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ORIGINAL ARTICLE

Development and characterization of cementitious mixtures with replacement of fine and coarse conventional aggregates by marble residue

Desenvolvimento e caracterização de misturas cimentícias com substituição dos agregados finos e graúdos convencionais por resíduo de mármore

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Abstract: Finding ways to reuse residue is an alternative not only to reduce its presence in urban and natural environments, but also to reduce costs and consumption of resources by industries. The objective of this paper is to evaluate the properties of concretes made by replacing conventional coarse and fine aggregates with marble residue. Morphological, granulometry, specific gravimetry and crystalline phase analyses were carried out in the aggregates. The composition of the constituents, by mass, was 1:1,2:3 of Portland cement CPIII-32 RS, fine aggregates, and coarse aggregate, respectively, with the addition of 0.4 of water, molded and cured in water. About 25 concrete samples were made, varying the amount of coarse/fine aggregate in the cement mass according to the aggregates proportions of: A= 100% gravel/ 100% sand, B= 100% coarse RRO/ 50% fine RRO + 50% sand, C= 50% coarse RRO + 50% gravel/ 100% fine RRO, and D = 100% coarse RRO/100% fine RRO. The cylindrical samples measuring Ø50 x 100 mm were subjected to water absorption and compressive strength tests, and the results were compared with standards and literature. The water absorption was smaller than 2.5% and the compressive strength values were between 27-33 MPa, close to the resistance of cement used. Composition B presented the best results among the others, indicating the possibility of using the residue from marble for production of concretes for various purposes such as low load paving, sidewalks, blocks, among others. However, for interlocking floors, higher performance cement should be used to achieve the required strength. This type of concrete adds value to the residue and mitigates environmental impacts.

Keywords: concrete, residue, marble.

Resumo: Encontrar formas de reaproveitar resíduos é uma alternativa não só para reduzir sua presença em ambientes urbanos e naturais, mas também para reduzir custos e consumo de matérias primas pelas indústrias. O objetivo deste trabalho é avaliar as propriedades de concretos fabricados através da substituição de agregados graúdos e miúdos convencionais por resíduo de mármore. Foram realizadas análises de morfologia, granulometria, massa específica e da fase cristalina dos agregados. A composição dos constituintes, em massa, foi 1:1,2:3 de cimento Portland CPIII-32 RS, agregados miúdos e agregados graúdos, respectivamente, com adição de 0,4 de água, moldados e curados em água. Foram confeccionadas cerca de 25 amostras de concreto, variando a quantidade de agregado graúdo/fino na massa de cimento de acordo com as proporções de agregados de: A= 100% brita/ 100% areia, B= 100% RRO grosso/ 50% RRO fino + 50% areia, C= 50% RRO grosso + 50% cascalho/ 100% RRO fino, e D = 100% RRO grosso/100% RRO fino. As amostras cilíndricas medindo Ø50 x 100 mm foram submetidas a testes de absorção de água e resistência à compressão, e os resultados obtidos foram comparados com algumas normas e com a literatura. A absorção de água foi inferior

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a 2,5% e a resistência à compressão ficou entre 27-33 MPa, valor próximo à resistência do cimento utilizado. A composição B apresenta os melhores resultados entre as demais, indicando a possibilidade de utilização do resíduo do mármore para produção de concretos para diversas finalidades como pavimentação de baixa carga, calçadas, blocos, entre outros. No entanto, para pisos intertravados, deve-se utilizar cimento de maior desempenho para atingir a resistência necessária. Esse tipo de concreto agrega valor ao resíduo e mitiga os impactos ambientais.

Palavras-chave: concreto, resíduo, mármore.

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1 INTRODUCTION

In 2011, Brazil was ranked as the world's sixth largest producer of ornamental stones, the seventh largest exporter in total physical volume and the third largest exporter of granite blocks and slate products. The state of Espírito Santo is the main national producer and exporter of dimension stones, mainly granite rocks, whose production in 2011 was around 3.6 million tons. The production chain of the ornamental stone industry has great economic and social importance in the country. According to ABIROCHAS [1], the production of dimension stones in Brazil, in 2015, was around 9.5 Mt, of which 2.32 Mt were exported.

