Production and characterization of artificial stone for coating with limestone waste laminated in polymeric matrix

Produção e caracterização de pedra artificial para revestimento com resíduo calcário laminado em matriz polimérica

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Abstract

Exploiting laminated limestone from Cariri Ceará for ornamental and coating purposes has generated about 2.9 million tons of waste, which is discarded of improperly, making it important to search for alternatives to this material. Thus, this work aims to develop artificial stone for wall and floor coating, using laminated limestone waste (Cariri Stone) and a polyester resin. Initially, Cariri stone waste was characterised, and then artificial stone was made with 25% resin and 75% waste, by volume. Water absorption tests and resistance to compression, flexion, abrasion and hard body impact were performed on the composites produced. The results showed that the artificial stone presented water absorption about 5 times lower and abrasion resistance 448% higher than the natural stone, compressive and flexural strength 38.8% and 72.9% lower than the natural stone, respectively, and energy values equivalent to the commercial natural stone in the hard body impact test. It can be observed that the composite produced has a technical potential for use as a coating in civil construction, after adjusting the production process of the artificial stone.

Keywords: Cariri stone. Unsaturated polyester resin. Ornamental stones.

Resumo

A exploração do calcário laminado do Cariri Ceará para fins ornamentais e de revestimento gerou cerca de 2,9 milhões de toneladas de resíduos, que são descartados de forma inadequada, tornando importante a busca por alternativas a este material. Assim, este trabalho teve como objetivo desenvolver uma pedra artificial para revestimento de paredes e pisos, utilizando o resíduo de calcário laminado (Pedra Cariri) e uma resina de poliéster insaturada. Inicialmente, caracterizou-se o resíduo de Pedra Cariri e, em seguida, a pedra artificial foi confeccionada com 25% de resina e 75% de resíduo, em volume. Ensaios de absorção de água e resistência à compressão, flexão, abrasão e impacto de corpo duro foram realizados nos compósitos produzidos. Os resultados mostraram que a pedra artificial apresentou absorção de água cerca de 5 vezes menor e resistência à abrasão 448% maior que a pedra natural, resistência à compressão e flexão 38,8% e 72,9% menor que a pedra natural, respectivamente, e valores de energia equivalentes à pedra natural comercial no teste de impacto de corpo duro. Pode-se observar que o compósito produzido possui potencial técnico para uso como revestimento na construção civil, após adequação do processo produtivo da pedra artificial.

Palavras-chave: *Pedra cariri. Resina de Poliéster insaturada. Rocha ornamental.*

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Introduction

While civil construction is one of the most important sectors for the social and economic development of a country, it is also responsible for a huge exploitation of natural resources (SILVA; SANTANA; POVOAS, 2019). In addition, the absence of efficient management programs results in increasing generation of waste that is not properly used and is improperly disposed of (MARIANI *et al*., 2019). Pinto and Lima Júnior (2014) pointed out that more than 50% of natural resource use is due to activities in the civil construction sector. According to the Brazilian Association of Public Cleaning and Waste Management - ABRELPE (ASSOCIAÇÃO BRASILEIRA DE LIMPEZA…, 2018), in 2017, around 45 million tons of construction waste were collected in Brazil.

Within this context, the exploitation of laminated limestone from Cariri Ceará for ornamental and coating purposes, specifically in the cities of Santana do Cariri and Nova Olinda, is one of the activities linked to local civil construction that generates a large amount of waste in the region. Silva *et al*. (2008) indicated that the volumes of the largest particles at that time were 755,000 m³ and 275,000 m³ in Nova Olinda and Santana do Cariri, respectively, which represents a total of 2.9 million tons of Cariri Stone Waste (CSW).

These wastes are discarded in inappropriate places, causing damage to the environment and hindering the advancement of exploration. In addition, liquid effluents, generated from powder, cause silting in streams around the sawmills and damage the soil cover, causing damage to the vegetation (MENEZES *et al*., 2010). Thus, seeking to reduce this environmental impact, alternatives must be sought to reuse these industrial wastes (GOMES *et al*., 2018).

