%, 97% and 75%, respectively.

Güneyisi et al. (2004) comments that the increased compressive strength due to silica fume tends to decrease with the addition of rubber particles in the concrete due to the progressive weakening of the matrix characteristics. The influence of the investigated

Table 6
ANOVA of the compressive strength for concretes without silica fume.

Source of variation	df	Sum of squares	Mean square	F-test	p value	Significance
Treatment type	1	14.20	14.20	20.70	0.0007	Yes
Rubber amount	2	2281.63	1140.82	1663.42	0.0000	Yes
Age	1	15.14	15.14	22.07	0.0005	Yes
Treatment type \times Rubber amount	2	25.57	12.78	18.64	0.0002	Yes
Treatment type \times Age	2	0.84	0.84	1.23	0.2891	No
Rubber amount \times Age	2	1.61	0.80	1.17	0.3433	No
Treatment type \times Rubber amount \times Age	2	0.37	0.18	0.27	0.7683	No
Error	12	8.23	0.69			

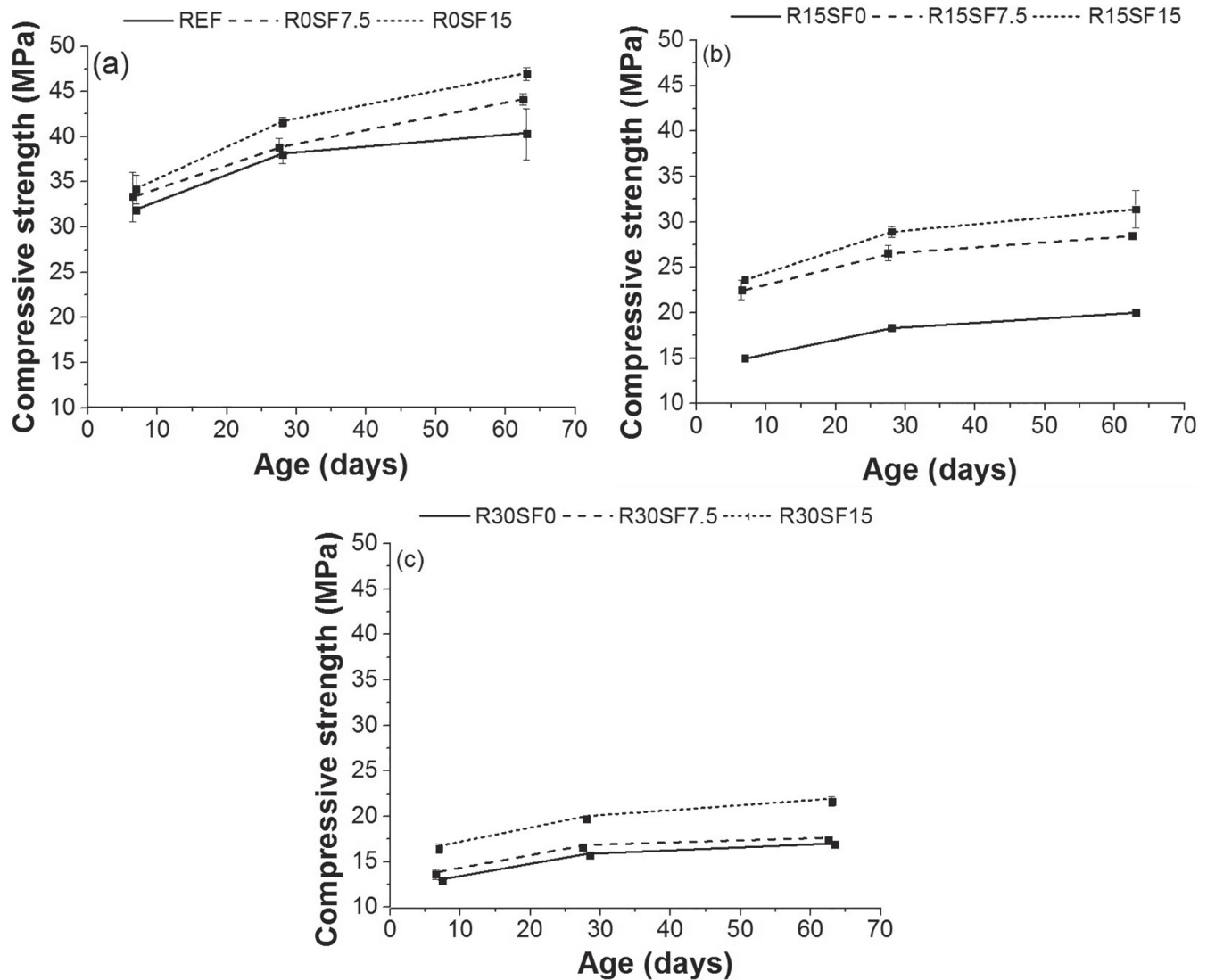


Fig. 7. Compressive strength for concretes with silica fume with: (a) no rubber, (b) 15% treated rubber, and (c) 30% treated rubber.

Table 7
ANOVA of compressive strength for concretes with silica fume.

Source	df	Sum of squares	Mean square	F-test	p value	Significance
Rubber amount	2	4096.41	2048.2	2136.82	0.0000	Yes
Silica fume	2	251.3	125.65	131.09	0.0000	Yes
Age	2	354.11	177.05	184.71	0.0000	Yes
Rubber amount \times Silica fume	4	117.87	29.47	30.74	0.0000	Yes
Rubber amount \times Age	4	43.64	10.91	11.38	0.0000	Yes
Silica fume \times Age	4	17.64	4.41	4.60	0.0058	Yes
Rubber amount \times Silica fume \times Age	8	22.59	2.82	2.95	0.0167	Yes
Error	27	25.88	0.96			

variables is presented in Table 7 through an ANOVA. The main parameters and all interactions are statistically significant for compressive strength. It can be seen that the amount of rubber had a higher effect than the silica fume content and the concrete age.

3.2.2. Splitting tensile strength ($f_{ct,sp}$)

The effect of pretreatment on the splitting tensile strength of the concretes is presented in Fig. 8. Compared with the reference concrete, there is a decrease in the tensile strength, regardless of the treatment used.