In this sense, the large amount of waste generated in the processing of ornamental stones has been worrying and bringing to the processing industry the need to research ways to use it. The blocks of rocks coming from the mining are divided into sheets, in equipment called looms, constituting the primary processing. During primary processing, almost 30% of the block, by volume, is transformed into fine waste. It is estimated an annual production of this waste in the country of more than 1.5 Mt [1].

This generates a high amount of waste during mining, and, during beneficiation, industries reach waste levels of 20-25% of the total processed. According to Ribeiro [2], in 2007 the ornamental stone sector reached 8 million tons of slabs produced and approximately 1.8 million tons of waste. These processing processes are responsible for the formation of the residue in the form of mud, which is formed due to the water used to cool the cutting equipment and to accelerate the process. Most of this waste is destined for decantation ponds, although some companies use filter presses to remove excess water, which is reused in the process, and pay companies to dispose of the solid waste in landfills. When there is no adequate destination, the waste is dumped in rivers, lakes or directly on the ground.

The concern with the environment, the intense inspection, the greater rigidity of the environmental control laws and the high costs of properly disposing of waste have stimulated the search for alternative forms of disposal, and the search for some way to reuse these residues [3].

Therefore, the use of the generated residue is very important, and it is in line with the National Policy on Solid Waste [4]. In this sense, several studies demonstrate the potential of ornamental rock waste, mainly from marble and granite, as an incorporation in the production of bricks, blocks, glass, floors and mortar [5]–[8]. This potential for reuse exists because the waste consists of quartz, alumina, potassium oxide, sodium oxide and lime, which are generally present as traditional resources used in the production of some ceramic products [9]. As a result, there is a need to better allocate waste from the processing of ornamental stones, opening up new fields of research and new techniques, giving commercial functionality to losses from production processes.

The objective of this work is to develop and analyze the properties of concretes from ornamental stone residue, in order to take advantage of the industrial residue to form a sustainable product, contributing to the reduction of the environmental impact generated in the mineral sector and adding value to the eco-product.

2 MATERIALS AND METHODS

The materials used in this paper were residue from dimension stones (collected at Seropédica marble factory, in the municipality of Seropédica-RJ-Brazil), medium sand, gravel 1 and cement CPIII-32 RS. The Seropédica marble factory has approximately 20-30% loss in ornamental stone plates due to the personalized cut of sinks, thresholds, tombs, among other applications in civil construction and decoration. These residues are pieces of different sizes and types of rocks, and they are usually discarded in boxes to be resold with very low added value.

The white marble residues were selected, and they were crushed in a jaw crusher that fractionates the particles into 3 parts of coarse aggregate, being equivalent to the classification size of gravel 0, 1 and 2. The white marble crushing process generates a large amount of dust (fine aggregate), which was collected and sieved through a #4.75 mm mesh sieve.

According to ABNT NBR 7211 [10], coarse aggregates are those whose grains pass through the #75 mm mesh sieve and are retained in the #4.75 mm mesh sieve. The different types of gravel are classified according to their granulometry, and particles that have dimensions known in civil engineering as Gravel 0: powder until 1.8 mm; Gravel 1: from 1.8 mm to 12.5 mm; Gravel 2: from 12.5 mm to 25 mm; Gravel 3: from 25 mm to 50 mm; Gravel 4: from 50 mm to 76 mm and Gravel 5: from 76 mm to 100 mm [11].

Each kind of aggregate is indicated for a specific application. The fact that gravel 0 is very fine is often used for fabricating slabs and tubes for example, while gravel 1 is used for columns and beams. Gravel 2 is coarser, and currently has not been used for common or self-compacting concrete, and it is used for thick floors or those that require greater resistance [12]. Thus, considering the 3 fractions of marble waste crushed in a jaw crusher, the size equivalent to 1 gravel was chosen to compose the concrete mass of this work.

The concrete mix was calculated considering the mixture of Portland cement CPIII, fine aggregate (medium granulometry sand), coarse aggregate with a size equivalent to gravel 1, and water. Figure 1 shows the image of the materials used to produce concrete.