Moura, Leite and Bastos (2013), Menezes *et al*. (2010) and Silva *et al*. (2008) evaluated the use of CSW and identified that this material can be used as a raw material in cement, concrete, ceramic block and mortar production. However, to the best of our knowledge, there is no research on the use of CSW in the manufacture of artificial stones, which is the final object of rock exploration in the region. These stones can be formed by a high content of natural aggregates and a low content of polymeric binder in their composition, presenting good mechanical properties and low porosity, compared to natural stones (RIBEIRO, 2015). Da Cunha Demartini, Rodríguez and Silva (2018), Gomes *et al*. (2021) and Shishegaran *et al*. (2022) proved the technical feasibility of producing artificial stone from the agglutination of resin and rock wastes, showing that this stone can be a technically, economically and ecologically viable alternative.

Data from the Brazilian Association of the Ornamental Stone Industry - ABIROCHAS (ASSOCIAÇÃO BRASILEIRA DA INDÚSTRIA…, 2018) show that, in Brazil, the production and exports of artificial rock materials are still not very relevant. On the other hand, Brazilian imports of these materials are significant. In the first half of 2018, imports of artificial rock materials accounted for 31.2 thousand tons, moving approximately US$ 21.8 million, while exports of these materials, in the same period, reached only 2,964.7 tons, valued at about of US$ 1.7 million. Thus, studies aimed at increasing the national production of artificial stones are of the utmost importance to reverse this scenario and provide more affordable prices for this national product.

Therefore, aiming to align the reduction of the environmental impact caused by civil construction and the increase in the national production of artificial stones, this study aims to develop an alternative artificial stone, using laminated limestone waste (Pedra Cariri) and a polymeric matrix, which presents technical feasibility to be used as a coating in civil construction.

Experimental program

Materials

Two types of waste from processing Pedra Cariri were used (plate chips and powder from the cut), both collected from a quarry located in the municipality of Nova Olinda/CE. The plate chips (Figures 1a) were crushed in a mechanical crusher (jaw mill), in which each chip passed between the mill's jaws only once, with the spacing between the jaws being set equal to the DMC of the aggregate (12.5 mm). Then, the material was sieved in sieves with an opening of 12.5 mm; 4.75 mm and 0.15 mm, generating material in three granulometric ranges: material passing through the 12.5 mm sieve and retained on the 4.75 mm (coarse aggregate – Figure 1b); passing through the 4.75 mm sieve and retained on the 0.15 mm (fine aggregate - Figure 1c) and passing through the 0.15 mm sieve (powder - Figure 1d). The powder was sieved only in the sieve with an opening of 0.15 mm, having been joined to the sieved material from the chips.

Figure 1 **- Cariri stone waste**

Table 1**- Properties of crystal orthophthalic unsaturated polyester resin**

Properties	Values	Properties	Values
Viscosity sp3/12 CSW (cP)	1100-1600	Maximum strength (MPa)	55
Density (g/cm³)	1.10-1.12	Modulus of Elasticity (GPa)	1.9
Gel Time (minutes)	9-14	Flexural strength (MPa)	80

Source: Oswaldo Cruz Química (2021).

The resin used was crystal orthophthalic unsaturated polyester (FORTCOM 6100) available in the local market and at a lower cost compared to other resins. The main properties of the resin are shown in Table 1. As recommended by the manufacturer, methyl ethyl ketone peroxide in liquid form (MEKP peroxide) was used as a curing activator in a proportion of 1% in relation to the resin.

The commercial Cariri stone (Figure 2) was used as comparative material in relation to the artificial stone properties produced from the same company where the waste was collected.

Research method

Slab production

Using EMMA software (Elkem Materials Mixture Analyser), the particles were packed to obtain the percentages of the granulometric ranges that provided a lower void index. After defining these percentages, 8 tests were carried out, varying the amount of resin and waste, using the quality of the surface finish (quantity and size of voids and homogeneity) as a criterion to select the best artificial stone. Thus, it was defined that 25% of resin and 75% of waste would be used, in volume, and the waste comprised 30% of coarse aggregate, 40% of fine aggregate and 30% of powder. Thus, 20 artificial stone slabs were produced in moulds measuring 155 mm x 135 mm x 20 mm (length x width x thickness), using a constant mass of resin equal to 134 g and waste equal to 839 g for each artificial stone, compacted in one layer.