Such behavior is consistent with the one observed by Youssf et al. (2016). They compared the reference concrete with concrete combined with untreated rubber. The authors observed a 65.85% decrease in the tensile strength. However, after the treatment of the rubber particles, was observed that the resistance was partially recovered due to the relatively slow movement of the rubber particles through the erosion effect of the surface caused by the acidic solution. In contrast, the treatment in the present research resulted in an increased tensile strength reduction. For Onuaguluchi and Panesar (2014), the reduction of tensile strength is attributed to

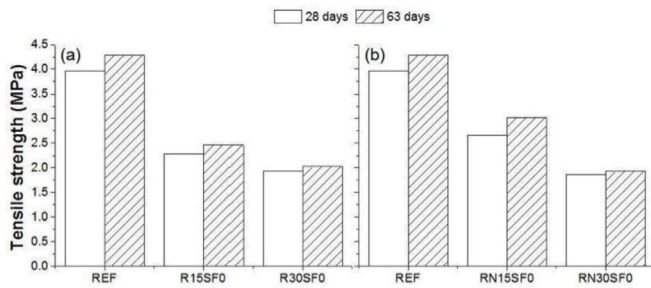


Fig. 8. Splitting tensile strength of concretes (a) with and (b) without treatment.

the same factors that affect the compressive strength. These factors include porosity, which increases with the increase of rubber particles; and the weak bond of rubber with the cement paste. As a result, the concrete starts to crack upon application of loads.

Based on ANOVA, Table 8 presents the statistical influence of the treatment, rubber amount, and age on the tensile strength of the concrete. It is of note that only the rubber replacement level presents a statistically significant influence, mainly due to the fiber effect associated with the rubber dimensions.

3.2.3. Elastic modulus

Fig. 9 present the results of the mean values observed for the elastic modulus of untreated and treated rubber concrete. The elastic modulus decreases upon incorporation of rubber. However, unlike the effect on compressive strength, the performance of treated rubber concrete was better than that of the ones with untreated rubber. For 15% of treated rubber, the elastic modulus increased by approximately 6.78% compared to the samples with untreated rubber. The rubber aggregate presented an overall low elastic modulus compared to natural aggregates for treated and untreated samples, similar to the results obtained by Gupta et al. (2014). The rubber aggregates when added to the concrete act as large pores in the matrix and, when subjected to external loads, do not significantly contribute to resistance due to the increased deformability of the material.

