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Mechanical and physical investigation of an artificial stone produced with granite residue and epoxy resin

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Abstract
The incorporation of ornamental stones residues to a polymer resin to produce artificial stone is an alternative to reducing the environmental impacts of inadequate waste disposal; it also represents a technically viable method. The objective of study was to develop an artificial granitic stone (AGS) using 85% in mass of granite agglutinated by 15% epoxy resin, through a vacuum, vibration and compression process. The artificial stone presented superior properties when compared with the natural granite, it had satisfactory mechanical and physical properties, resistant to temperature oscillations, to impact and wear, therefore suitable for applications for flooring subject to instantaneous tension and high-traffic.

Keywords
Artificial stone, residue, granite, epoxy resin, properties

Introduction

Brazil is one of the largest natural stone producers for cladding and decoration purposes in the world.[1] On the first process for production of ornamental stone, extraction, around 40 to 60% is residue, while during the final processes (cutting, polishing, and finishing), this value of 30 to 40%.[2] However, much of this type of residue is discarded in a faulty manner, causing environmental problems.[3] It is estimated that in Brazil from the production process to obtaining the slabs in the comercial dimensions there is a raw material loss of 83%.[4]

A sustainable alternative intended to minimize the quantity of residues is to recycle and re-introduce them as reusable raw materials in other production processes.[5] This research proposes the use of particles of this granite residue agglutinated by epoxy resin to manufacture an artificial stone that could be used in construction.

Epoxy resin is a thermo-rigid material with excellent mechanical, thermal and chemical strength and also resistant to corrosion. It also has low viscosity, good adhesion with other materials, it can be processed under a variety of conditions, and it has low contraction during the curing process.[6] The application of epoxy resin has been gaining more space in the commercial and academic environment, and have been widely applied as a matrix to develop new composites.[7] Epoxy matrix composites when incorporated with conductive loads can also be used as electromagnetic coating, a practice widely used in electronic devices.[8]

Chauhan and Bhushan[9] verified that when carbon black particles are incorporated to carbon fiber-reinforced epoxy composites, the material displays higher mechanical performance than these conventional carbon fiber-reinforced epoxy matrix composites. Zaheer et al.[10] incorporated homogeneous nanopallets to vacuum molded composites after

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ozone-functionalization, followed by introducing glass fibers. This was very efficient because the nanopallets increased the interface of glass fiber and epoxy, and consequently increased the mechanical strength of the composite.

Artificial stones are characterized by their high natural aggregate content, about 97%, including mainly particles of marble, glass crystals, granite, quartz, and others, aggregated by a polymer resin.[11]

There are many academic researches that aim towards the production and characterization of this type of materials, using many different variations of residues and resins. Peng and Qin[12] investigated the mechanical properties of an artificial stone made of SiO_2 residue and quartz sand; Sarami and Mahdavian[13] studied the properties of artificial stones using travertine, marmarit stone with cement, water and unsaturated polyester resin. Baramaleki and Mahdavian[14] also developed an artificial stone using the same material of Sarami and Mahdavian[13] however, they used TiO_2, ZnO and SiC nano-fillers to evaluate its effects on properties. Borsellino et al.[15] developed different artificial marbles using epoxy or polyester resin varying the power concentration of marble particles.

The artificial stones also present superior technological advantages when compared to natural stones. They are lighter because they have a polymer matrix, which is a low density material. Another big advantage is the lower incidence of pores and empty area in artificial stones; these make it possible for the artificial stones to be mechanically more resistant, as these characteristics work as tension concentrators and facilitate crack expansion. Furthermore, the low porosity rate makes the artificial stones more resistant to infiltration of fluids, so they can be used in humid environments without compromising their performance long-term.[16]

It is importante to note that the artificial stone proposal is not detrimental to the environment under regular work condition, as the second study realized by Braga et al.[17] suggests. In this study granite is classified as a non-dangerous and inert material, which means that it does not react with or dissolves in water.[11] Also, the epoxy resin after its curing process is nontoxic, and in term of its chemical stability, it is one of the most inert thermo-rigid materials with good dimensional stability in service.[18]