The sequence used to prepare the composites is shown in Figure 3, which took place as follows:

(a) initially, the MEKP curing trigger and the resin were manually mixed in a container for 30 seconds;

(b) the CSW waste was added to the mixture;

(c) the three elements were manually mixed for approximately 2 minutes, until the waste particles were totally wet;

(d) the mixture was placed in the capped mould;

(e) the mould was taken to a hydraulic press and a compressive stress of 0.8 MPa was applied until the slab reached the desired thickness;

(f) the mould was removed from the press and placed in the oven for 2h at 80 °C to ensure the complete cure of the resin and to remove the remaining moisture, which increases the intermolecular forces;

(g) the slabs were left at room temperature for cooling, and

(h) the slabs were sanded and only then cut to the sizes of the test specimens, resulting in the final product, shown in Figure 4.

The specimens were cut in length and width directions.

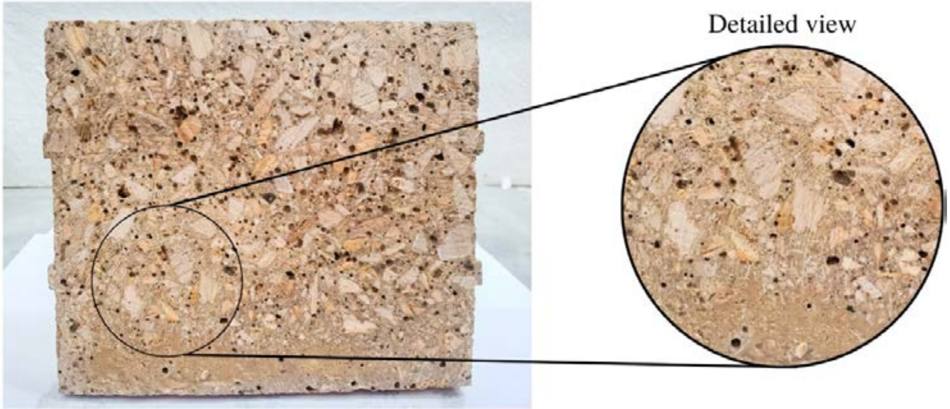
Characterisation of the waste

The characterisation of Cariri stone waste was based on granulometric analysis, specific mass and water absorption tests. Regarding the Los Angeles abrasion test, other authors already found values greater than 60%, indicating that the material does not have good resistance to frictional wear (OLIVEIRA, 2016).

Figure 2 **- Commercial Stone**

Figure 3 **- Slab production**

Figure 4 **- Artificial stone**



The determination of the waste granulometry was carried out using the sieving method, according to the NBR NM 248 technical standard (ABNT, 2003), which establishes the method for determining the granulometric composition of fine and coarse aggregates. The specific mass of the waste and water absorption of the coarse aggregates were determined according to the NBR 16917 standard (ABNT, 2021a). The water absorption of fine aggregates was obtained from the NBR 16916 standard (ABNT, 2021b).

Characterisation of artificial and commercial slabs

According to Ribeiro (2015), the tests recommended for artificial rocks depend on for what purpose they are required, in which uniaxial compressive strength and flexural strength are the tests common to all types of use. In addition to these, abrasion wear and hard body impact tests are desirable for wall and floor coverings.

Thus, as Cariri stone is mainly used in external coatings and floors, the following physical and mechanical properties of artificial stones were analysed: water absorption, compressive strength, flexural strength, abrasive wear and hard body impact resistance.

The water absorption of artificial and commercial stones was evaluated, for comparative purposes, according to NBR ISO 10545-3 (ABNT, 2020a), using 5 specimens of each type measuring 155 mm x 135 mm x 20 mm (length x width x thickness).

The simple compressive strength test is not included in NBR ISO 10545 (ABNT, 2020a, 2020b), which sets the tests for ceramic cladding slabs, however, in the literature, there are studies that carried out this test in order to verify this property in the cladding slabs produced with wastes of natural rocks in polymeric matrix.

Therefore, in this work, the compressive strength test was carried out based on studies by Demartini (2017) and Ribeiro (2015). Twelve cubic specimens with 20 mm edges were used, which were broken in the universal testing machine (EMIC), as shown in Figure 5. At the end of the test, in each specimen, the maximum rupture stress was calculated by the relation between the maximum force obtained at rupture and the area of the specimen face subjected to loading. After an analysis of variance (ANOVA), outliers were removed. Thus, from the 12 specimens tested, two values were excluded, and the average was taken with the results of the 10 remaining specimens.