Table 9 presents the ANOVA for the parameters that influence the elastic modulus. The influence of the treatment is not statistically significant for the elastic modulus, as shown in Fig. 9. The biggest differences are observed for 15% of rubber content. However, the reference and the concretes with 30% of rubber presented similar values.

Fig. 10 shows the effects of silica fume on the elastic modulus, which tends to decrease as the rubber replacement content increases. When silica fume is added as a partial replacement of Portland cement, the negative rubber effect on elastic modulus decreases. The most expressive improvement (12%) in the elastic modulus was observed in concretes with 15% rubber and 7.5% silica fume (Fig. 10(b)), compared to concrete with 15% rubber and without silica fume (Fig. 10(a)). The other concretes (Fig. 10(b) and

(c)) showed approximately 2% improvements in the elastic modulus compared to concretes without silica fume (Fig. 10(a)). For all concretes, the effect of the elastic modulus at different ages showed a small increase. The most significant increase was of approximately 5.4% on concretes with 15% rubber and without silica fume (Fig. 10(a)). Compared to concrete with 30% rubber and 15% silica, the elastic modulus of the concrete with 30% untreated rubber and without silica fume was 9.92% higher at 63 days.

Table 10 presents the ANOVA that evaluates the statistical significance of silica fume and rubber content in the elastic modulus of concrete. The main parameters (rubber amount, silica fume replacement, and age) have significant influence on the elastic modulus. Moreover, considering the F-test, the rubber amount presented a higher influence than silica fume.

This same behavior was observed by Gupta et al. (2016), when they compared the elastic modulus of rubber concrete with and without silica fume. With increased rubber content, the modulus decreased by approximately 24.45%. However, in the presence of silica, the elastic modulus increased by 24.4%. The main reason attributed by the authors was the filling of pores with silica fume between the aggregate and the cement paste. The modulus decrease has been attributed to the replacement of rigid fine aggregates by rubber particles with low elastic modulus because the elastic modulus of the concrete depends on the elastic modulus of the aggregates (Onuaguluchi and Panesar, 2014). According to Pelisser et al. (2011), the rubber elastic modulus is considerably smaller than the elastic modulus of the natural aggregate making rubber concrete more deformable. Another reason could be possible cracks around the rubbers (observed through particle microscopy), which can affect resistance owing to the poor adhesion of the rubber with the paste (Mohammed et al., 2012).

In Fig. 11, it is possible to see a correlation between compressive strength and elastic modulus of the concrete for different rubber and silica fume contents. The compressive strength and elastic modulus tend to decrease as the rubber is incorporated. In contrast, these properties increase with the incorporation of silica fume, mainly due to the microstructural densification in transition zone owing to the pozzolanic effect (Onuaguluchi and Panesar, 2014).

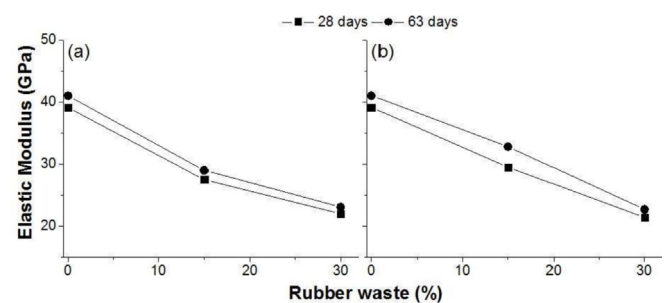


Fig. 9. Elastic modulus of concretes with (a) untreated and (b) treated rubber.

Table 8
Statistical significance of parameters on the tensile strength of concretes.

Source of variation	df	Sum of squares	Mean square	F-test	p value	Significance
Treatment type	1	0.0805	0.0805	0.859	0.3722	No
Rubber amount	2	19.80	9.90	105.67	0.0000	Yes
Age	1	0.2795	0.2795	2.983	0.1097	No
Treatment type × Rubber amount	2	0.3984	1.992	2.126	0.1620	No
Treatment type × Age	1	0.0000	0.0000	0.000	0.9844	No
Rubber amount × Age	2	0.0463	0.0231	0.247	0.7851	No
Treatment type × Rubber amount × Age	2	0.0209	0.0105	0.112	0.8952	No
Error	12	1.1242	0.0937			

Table 9
Statistical influence of variables in concrete elastic modulus.