The success of recycling techniques depends on the profit they can produce. The development of an artificial stone must take in consideration the price of the recycled materials and the market demand. The export of artificial stone materials from Brazil in the first trimester of 2019 summed 1.36 million U.S. dollars, with average price of USD790/t, while for natural stones this number was of USD451/t. In terms of imports, the total value imported was 13.02 million U.S. dollars and the avarege price was of USD641/t for artificial stone and USD555.9/t for natural stone.[19]

Based on the data previously stated, the value per ton of the artificial stone is higher that this value for natural stone. Exporting this artificial stone has also great chances of growing due to the abundance of raw materials obtained from the natural stone industry, and also due to the growing necessity of recycling these industry's byproducts into synthetic materials. Furthermore, when considering the AGS properties, the raw materials used, the electricity used during manufacturing, specialized labor, and transportation costs, it is estimated that the price of the AGS will be close to the value of artificial stones already present in the market.

The purpose of this research is to produce an artificial stone using granite residue and epoxy resin, with satisfactory physical and mechanical properties, and to compare them with natural granite that was used to produce such artificial stone.

Materials and methods

White granite

To realize this project, the residue outcome of the cutting process of granite was used, provided by the company Brumagran, Brazil. Initially, the residue was crushed and passed through a mil, later on it was sieved to obtain three distinct grain sizes: coarse (2 mm to 0.42 mm), medium (between 0.42 mm and 0.075 mm), and fine (grains smaller than 0,075 mm). The residue was left in an over at the temperature of 70°C for 24 hours to remove its humidity, so that the resin curing process and particle adhesion between granite and epoxy resin could be improved.[20]

Based on the three grain sizes (coarse, medium and fine), there were proposed ten different mixtures of these particles, based on the Simplex-centroid Model. The objective of this step was to identify the mixtures with the highest dry apparent density. The mixture that presented the highest dry apparent density of particles, 1.95 g/cm^3, was the one that contained 2/3 coarse particles, 1/6 medium particles, and 1/6 fine particles, so this was picked for the composition of the AGS.

In order to calculate the minimum resin content (MRC%) of epoxy (density: 1.05 g/cm^3), that acted as a binder in the production of AGS, first there is a need to identify the percentage of void volume (VV%) located between the particles (pores) of granite. This VV% is calculated using the equation below. The apparent density of granite used is 2.62 g/cm^3, which was calculated by pycnometry.

$$VV\% = \left(1 - \frac{dry\ apparent\ density\ of\ particles}{apparent\ density\ of\ granite}\right) \cdot 100$$

The result obtained in $VV\%$, was replaced in the next equation, which is based on the weighted average, to calculate the $MRC\%$

$$MRC\% = \frac{VV\% \ . \ \rho_{resin}}{VV\% \ . \ \rho_{resin} + (100 - VV\%) \ . \ \rho_{granite}} \cdot 100$$

The value found for MRC%, 12.08%, represents the minimum percentage of weight of resin needed to fill the void volume, however, 15% of resin was used in the formulation of AGS as a margin of safety.

Epoxy resin

The epoxy resin type Bisphenol A diglycidyl ether (BADGE) was used to agglutinate the granite particles, mixed with the hardening agent Triethylenetetramine (TETA) with stoichiometry of phr=13, both provided by EPOXYFIBER, Brazil.

Production of artificial granitic stone

The minimum resin content necessary to fill the void space between the particles was calculated. The mixture used was of 85% in mass of granite particles and 15% of resin.

This mixture was poured into a metallic mold ($100 \times 100 \times 10\,mm^3$), and vibrated for two minutes, while simultaneously it was put into a vacuum; later it was taken to a hydraulic press under a 10 MPa pressure and 90°C temperature, where it stayed for another 20 minutes.

The AGS produced were sanded and cut to the specific dimensions for the necessary tests for its characterization.

Physical properties

Apparent density, absorption water and porosity. The ABNT NBR 15845[21] standard was used to determine the apparent density, water absorption, and porosity of the AGS and of the natural granite. Ten test bodies of each material were used in the dimension $30 \times 30 \times 10\,mm^3$.

Mechanical properties

Three-point flexural strength. The flexural strength was determined for the AGS, white granite and epoxy resin. For the three-point flexural strength test, a universal test machine INSTRON model 5582 was used. The test is standardized EN 14617-2[22] and ABNT NBR 15845.[21] In all there were six test bodies for each stone used and their dimensions were $10 \times 25 \times 100\,mm^3$.