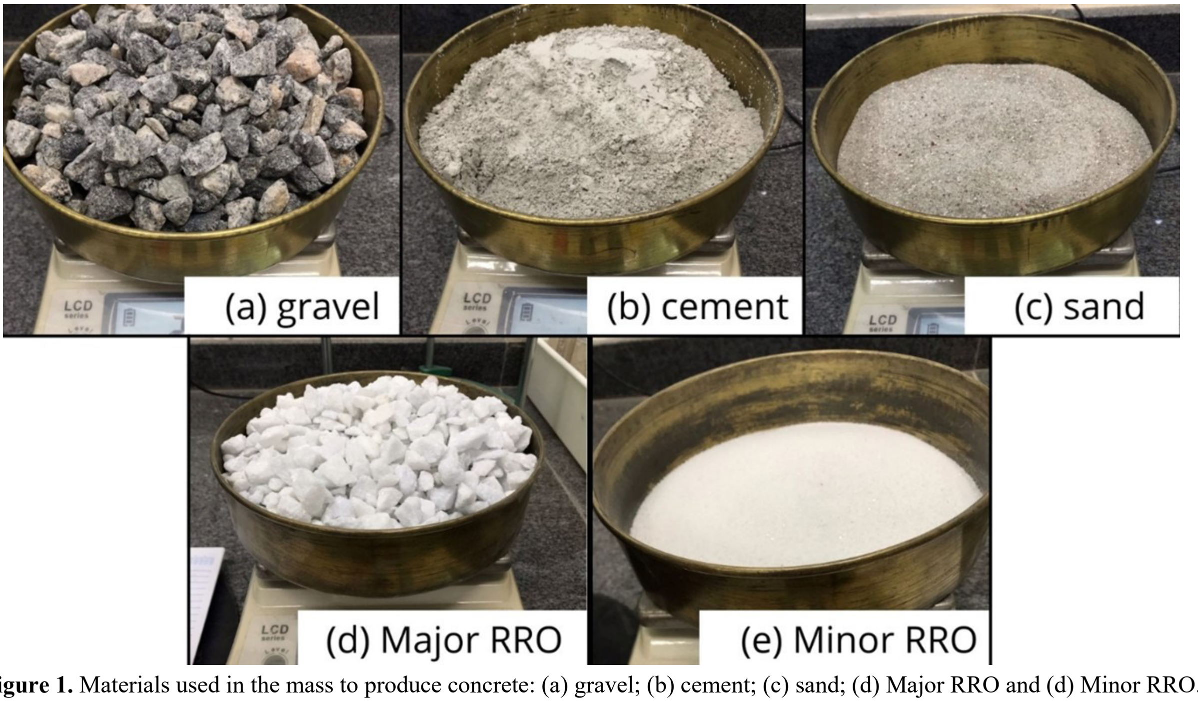

Figure 1. Materials used in the mass to produce concrete: (a) gravel; (b) cement; (c) sand; (d) Major RRO and (d) Minor RRO.

The proportion of the constituents, in masses, used for the elaboration of the samples were 1:1, 2:3 of Portland cement, fine and coarse aggregates, respectively. Most papers replace 5-70% of waste in the concrete composition, being that some replacement is carried out in the amount of cement, others in sand and others in both [13]–[19]. This work aimed to keep the amount of binder (cement) fixed and the traditional coarse and fine aggregates (sand and gravel) were partially and totally replaced by RRO, in order to identify their influence on the properties of the concrete.

The amount of water needed for the mass to acquire plasticity was determined by the water/cement ratio obtained by Abrams curves, and the value used was w/c= 0.4 [20]. The samples (CP) were developed for the analysis of water absorption and compressive strength following the nomenclature shown in Table 1.

The cement content (C) was calculated using the Equation 1 [21] considering the specific mass of 2.2 g/cm^3 and the trace shown in Table 1. Considering the proportion used in this work, the calculated amount of cement was 392 kg/m^3.

$$C = \frac{\gamma}{1+a+p+{}^{w}/_{c}} \quad (1)$$

Where γ = specific mass of concrete, *a* = content of fine aggregate and cement; *p* = coarse aggregate and cement content; and w/c = water cement ratio.