The three-point bending test consists of the application of an increasing load at certain points of a bar of standardised geometry. The applied load starts from an initial value of zero and slowly increases until the specimen ruptures. This test is mainly used to evaluate mechanical properties of brittle and ductile materials (COSTA, 2017). For this article, the test was run following the NBR 10545-4 recommendations (ABNT, 2020b), which specifies a method for determining the flexural strength of ceramic tiles for coating, in which 12 specimens were made for each sample measuring 70 mm x 20 mm x 20 mm (length x width x thickness) and the average of the results were found, disregarding two outliers.

According to NBR 12042 (ABNT, 2012), wear is the surface disaggregation and removal of particles from a given material subjected to frictional forces. The abrasion resistance test aims to determine the ability of the rock material not to wear out when rubbed by particles of high hardness, rubbed against it mainly by the movement of people and vehicles, resulting in loss of brightness and thickness (DEMARTINI, 2017).

For this article, the test was performed on an Amsler Machine (MAQTEST) in accordance with NBR 12042 (ABNT, 2012), in which two specimens of each sample were tested measuring 70 mm x 70 mm x 40 mm (length x width x thickness). Figure 6 shows examples of specimens before and after the test.

The hard body impact resistance test was carried out in accordance with NBR 15845-8 (ABNT, 2015), using 5 specimens for each sample measuring 155 mm x 135 mm x 20 mm (length x width x thickness). Figure 6 shows how the specimens were after the test was performed. It is noteworthy that this test is recommended by NBR ISO 10545 (ABNT, 2020a, 2020b), which sets the tests for ceramic tile coating. However, due to equipment availability, the test was performed using the equipment recommended by NBR 15845-8 (ABNT, 2015), as in other studies in the literature.

Figure 5 - **Compressive strength test**

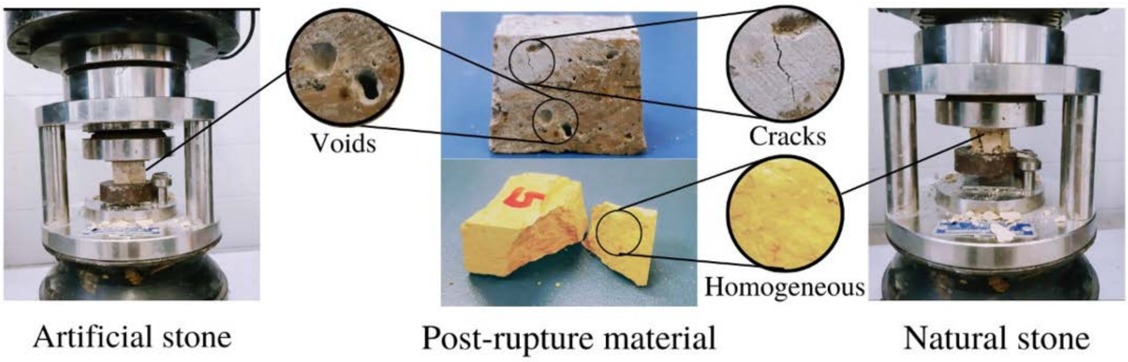
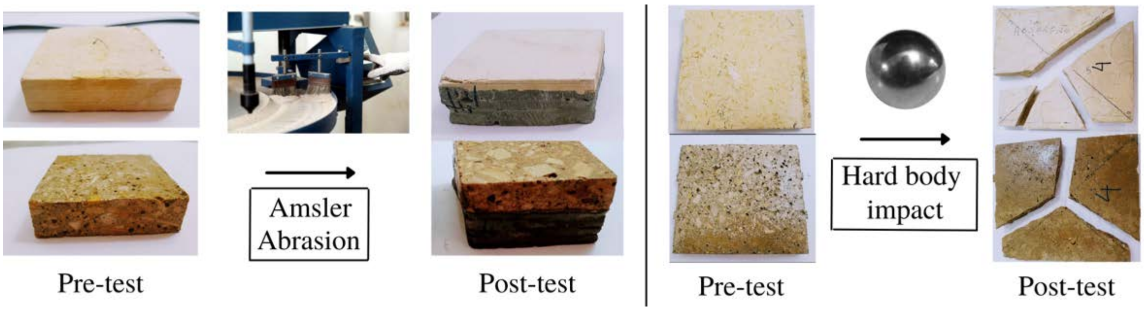