Microstructure. Samples were obtained and observed by scanning electron microscopy (SEM) in a model SSX-550 Shimadzu equipment at 20kV secondary electrons. These samples were gold plated and were taken from the fracture surface of specimens after bend rupture.

Frost resistance. To evaluate the effect of freezing and thawing cycles in the stoned the orientations followed were from the ABNT NBR 15845.[21] To verify if there was a change in the mechanical behavior of the AGS and the natural granite, the three-point flexural strength test was performed on the same conditions used previously in order to compare the results.

Abrasive wear. Two prismatic specimens underwent abrasive wear tests for each type of stone, measuring $70 \times 70 \times 40\,mm^3$, in accordance to the ABNT NBR 12042[23] standard, using a MAQTEST Amsler equipment. The specimens' thicknesses were measured before the test, and also after undergoing 500 and 1,000 meters of runway.

Hard body impact resistance. The hard body impact resistance test evaluates the material capacity to withstand instantaneous mechanical actions. The test followed the orientations from ABNT NBR 15845.[21] Three test bodies were used with dimensions $10 \times 200 \times 200\,mm^3$.

Results and discussion

Physical properties

Table 1 shows the values for physical properties of the AGS produced under the vacuum, vibration and compression conditions.

Table 1. Physical properties of AGS and natural granite.

Physical properties	AGS	Natural granite
Density (g/cm^3)	2.30 ± 0.01	2.62 ± 0.01
Water absorption (%)	0.09 ± 0.06	0.38 ± 0.02
Apparent porosity (%)	0.23 ± 0.03	0.99 ± 0.06

It can be noted that as expected, the artificial stone is less dense than the natural granite, because of the presence of the polymer in its composition, which is a lighter material; this fact makes it so that the transportation cost of this material is lower.[24] For instance, one container has a maximum weight capacity that can be transported and the freight cost varies in terms of destination and other individual characteristics of the freight. Since the AGS has a lower density than natural granite to around 14%, this would be equivalent to transporting this much more material in volume in the same freight, within the same maximum weight limit. This would make the fixed cost of freight for the AGS cheaper per volume within the same original destination and individual characteristics of the freight.

Lee and Shin[25] in his research used glass crystals and PET to manufacture an artificial stone, varying compression, vacuum and vibration levels, found values between 2.03 and 2.45 g/cm^3. The value found was within the expectation.

It can be seen that the water absorption found for the AGS (0.09% $\pm$ 0.01) was 4.2 times less than that found for natural granite (0.38% $\pm$ 0.02), which means that the AGS is more resistant to humid environments. Chiodi and Rodriguez[26] in their studies established that the stones used for cover applications are of high quality when this number does not go beyond 0.1%.

In terms of relative apparent porosity, the AGS presented the value of 0.23% $\pm$ 0.04 while for the natural granite it was 0.99% $\pm$ 0.06, which means that the porosity rate of granite is much higher, about 4.3 times more than the artificial stone. This indicates good particles and matrix adherence, and that the epoxy resin effectively filled the voids between the granite particles.

Chiodi and Rodriguez[26] classified as high quality materials those that have apparent porosity lower than 0.5%, which confirms the quality of the material produced. The results of water absorption and apparent porosity show that the material has a high level of impermeability, being less susceptible to stains, which allows the artificial stone to be used as a residue in damp places and exposed to liquids such as kitchen and washroom countertops.

The standard ABNT NBR 15844[27] specifies the typical physical properties of granite used in cladding. According to the standard the apparent density and the water absorption should not be higher than 2.55g/cm^3 and 0.4% respectively, and maximum apparent porosity of 1.0%. Thus, it can be said that the granite analyzed is in accordance to the standard and reaffirms that the artificial stone has superior physical properties.

Mechanical properties

Three-point flexural strength. In the three-point flexural strength test, through the tension deformation curves of the materials analyzed, the value obtained for flexural strength for the AGS was 32.92 $\pm$ 2.92 MPa, for granite it was 13.49 $\pm$ 1.14 MPa and for the epoxy resin it was 93.59 $\pm$ 4.7 MPa. Figure 1 presents the graph of flexural stress versus strain for pure epoxy resin, displayed with permission of Carvalho et al.,[28] together with that for the AGS and natural granite.