Source of variation	df	Sum of squares	Mean square	F-test	p value	Significance
Treatment type	1	1.69	1.69	1.97	0.1728	No
Rubber amount	2	1851.56	925.78	1081.38	0.0000	Yes
Age	1	28.44	28.44	33.23	0.0000	Yes
Treatment type \times Rubber amount	2	10.55	5.28	6.16	0.0069	Yes
Treatment type \times Age	1	3.36	3.36	3.93	0.0591	Yes
Rubber amount \times Age	2	17.51	8.76	10.23	0.0006	Yes
Treatment type \times Rubber amount \times Age	2	8.19	4.10	4.79	0.0178	Yes
Error	24	20.55	0.86			

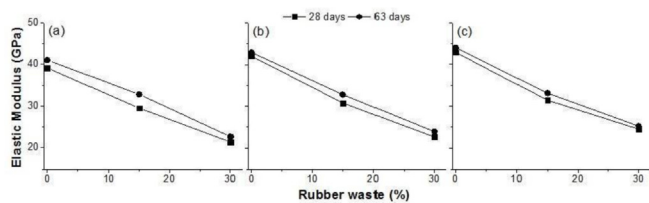


Fig. 10. Elastic modulus for concretes with rubber treated with: (a) no silica fume, (b) 7.5% silica fume, and (c) 15% silica fume.

3.3. Microstructural analysis

3.3.1. X-ray microtomography

The X-ray microtomography was used to verify the rubber distribution and the void content of the concretes. The dimension of all samples were $25 \times 25 \times 40$ mm, and 1200 slices were analyzed. Pores, rubber particles, and cement paste were observed. Visually, pores have a very characteristic appearance, with a rounded and darker color. In contrast, rubber particles have a more irregular shape and a grey color. The cement paste presents a color classified like light grey. On Fig. 12, a sample of concrete without silica fume and with different rubber contents

(15% and 30%) can be observed.

There was an increase in the amount of pores with the incorporation of rubber on the concrete matrix. Similar results were obtained by Kashani et al. (2017) and Onuaguluchi and Panesar (2014), who employed up to 30% rubber and observed a high variability on pore distribution based on tomography analysis.

The microtomography determined the voids volume on the selected cubic region of the concrete. The mechanical resistance and immersion water absorption of the reference specimen, and the 15 and 30% rubber concrete are shown in Fig. 13. It was verified, through a comparative analysis, that the concretes with higher compressive strength values present the lowest water absorption and voids volume. Similar results were obtained by Pacheco et al. (2018), whose authors used X-ray microtomography and pore size distribution to investigate the influence of water/cement (w/c) ratio, cement consumption, and environmental aggressiveness in the durability of a concrete mix (proportions according to the European concrete standard EN 206).

Fig. 14 shows the images of concretes without rubber and with different silica fume levels. The volume of voids decreased with the increased level of silica fume. This result can be explained by the silica pozzolanic effect. The silica fume reacts with calcium hydroxide, produced through calcium silicate

Table 10
Statistical influence of silica fume in elastic modulus.

Source of variation	df	Sum of squares	Mean square	F-test	p value	Significance
Rubber amount	2	2826.26	1413.13	1540.24	0.0000	Yes
Silica fume	2	74.46	37.23	40.58	0.0000	Yes
Age	1	16.27	16.27	17.73	0.0002	Yes
Rubber amount \times Silica fume	4	14.2	3.55	3.87	0.1051	No
Rubber amount \times Age	2	2.66	1.33	1.45	0.2489	No
Silica fume \times Age	2	0.03	0.015	0.02	0.9839	No
Rubber amount \times Silica fume \times Age	4	2.96	0.74	0.81	0.5291	No
Error	35	32.11	0.92			

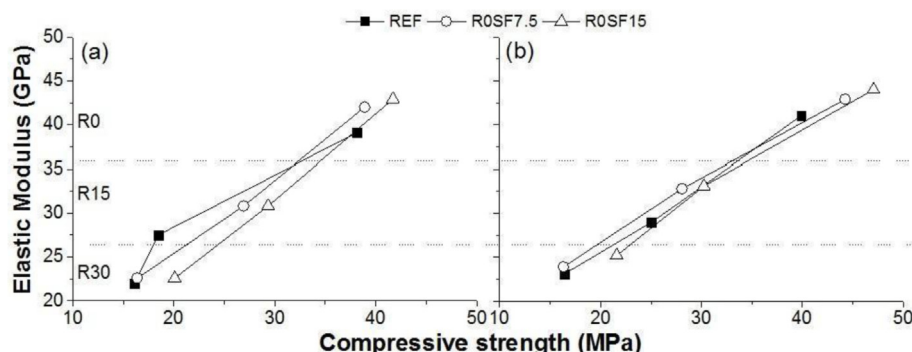


Fig. 11. Correlation between compressive strength and elastic modulus for (a) 28 d and (b) 63 d.