Figure 6 - **Abrasion resistance and hard body impact test**



Results and discussion

Characterisation of the waste

The water absorption of the coarse aggregate from Cariri stone was 5.86% while that of the fine aggregate was 6.41%. The explanation for this high absorption is due to the fact that sedimentary rocks, in particular laminated limestone, are porous materials (OLIVEIRA, 2016). This high absorption may have contributed to the increased amount of resin when artificial stones were produced. Oliveira (2016) found, on average, water absorption values of 5.4% for the large fraction and 9.7% for the fine fraction.

Similar to Farias (2017) and Moura, Leite and Bastos (2013), the value found for the specific mass of the Cariri stone waste was 2.67g/cm³.

Figure 7 shows the granulometric curve of the Cariri stone waste used, considering the three aggregates (coarse aggregate, fine aggregate and powder), shown in Figure 1. The maximum aggregate diameter (MAD) was 12.5 mm, the fineness was 3.34, presenting a smooth curved graph that indicates a continuous distribution of the aggregates. This distribution favors the packing of the grains, providing a lower void index. It can be observed that there is a significant amount of fine materials (diameter less than 0.15 mm), which can provide an adequate destination for one of the most representative portions of the waste: the powder. In addition, materials with this composition tend to have lower amounts of voids, having a more homogeneous particle distribution (RIBEIRO, 2015).

Characterisation of artificial and commercial slabs

Regarding the water absorption test, the artificial stone presented an average value of 0.99% with a standard deviation of 0.38, while the commercial stone obtained an absorption of 5.20% with a standard deviation of 0.40, which is equivalent to an absorption 5 times higher than that of artificial stone. The difference in the values of the absorption results may be associated with waste replacement, a material that has a high rate of water absorption, by resin, and a material with practically zero absorption (SANCHEZ *et al*., 2010).

The compressive and flexural strength results of the artificial and commercial stone are presented in Figure 8. From the average values, it can be observed that the commercial stone had better compressive strength results (31.96 MPa) compared to the artificial stone (12.45 MPa), which is 157% higher. Regarding the flexural strength, the natural stone presented the value of 4.35 MPa and the artificial stone 3.17 MPa, which is 37% higher. It is assumed that this occurred due to the low energy of compaction and vibration when producing the artificial slabs, as these steps are important for the complete accommodation of the particles and filling of the voids and the reduced consumption of resin, due to economic factors, as resin is a very expensive item and has an impact on the production cost of artificial slabs.