Once again it is clear that the artificial stone's properties are superior in comparison to the natural stone, making it evident that there was a good interaction between the particles and the matrix, and confirming that the low incidence of pores in the artificial stone created a material with better mechanical properties.

The good mechanical performance of the stone is mainly due to the methodology applied during its fabrication, especially the use of the vacuum, which reduced the number of voids, and the appropriate proportion of load/matrix, which gave it good wettability, thus, goof adhesion between the phases. It is also noted that there is little dispersion of the results obtained for the AGS, which indicates that it is a stable material, with a standardized result in the production process.

According to Chiodi and Rodriguez,[26] construction artificial stones can be classified as high resistance materials when their flexural strength is greater than 20MPa, thus, the AGS obtained with a value of 32.92MPa, approximately 60% above the recommended value, verifying that the material has good mechanical performance.

Microstructure. Figure 2 shows the micrographic analysis of the fractured region after the three-point flexural strength test, through a scanning electron microscope.

Through the microscopic images show on Figure 2 (a) and (b), it is possible to confirm the good

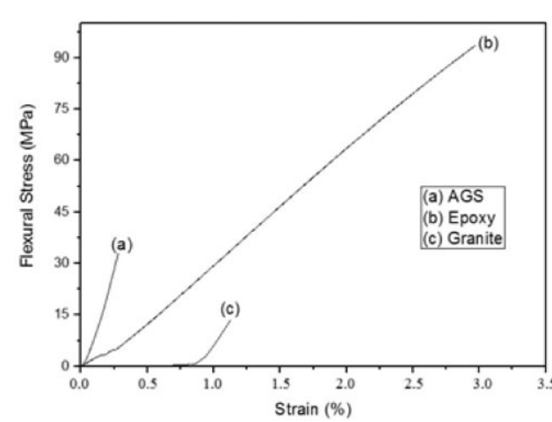

Figure 1. Flexural stress $\times$ strain curves for (a) AGS (b) epoxy and (c) granite.

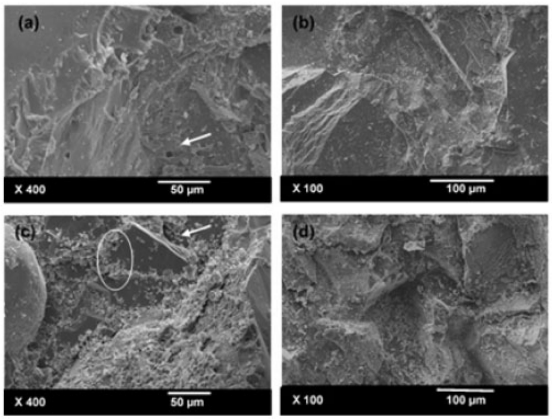

Figure 2. Microscopic images of the fracture region obtained through scanning electron microscope (a) of the artificial stone AGS with zoom 400×, (b) AGS with zoom 100×; (c) of the granite with zoom 400×, and (d) granite with zoom 100×.

interaction between the granite particles and the epoxy resin. This fact indicates that there was a good homogeneity in the mixture. Also, it can be seen the low incidence of pores, they appear in isolated form, which is determinant to the low water absorption rates and apparent porosity. Using vacuum could have contributed to the air removal and consequently lowering the amount of empty space between the particles.

On the other hand, it can be seen on Figure 2(c) and (d) the microscopic images of the granite, and it shows higher pore incidence and higher amounts of voids, opposite to what happens in the AGS.

Frost resistance. Upon freezing and thawing cycles, no alterations were observed, such as fissures or detachment of fragments of the test bodies.

The three-point flexural strength test was performed using the same standard that was applied to the test bodies that did not go through freezing and thawing cycles. The values obtained for flexural strength for the granite was 12.9 ± 1.34 MPa and for the AGS it was 32.49 ± 2.00 MPa while the values found for the bending test at room temperature was 32.92 ± 2.92 MPa for AGS and 13.49 ± 1.14 MPa for granite, which means that there wasn't a significant difference, and the values were statistically the same for the materials that went through the cycles This indicates that both, granite and AGS can be applied in environments subject to freezing temperatures of the infiltrated water within their pores, without going through a loss of mechanical resistance.