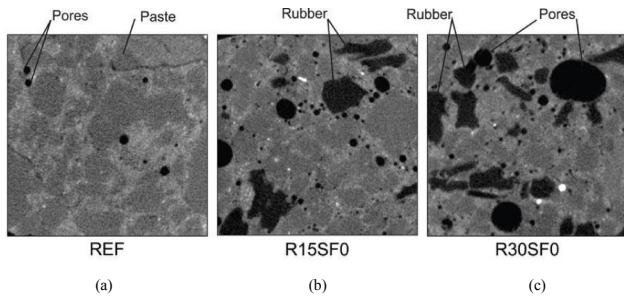


Fig. 12. X-ray microtomography of concretes samples: (a) reference, (b) R15SF0, (c) R30SF0.

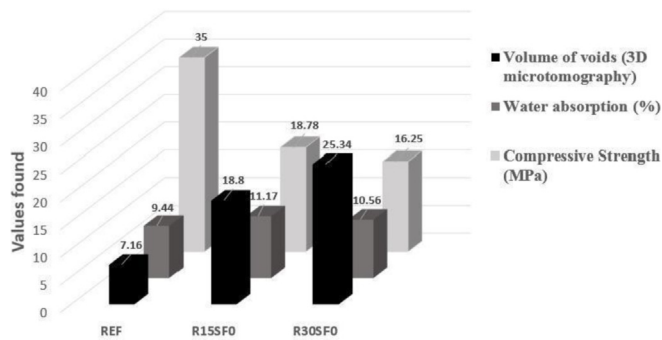


Fig. 13. Comparative analysis of parameters investigated for concretes with rubber.

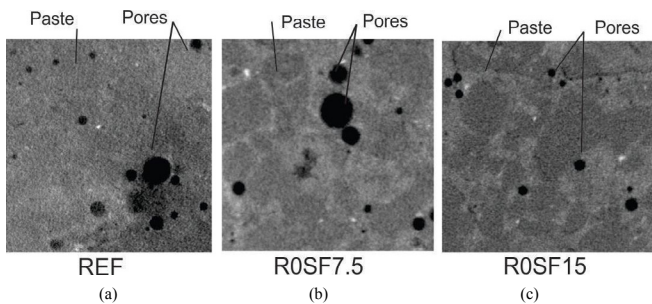


Fig. 14. X-ray microtomography of concretes: (a) reference, (b) R0SF7.5, (c) R0SF15.

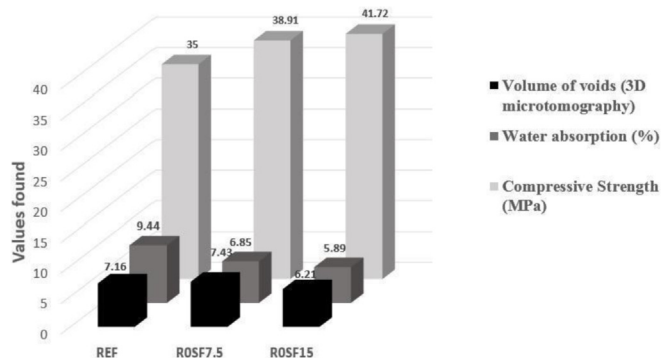


Fig. 15. Comparative analysis of parameters investigated for concretes with silica fume.

hydration, to form the CSH. As a result, the voids are filled, increasing the mechanical properties of the concrete (Andrade and Buják, 2013; Schumacher and Juniper, 2013).

Fig. 15 presents the volume of voids obtained through

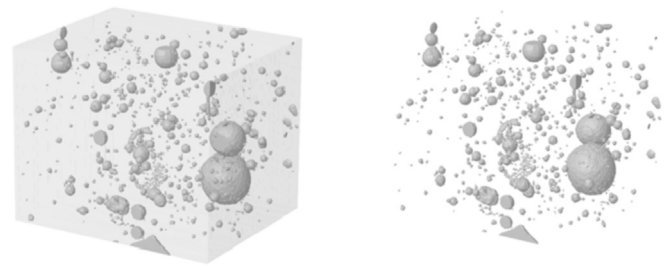


Fig. 16. Reconstructed image of reference concrete.