Figure 7 **- Granulometric Curve**

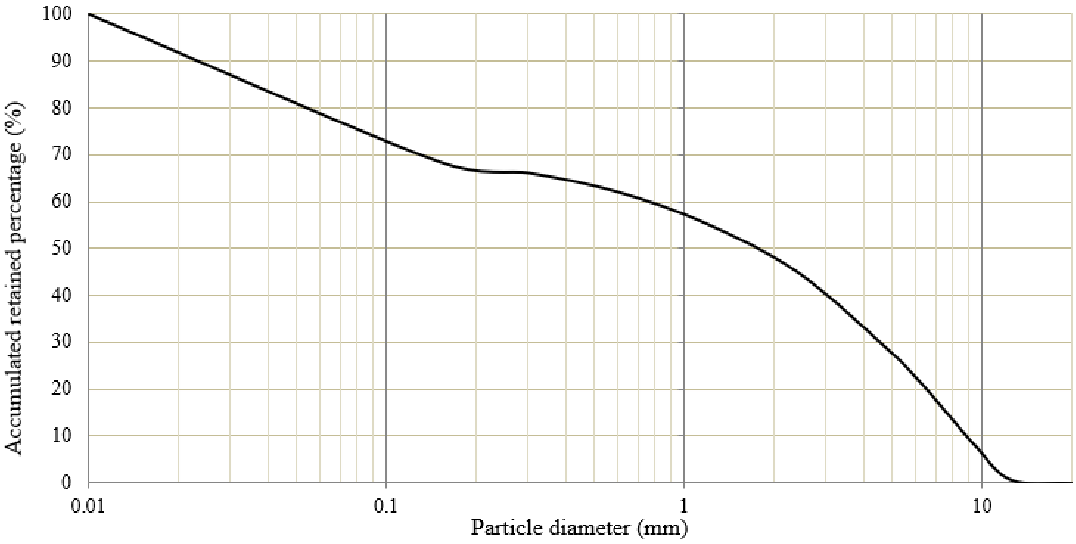
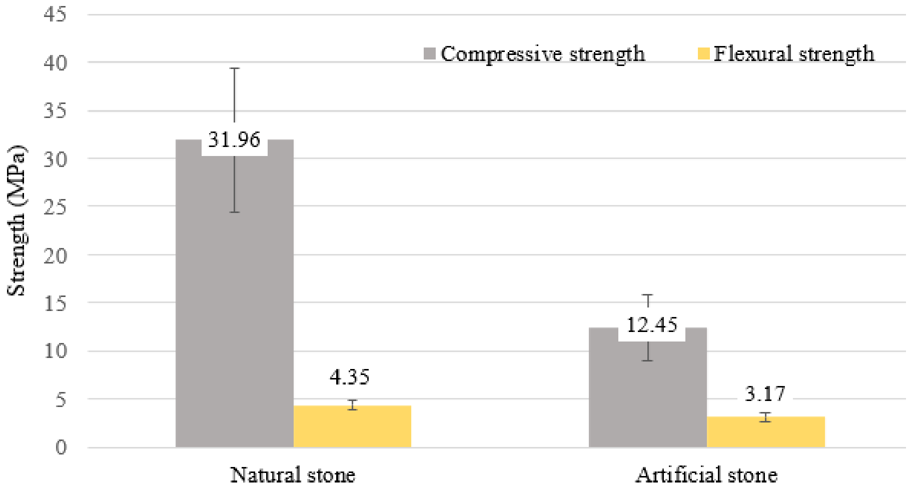


Figure 8 - **Compressive and flexural strength results**



Although commercial stone has high-water absorption due to the amount of pores found in the material, artificial stone voids have larger sizes, which reduces the mechanical strength of this composite. Borsellino, Calabrese and Di Bella (2009) found low flexural strength values due to large voids found in their artificial stones. Ribeiro (2015) states that greater packing leads to an increase in mechanical properties. Therefore, for the mechanical strength of artificial slabs to increase, it is necessary to reduce the number of voids in the samples and/or increase the amount of resin.

In addition, it was observed that the artificial slabs always ruptured in the aggregate, that is, in the Cariri stone waste, which is a brittle material, as reported in the literature in the research by Oliveira (2016) and Farias (2017). It was also noticed that, during the test, the artificial slab samples showed characteristics of a ductile material, because, after the maximum stress peak, the material continued to strain. On the other hand, the commercial slab samples did not strain after reaching the maximum stress peak, resembling a brittle material. Figure 5 shows this as the natural stone disintegrated, while the artificial stone showed only cracks. The ductility of commercial stones may be related to the presence of resin, which has a low modulus of elasticity (1.9 GPa) compared to sedimentary rocks (greater than 30 GPa) (SIMÕES FILHO, 2013).

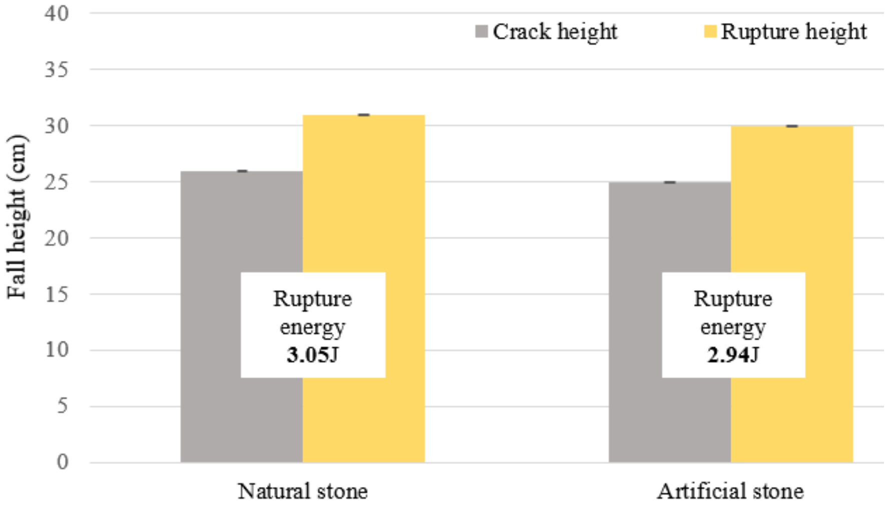
There are no standardised limit values for the compressive strength of these slabs. However, when comparing with the results found in the literature, it can be seen that those of artificial stone, obtained in this study, have low strengths. Demartini (2017) found an average of 96.49 MPa for artificial marble stone, while Ribeiro (2015) obtained average values of 14.17 MPa, 15.10% lower than the artificial marble stone under study. This low mechanical strength was expected given the number and size of voids found in the artificial stone samples.