The compositional analysis of the crystalline phases and the granulometry of the aggregates were performed. The composition was characterized in an X-ray diffractometer (XRD), where the crystalline phases were analyzed in an X-ray diffractometer, XRD, with CuKα radiation (λ = 1.5418 Å), in the angular interval 2θ = 10–80º, step angular 0.02º and counting time 2 seconds. The phases were identified according to Bragg's Law and indexed using the JCPDS sheets.

Table 1. Proportions (in mass) of the constituents.

Sample	Cement	Fine aggregates		Corse aggregates		w/c
		Sand	Fine RRO	Gravel	Corse RRO	
A	1	1.2	-	3	-	0.4
B	1	0.6	0.6	-	3	0.4
C	1	-	1.2	1.5	1.5	0.4
D	1	-	1.2	-	3	0.4

To determine the particle size distribution of the sand and ornamental rock residue, the laser diffraction technique was used on a Malvern Mastersizer 2000 diffractometer. The specific gravity of the marble waste powder was determined according to ABNT NBR 6458 [22].

The conformation of the samples was carried out in cylindrical PVC molds measuring 100 mm in height by 50 mm in diameter [23], and hexagonal as shown in Figure 2, with DESMOL CD release agent from Vedacit being applied with the aid of a brush on the interior walls of the mold. The prepared masses were inserted into the molds with the aid of a metal rod and subjected to vibration for 30 seconds to promote consolidation and prevent the appearance of bubbles inside the sample, ensuring complete filling.

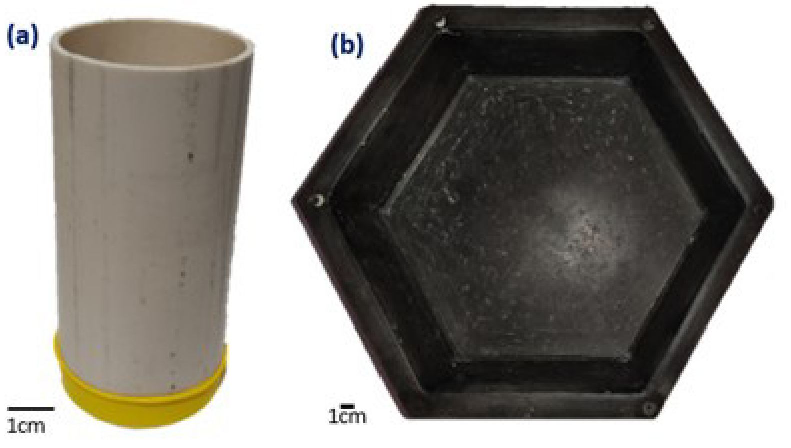

Figure 2. (a) Molds for cylindrical specimen and (b) hexagonal interlocking floor.

The water absorption test was performed according to the standard ABNT 9781 [24], for the twelve cylindrical specimens, three for each composition. After 24 hours of molding, the samples were demolded and submerged for 7 days in a tank with water to carry out the cement hydration process. After curing, these were removed and dried with a damp cloth. To obtain the saturated mass and dry mass, the samples were weighed at intervals of 2 hours, until obtaining a constant mass with a variation of up to 0.05% between measurements, for each sample, according to the standard. To obtain the dry mass, the samples were dried in an oven at 100 °C ± 5 °C and weighed, according to the standard. Equation 2 represents the water absorption of each cylindrical specimen, in percentage according to the standard, and subsequently obtained the arithmetic mean of these values for each composition.

$$a = \frac{(m_1 - m_2)}{m_1} .100 \tag{2}$$

The compressive strength test was performed according to the methodology specified in the ABNT NBR 7211 standard [10], considering six cylindrical specimens for each composition, which were kept in the tank with water for 28 days to ensure post-curing mechanical strength. Some samples did not obtain a homogeneous appearance after consolidation, being discarded before the mechanical test. Of the samples used, five were for composition A, six for composition B, two for composition C and three for composition D. The value of applied force was defined by the standard equal to loads of 0.3 MPa/s at 0.8 MPa/s being the stress values for each CP defined by Equation 3, where F = force and A = cross-sectional area of the cylindrical specimens. The dimensions of the samples were measured to calculate the cross-sectional area with the aid of a caliper in three regions of the diameter, with an error range of ± 0.05 mm.