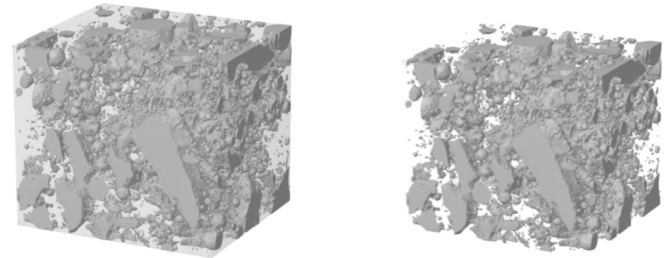


Fig. 17. Reconstructed image of concrete with 30% of rubber and without silica fume (R30SF0).

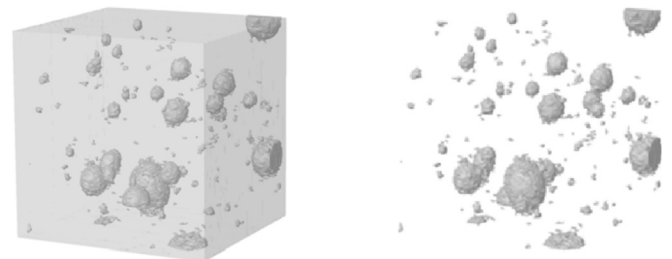


Fig. 18. Reconstructed image of concrete without rubber and with 15% of silica fume (R0SF15).

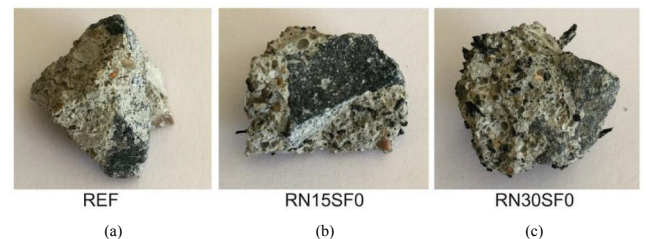


Fig. 19. Images of concretes: (a) reference, (b) RN15SF0, (c) RN30SF0.

microtomography, the absorption of water obtained by immersion, and the compressive strength at 28 d for concretes with silica fume. Absorption and volume of voids tend to decrease with increasing compressive strength. These results are similar to those obtained by Nežerka et al. (2019) and Siddique (2011).

With the help of a reconstruction software, 3D images of the concrete samples were obtained. The pores and rubber are presented as circular and irregular shapes, the cement matrix is presented in light-grey color. The reconstructed images of the reference concrete, the concrete with 30% rubber, and with 15% silica fume are presented on Figs. 16–18, respectively.

Comparing Figs. 16 and 17, it is possible to observe the high quantity of rubber particles and voids on the matrix. In addition, the incorporation of silica fume into the concrete matrix led to a porosity decrease from 13.7–15% to 9.1–9.8% (Figs. 16 and 18). This decrease can be associated with the pozzolanic reaction which results in CSH. In turn, CSH fills the transition zone, which becomes more stable and stronger Onuaguluchi and Panesar (2014).

3.3.2. Scanning electron microscopy (SEM)

Due to the size of the rubber particles, it was possible to observe in a macroscopic scale the distribution of materials in the concrete matrix. Fig. 19 (a), (b), and (c) show the reference concrete, and the concrete with 15% and 30% replacement of sand by rubber, respectively.

By microscopically analyzing the concretes with 30% treated and untreated rubber (Fig. 20(a) and (b)), it is possible to observe that no significant changes were identified in the transition zone after the NaOH treatment. Microcracks can be verified in both cases. This analysis confirms that, when subjected to compressive strength, untreated rubber concrete obtained better results than treated rubber concrete. Similar results were obtained by Mohammed et al. (2012). They observed that concrete with treated rubber, despite its rougher particle surface, did not show significant improvements, considering the adhesion of rubber with concrete in the all treatment methods used (variation of 20 m to 7 d).