Abrasive wear. Table 2 presents the test results for the abrasive wear of the AGS and the granite.

Table 2. Wear thickness reduction of AGS and natural granite.

Wear thickness reduction (mm)		
	Running distance	
Stone	500 m	1,000 m
AGS	0.72 ± 0.02	$1,46 \pm 0.01$
Granite	0.53 ± 0.01	1.13 ± 0.02

According to Chiodi and Rodriguez,[26] the thickness reduction for a stone floor to be considered suitable for high-traffic should be no more than 1.5 mm, medium traffic should be no more than 3mm, and low traffic should be no more than 6 mm.

For the granite, the thickness reduction in the abrasive wear test in 1,000 m track was 1.13 mm, so it could be applied to a high traffic zone and for the AGS the reduction of thickness was 1.46 mm, which represents that it is a resistant-to-wearing stone; that can also be applied to high traffic flooring areas. It is noted that the natural rock suffered less wear when compared to artificial. This can be explained because the wear test evaluates the pullout of the material when subjected to friction; therefore the greater wear of the AGS can be explained by the easier removal of granite particles from the epoxy matrix.[29]

Carvalho et al.,[30] when testing an artificial stone composed of 80% residue obtained from an electromagnetic precipitator of a sintering process and 20% epoxy resin, it presented a thickness reduction of 1.04 mm and 2.16 mm for 500 and 1,000 m, thus considered suitable for a medium traffic flooring.

However, according to the norm ABNT NBR 15844[27] that classifies granite according to its properties, in a 1,000 m path the thickness reduction should not go beyond 1,0 mm. In terms of this specific norm, the AGS would not fit the criteria, and could present performance problems in a long term basis. This fact limits its applications and more caution should be used when applying it as paving for certain environments.

Hard body impact resistance. The hard body impact resistance test was used to evaluate how much energy the stone is capable of withstand depending on the maximum height of a falling object on its surface, and also to evaluate its level of cohesion.

On this test both the granite and the AGS presented the same results, with rupture energy of 4J and average height of rupture between the three test bodies was of 0.4 m. However, it is important to note that the materials tested did not have the same dimensions, and the natural granite had approximately double the thickness of the AGS. This shows that even with a lower thickness the artificial stone produced has the same resistance of the natural stone, indicating that it is a more tenacious material.

Frazão and Farjallat[31] established that with granite, the average rupture height must be equal or higher than 0.4m to display good resistance to instantaneous tension. According the ABNT NBR 15844[27] the minimum height must be 0.3m, this makes it possible to see that according both standard the materials are suitable for applications in environments subject to this type of load, such as commercial lots and airports. Further investigations upon the used of this granite residue for additive manufacturing[32] and high performance resins[33] can diversify the use of this waste into different polymer composite products as a possible material substitution strategy.

Conclusion

This research investigated the manufacturing of an artificial stone made of granite residue agglutinated in epoxy resin and its physical and mechanical properties. From the results obtained the following conclusions can be presented:

- The artificial stone produced presented better properties in comparison to natural granite;
- Density of 2.24 g/cm^3, water absorption of 0.19%, and apparent porosity of 0.42% for the AGS are all values within the numbers expected taking in consideration the Brazilian standards;
- The AGS appeared to have high mechanical resistance, with flexural strength of 32 MPa. The microscopic images obtained through SEM make it possible to see the good adherence between the particles and the epoxy matrix, and it point to a low void incidence, which explains the good physical and mechanical properties of the material;
- As a conclusion, the AGS produced can be applied in cold environments, because the water frozen within its pores did not influence on its mechanical performance.
- Through the abrasive wear test, it is seen that the thickness reductions of the artificial stone was low, which indicates that it can be used in flooring for high-traffic areas;
- The artificial stone produced was resistant to instantaneous tensions, and capable of absorbing energy, which allows its use in commercial areas;
- Taking in consideration the satisfying results obtained, it can be inferred that the methodology used to produce the artificial stone was efficient, creating a homogeneous mixture, with low porosity and void incidence, and a well-compacted material, which could be used in civil projects and construction.

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