$$RC = \frac{F}{A} \tag{3}$$

3 RESULTS AND DISCUSSIONS

3.1 Specific gravity and granulometry test

The specific gravimetry obtained by the pycnometer method for CPII Portland Cement was 3.05 g/cm^3, for medium sand it was 2.59 g/cm^3, and for marble residue it was 2.58 g/cm^3. Bauer [11] mentions that the common density for hardened concrete is approximately 2.2 g/cm^3, and it may also vary according to the composition and application of the concrete in values ranging from 0.3 g/cm^3 to 5.5 g/cm^3.

The values of retained percentage (%) of fine aggregates obtained in the granulometry test are shown in Figure 3, in which the grain size for RRO was 254 μm and the grain size for sand was 252 μm, indicating that the particle sizes are very similar, and do not influence the consumption of cement to acquire mechanical resistance.

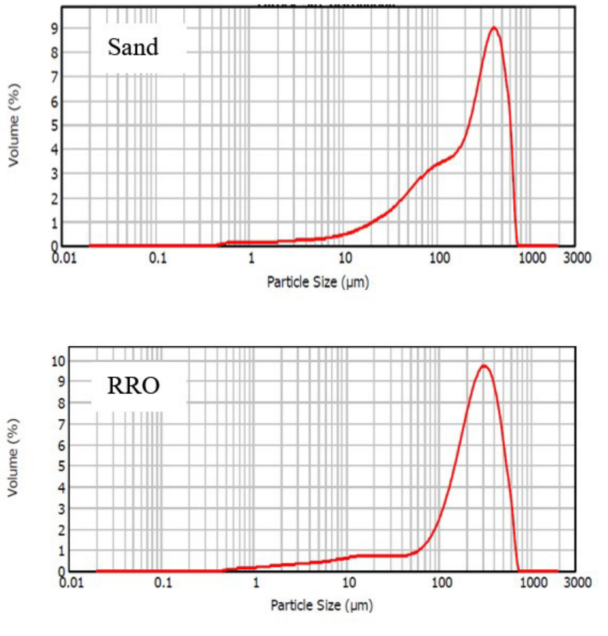

Figure 3. Particle Size Distribution of Sand and Ornamental Rock Residue (RRO).

In the analysis carried out through the granulometry test, it can be seen that the classification equivalent to the soil for the fine ornamental rock residue and for the sand are in accordance with ABNT NBR 6502 [25], both fall within the coarse grain. However, the fine rock residue has more than 12% of fines in its composition and can therefore be classified as silty sand or clayey gravel. The sand has a percentage of fines less than 12% in its composition and can therefore be classified as silty or clayey sandy granular soil.

The specific mass of the sand is similar to the marble residue, it is expected that the density does not influence the properties of the concrete by replacing the fine aggregate. However, the particle size of the residue is smaller than the sand, and this may influence the properties of the concrete.

D'Agostin et al. [26] state that the addition of waste improves the fluidity of fresh concrete. Gonçalves et al. [27] noted that the partial addition of residue increases cohesion and consistency in concrete, due to the presence of fines. However, the addition of more than 10% by mass of the residue in the concrete mass leads to a decrease in the mechanical properties. Hameed et al. [28] studied self-compacting concrete, and confirmed that the addition of marble powder required a higher water/cement ratio compared to conventional concrete, consequently, the compressive strength was considerably affected by the presence of the residue. Singh et al. [19] studied the replacement of 0%, 10%, 25%, 40%, 55% and 70% of river sand by waste and water-cement ratios of 0.30, 0.35 and 0.40. Their results showed that replacements between 25 and 40% of sand per residue positively influenced the investigated parameters.

Although the fine residue improves the consistency of the fresh concrete, it will require a greater amount of water to promote the approximation and involvement of the particles during the cement reactions and the curing process. There is a compromise of the water/cement ratio with the particle size to promote good concrete properties.