A densification of ITZ was observed upon incorporation of silica fume in the concretes, as observed in Fig. 21(a) and (b). Rubber particles were better adhered to the cement paste, similar to the behavior observed by Pelisser et al. (2011) and Gupta et al. (2014). The ITZ densification leads an increase of the mechanical properties of concretes with silica fume, as shown in Fig. 21.

4. Conclusions

This research investigated the influence of chemical

pretreatment of tire rubber and silica fume on the physical-mechanical properties of their concrete matrix. Based on the obtained results, the following considerations can be made:

- The replacement of natural fine aggregates by tire rubber residues led to a decrease in physical-mechanical properties of concretes with lower density (between 10.5%), higher porosity (18%) and water absorption (between 2 and 4%) than reference concretes;
- The use of silica fume in concretes contributes to fill the voids caused by the increase of rubber particles, mainly in ITZ, as presented in SEM images. Was observed a decrease of 13% in volume of voids measured through 3D microtomography. The structure of the concrete presents less pores and it is more densified. Consequently, the mechanical properties investigated are improved.
- The use of rubber particles reduces the mechanical properties of concrete, regardless of the treatment application. According to the ANOVA results, the proposed NaOH chemical pre-treatment for rubbers did not result in significant gains for the evaluated physical-mechanical properties. The divergence related to effective rubber performance found in the results reported in literature must be related to the several manufacturing process of tires in the world, due to the different raw material and additives used. Regards to a better understanding of the chemistry and surface morphology of the treated particles and their interaction with the cement paste is still needed.

According to this investigation it was possible to evaluate the use of rubber from tires for use in concrete. Although there are many factors that still need to be investigated, was verified that the reuse of rubber waste in concrete without any pre-treatment do not have significance in properties investigated – avoiding pre-processing costs – and can contribute to provide an appropriate destination of this material.

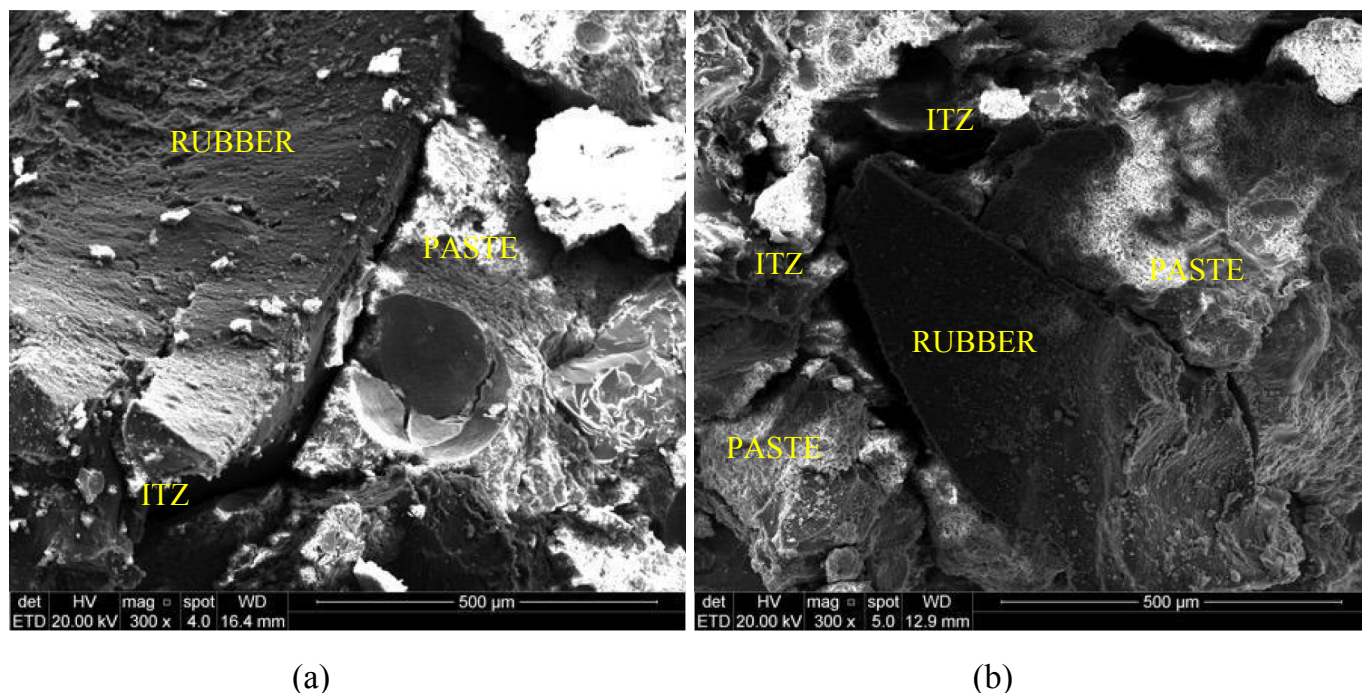


Fig. 20. SEM images of concretes: (a) RN30SF0, (b) R30SF0.