Lee *et al.* (2008) found flexural strength values of 27.9 MPa and 46.3 MPa for artificial stones made from glass powder waste, granite aggregates and unsaturated polymeric resin, using a pressing stress of 4.9 MPa and 9.8 MPa, respectively. The authors concluded that greater flexural strength is also associated with greater pressing stress. Therefore, as in this research the compressive stress was on average 0.8 MPa, it can be said that the flexural strength results found are consistent and that the increase in pressing stress may result in flexural strength values higher than those found.

Figure 9 presents the results of hard body impact resistance of the studied stones. It can be seen that both natural and artificial stone presented very similar results, with average rupture energy of 3.06 J and 2.94 J, average rupture height of 0.31 m and 0.30 m and cracking height of 0, 26 m and 0.25 m, respectively. It is believed that the low crack height is due to the mineralogical constitution of the material, since, in limestone, the high porosity facilitates crack formation as the pores act as stress concentrators (COSTA *et al.*, 2021). However, these pores also act as barriers to crack propagation. Thus, the character of these laminar materials provides a series of crack propagation planes, which consume impact energy and minimise damage to the material as a whole.

These results are consistent with those obtained by other authors who used different types of waste, such as Agrizzi (2020) who developed artificial stones from quartzite waste and, when evaluating the hard body impact resistance, realised that the stones had a rupture energy of 3.856 J for the average failure height of 0.393 m, while the natural rock presented failure energy of 1.961 J and failure height equal to 0.2 m.

Figure 9 - **Hard body impact resistance results**



The abrasion resistance results (Figure 10) indicate that the artificial stone performed better with average values of wear of 1.64 mm and 3.01 mm, for cycles of 500 meters and 1000 meters, respectively, while the natural stone showed a loss of 7.89 mm and 13.50 mm at 500 meters and 1000 meters, respectively. These values indicate that artificial stone is 448% more resistant to abrasion than natural stone, commercialised on a large scale. Another study involving artificial rocks, based on granitic rocks, found little variation in resistance, ranging from 1.13 mm (natural rock) to 1.46 mm (artificial rock), in a cycle of 1000 meters (GOMES *et al.*, 2021), demonstrating a greater performance gain in the composite developed in this study. This gain in abrasion resistance occurs due to the presence of the resin that has greater mechanical strength. It is worth noting that the abrasion resistance values are closely related to the mineral hardness and material porosity (COSTA *et al.*, 2021).

Boxplots were constructed showing the results of 20 compressive strength test samples, 20 flexural strength samples and 10 rupture energy samples, obtained from the hard body impact test, considering natural stone and artificial stone samples. Figure 11 shows boxplots, which are intended to show the significant differences between the slabs with natural material and the slabs with artificial material. In the boxplot, the upper and lower limits represent the maximum and minimum value of the sample, respectively, and "x" indicates the mean value. The horizontal lines of the rectangle show the values of the 1st quartile, median and 3rd quartile.

The data from the three natural stone graphs are asymmetrical as most of the values are located at the top or bottom of the graph. On the other hand, the data from the artificial stone are more symmetrical, indicating that the set is normally distributed. Regarding the data dispersion, the rupture energy of the natural stone presented the biggest difference between the third quartile and the first quartile (box size), indicating that it had a greater variability when compared to the others. The data from the artificial stone presented the best results for this parameter. Regarding the standard deviation, the compressive strength test showed the highest values, with 7.5 and 3.5 for natural and artificial stone, respectively. For the bending test, the deviation values were approximately equal for the two samples, around 0.50. The standard deviation values for the rupture energy were 0.62 for natural stone and 0.35 for artificial stone.