3.2 Crystalline phase analysis

The crystalline phases of the sand powders obtained by XRD showed patterns of Quartz (Silicon Dioxide - SiO_2) indexed in the file JCPDS 01-03-0539, and it is represented in Figure 4. The marble waste sample showed patterns with the presence of Dolomite, (Calcium Carbonate and of Magnesium $CaMg(CO_3)_2$) indexed in the files JCPDS 01-089-1305 and JCPDS 01-071-1662 as showed in Figure 5. These crystalline phases indexed for sand and marble residue are the usual ones found in the literature.

The main raw materials used in cement production are calcium carbonate and dolomite, and several studies have been carried out on the partial replacement of cement in concrete and mortar to reduce environmental problems. Marble waste, as it contains mostly calcium and magnesium carbonate, can be an aggregate that is chemically compatible with cement, and may favor hydration reactions and particle cohesion.

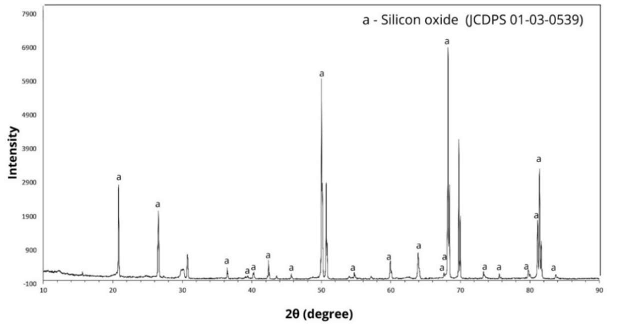

Figure 4. X-ray diffractogram obtained from sand. (a): Silicon oxide (JCDPS 01-03-0539).

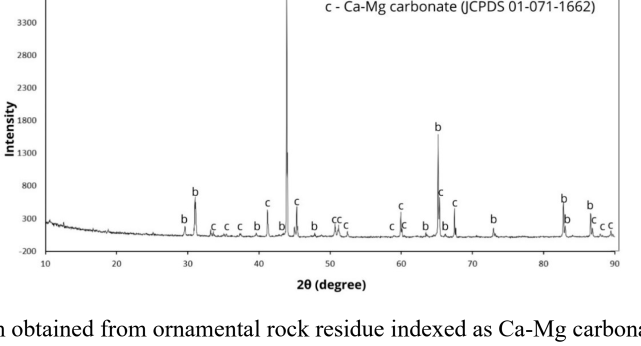

Figure 5. X-ray diffractogram obtained from ornamental rock residue indexed as Ca-Mg carbonate (JCPDS 01-089-1305 and JCPDS 01-071-1662).

3.3 Water absorption and compression strength test

The cylindrical sample and the hexagonal floor are shown in Figure 6, and it can be seen both have little porosity and a homogeneous surface, as well as the other samples studied.

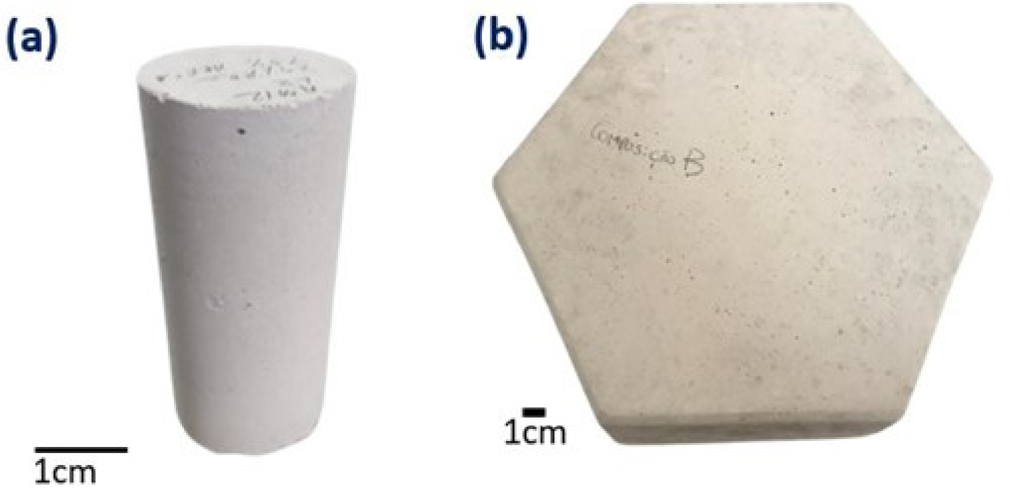

Figure 6. Models: (a) specimen and (b) hexagonal interlocking floor.