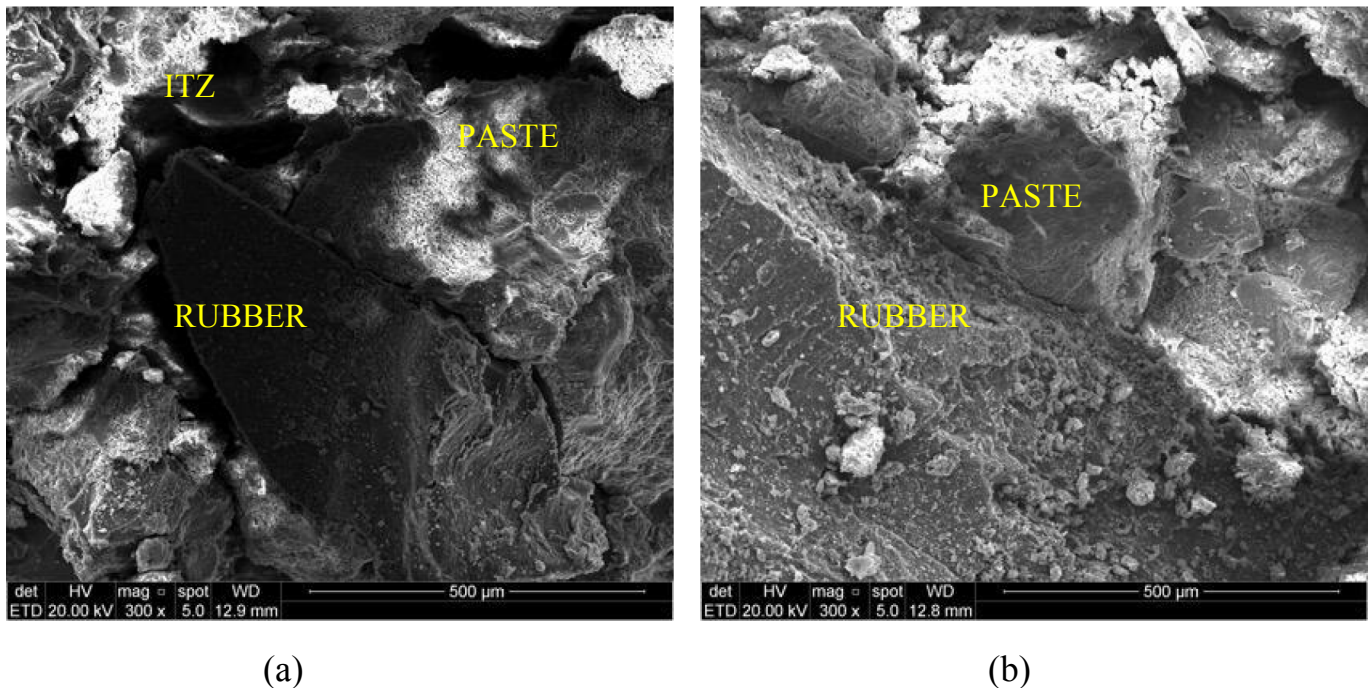


Fig. 21. SEM images of concretes: (a) R30SF0, (b) R30SF15.

Author contributions section

Cauana Melo Copetti: Conceptualization, Methodology, Experimental Work, Results and Discussion, Writing-Original draft preparation; Pietra Moraes Borges: Experimental Work, Results and Discussion, Writing-Original draft preparation. Jéssica Zamboni Squiavon: Results and Discussion, Writing-Original draft preparation. Sérgio Roberto da Silva: Results and Discussion, Writing-Original draft preparation; Jairo José de Oliveira Andrade: Conceptualization, Methodology, Supervision, Writing- Reviewing and Editing.

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