Figure 10 **- Abrasion resistance results**

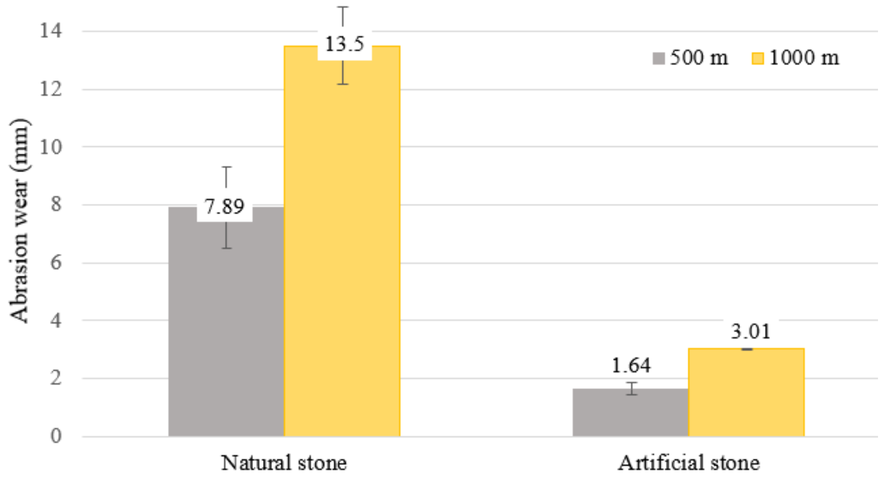
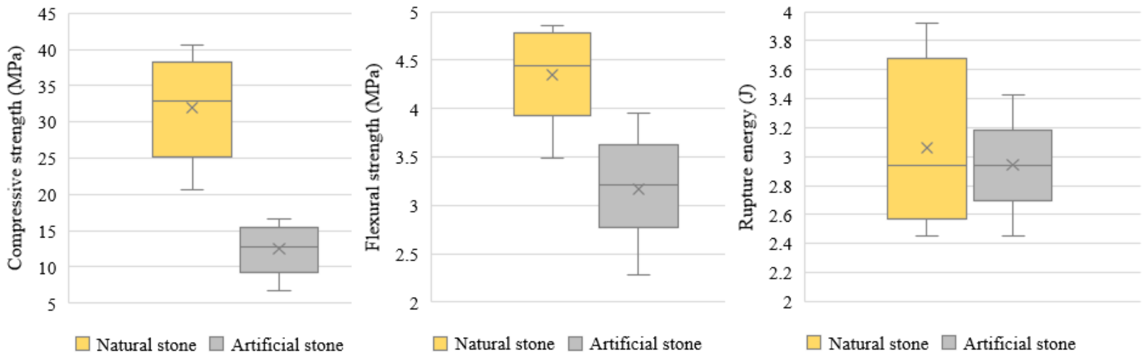


Figure 11 **- Boxplots for natural and artificial stone for compressive, flexural and rupture energy strengths**



Conclusion

According to the results obtained in the tests carried out, it can be concluded that composites produced with Cariri stone waste and unsaturated polyester resin presented compressive and flexural strengths of 12.45 MPa and 3.17 MPa, with 38.82% and 72, 87% lower than natural slabs, respectively, however these values are still compatible with the application for covering walls and floors, as this material property has little influence on the system's performance. Regarding the hard body impact results, both samples showed equivalent values, with the rupture energy of the artificial stone in the value of 2.94 J. The abrasion resistance results indicated that the artificial stone is 448% more resistant to abrasion than the natural one, for the 1000 m cycle. As for the water absorption test, the artificial stone performed better than the commercial stone, showing absorption approximately 5 times lower. The performance of artificial slabs can be improved with an adjustment in the granulometric composition of the Cariri stone and/or with improvements in the slab production process, such as an increase in compaction stress during slab manufacturing or with an increase in the resin content, which would lead to an increase in the commercial value of the artificial stone, as it is this material that has the highest cost. Another factor that can be improved with adjustments in the production process is the uniformity of the slabs, which today still represents a limitation of the composite produced by the production method used.

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