The individual values of water absorption (%) obtained in the test are displayed in Table 2, showing very low values of water absorption, less than 2.5%. According to Helene [29], results of water absorption with values lower than 4.2% are classified as durable concretes, values of 4.2 to 6.3% as normal concrete and values greater than 6.3% are classified as deficient concrete. Based on this classification, the concretes obtained in this work can be considered durable, due to values smaller than 4.2% of water absorption.

The ABNT 9781 [24] standard indicates for interlocking floors a water absorption of less than or equal to 6%. To produce concrete blocks for example, the maximum water absorption is 10%, according to NBR 6136 [30].

Table 2. Water absorption values (%) for each sample.

A1	A2	A3	B1	B2	B3	C1	C2	C3	D1	D2	D3
2.48	2.46	2.49	2.00	2.16	1.93	2.38	2.38	2.50	2.43	2.22	2.11

The individual compressive strength results of the tested samples are shown in Table 3. Concretes obtained with CPIII-32 RS cement can present approximately 32 MPa of compressive strength. The reference samples (A) containing sand and gravel in the composition, had resistance values up to 33.2 MPa. The specimens (B) presented up to 39.1 MPa; (C) up to 29.1 MPa and (D) up to 31.5 MPa. This result shows that the proposed compositions (A) and (B) presented some samples with values higher than expected for the cement used, except for samples (C) and (D), and in the latter sample the value was very close to 32 MPa.

Table 3. Compressive strength values (MPa) for each sample.

A4	A5	A6	A7	A8	B4	B5	B6
31.63	**33.22**	**29.53**	**28.25**	**28.94**	**34.67**	**28.34**	**32.67**
B7	B8	B9	C4	C5	D4	D5	D6
39.16	37.01	28.03	29.11	27.31	25.94	27.23	31.49

In the compression test, ceramic materials undergo brittle fracture, with little or no absorption of energy by plastic deformation. This type of behavior in terms of mechanical strength is also characteristic and predictable for concretes.

According to Moura et al. [6], the proportion of sand and fines that promoted a lower void ratio in the mixtures, determined by the MB 3324 method [31], was considering 50% of silty sand and 50% of crushed fines. This better packing of fine aggregates can also influence the mechanical strength of concrete, and this may have been the reason why composition B (with 100% replacement for coarse aggregates and 50% for fine aggregates of sand and marble residue) had the best compressive strength result compared to the other studied compositions.

The average water absorption values (%) and the average compressive strength values (MPa) for each composition are shown in Figure 7. It should be noted that all compositions studied show average compressive strength values very close to cement resistance, indicating the necessity of some processing adjustment to improve concretes properties.

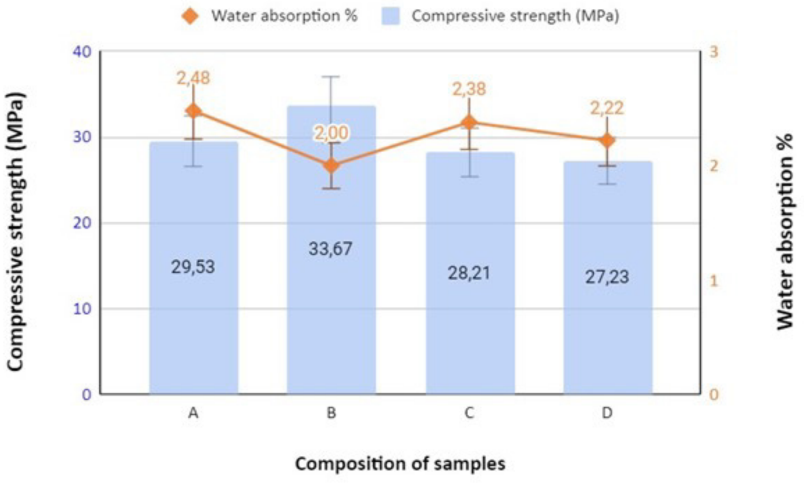

Figure 7. Average values and standard deviations of the compressive strength and water absorption results of the samples.

According to the ABNT NBR 9781 [24] standard, it determines values of minimum compressive strength greater than 35.0 MPa for 28 days for interlocking floors, showing that the compositions proposed in this work (A-D) could not be used for interlocking floors using cement CPIII 32 RS. Moura and Leite [13] mentioned that to produce concrete blocks, for example, the average compressive strength must be at least 2.5 MPa [30], showing that the studied concretes have the potential to be used in the composition of blocks.

The low water absorption values obtained in the tested concretes (less than 2.5%) and the compressive strength values (from 27 - 33 MPa) close to the resistance value of the CPIII 32 RS cement (32 MPa) show that it is possible the insertion of marble waste in concrete composition, considering several common applications (low-load paving, blocks, etc.), but it could not be applied to interlocking flooring because it does not reach 35 MPa of resistance. For interlocking flooring, the concrete containing marble must be produced with higher performance cement to obtain at least 35 MPa of compressive strength considering light traffic, or more than 50 MPa for heavy traffic.

Samples B, C and D have respectively 69%, 51% and 81% of replacement of marble waste (fine + coarse) in relation to concrete with traditional aggregates (A). The possibility of using fine and coarse aggregate in the composition of concrete has a decisive impact on the cost of the product.

4 CONCLUSIONS

In this work, a significant amount of marble waste was used to replace traditional concrete aggregates (sand and gravel). It was verified that the specific mass of sand and marble waste are similar, and this does not influence the properties of concrete. The particle size of the marble residue is smaller than the average sand used to make the concrete, and it can influence the amount of water needed for the fresh consistency of the concrete, requiring greater care in the mixing and molding stage, since the same water/cement ratio was used for all conditions (w/c=0.4).

The main raw materials used in cement production are calcium carbonate and dolomite, and several studies have been carried out on the partial replacement of cement in concrete and mortar to reduce environmental problems. Marble waste, as it contains mostly calcium and magnesium carbonate, can be an aggregate that is chemically compatible with cement, and may favor hydration reactions and particle cohesion.

The water absorption of concretes obtained here were less than 2.5%, and values smaller than 4.2% of water absorption can be considered durable concrete. Concretes obtained with CPIII-32 RS cement can present a compressive strength of approximately 32 MPa. The compressive strength shows that the proposed compositions (A) and (B) presented some samples with values higher than expected for the cement used, except for samples (C) and (D), and in the latter sample the value was very close to 32 MPa. The concrete with marble waste could not be applied to interlocking flooring because it does not reach 35 MPa of resistance. It was expected that with the value of C = 392kg/m^3, a higher compressive strength value would be obtained than that achieved (27 to 33MPa), indicating the necessity of some processing adjustment to improve the concretes properties, opening up new fields of study such as concrete made from higher performance cements, studies with different grain sizes and compositions.

Composition B, which uses 100% replacement of coarse aggregate and 50% replacement of fine aggregate and sand, obtained the best result, both in terms of compressive strength (MPa) and water absorption (%). Optimized particle packing (fine dust of marble waste with coarser particles from sand) can also influence the mechanical strength of concrete, and this can be the reason why composition B had the best compressive strength result compared to the other studied compositions.

The contribution of marble residue in the composition of concrete is essential, especially for products that do not require very high compressive strength such as blocks, low-load paving, sidewalks, coated paving among others, because the use of waste can represent more than 50% of the raw material used to produce concrete, considering the insertion of fine and coarse aggregates. However, for interlocking floors, marble residue is not recommended because it has lower compressive strength than that indicated by the ABNT NBR 9781 standard.

The use of marble residues into concrete reduces the environmental impacts caused by mining companies, reduces the amount of raw material exploited from the environment, adding value to the product, with potential cost reduction.

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