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# DEVELOPMENT AND MECHANICAL CHARACTERIZATION THE OF PARTICULATE POLYMER COMPOSITE USING REJECTS OF LIMESTONE AND POLYESTER

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***Abstract.*** *Due to recurring environmental impacts, the current engineering works in search of solutions that minimize the environmental degradation, for example, using industrial residue to develop new materials. In the west region of Rio Grande do Norte, there is the accumulation of solid waste generated by quarrying. In this way, seeking to minimize this environmental degradation, this work aims to propose the utilization of these industrial residues in a particulate polymer composite. For this, it was developed two materials using limestone, as mineral filler and polyester as matrix, the only difference between the materials is percentage limestone, being used the percentages of 10% and 30%. The limestone powder was used in the granulometry between # 100 and # 200 and was analyzed by X-ray fluorescence. To analyze the effect of the mineral filler on the material, the material constituted for 100% of polyester was made. The mechanical properties were analyzed by the uniaxial tensile test, based on the standard ASTM D638. It is noteworthy that by increasing the percentage of limestone powder, improves the rigidity and the decreasing in the tensile strength. Therefore, it is suggested that residue of limestone powder in quarries can be reused in the manufacture of new materials, reducing their quantity and avoiding damages to the environment, since it obtained advantageous results in the property of rigidity of the material.*

***Keywords:*** *Environment, residues, particulate polymer composites, limestone powder, mechanical properties.*

## 1. INTRODUCTION

The environmental concern is a recurring issue in the actuality and on this question arises the necessary of searching solutions that aiming that aim to minimization environmental degradation. An example to minimize environmental degradation is the utilization of industrial residues in the development of new materials. In the west region of Rio Grande do Norte, there is a problem with solid residues generated by quarrying that cause environmental degradation, since the industrialization of the ore of rough stone produces the stone powder that is not used in other processes and ends up accumulating in all surrounding area. Therefore, it is necessary to find means to reuse these industrial residues, in order to minimize these problems.

In this context, it highlights the composites, which are materials consisted of the macroscopic combination of two or more different materials. In the what it seeks with its development, combine desirable characteristics of two or more different materials, generating a new material with optimized characteristics of interest and singulars (Casaril, 2009). The composites are formed by two constituents, the first constituent is called the matrix. The matrix connects, gives the shape and protects an arrangement of a more resistant material, denominated as reinforcement (second constituent) (Galli, 2016).

For their manufacture, different kinds of materials for reinforcing may be used, in the form of fibers or particles. For the use of fibers there are, for example, natural fibers or synthetic fibers, in the use of residues or particles there is, for example, the use of stone powder.

Thus, studies are conducted with diversified reinforcements such as: unidirectional carbon fibers (Galli, 2016), green coconut fiber (Ishizaki, *et al*, 2006), synthetics fiber combined partially with natural fibers (Silva, Freire Júnior and Aquino, 2004), carauá fiber (Mothé and Araújo, 2004), natural vegetables (Marinelli, *et al*, 2008), sugarcane bagasse (Sanchez, *et al*, 2010), naturals with residue from the timber industry (Correa, *et al*, 2003), sisal fiber (Gomes, *et al*, 2014), banana tree with synthetic resin (Iozzi, *et al*, 2010), waste from joinery (Mota and Mendes, 2010), nanomodified carbons (Peixoto, *et al*, 2010).

In view of this, this work aims to develop a new material that uses stone powder (limestone), which are accumulated as residues, to minimize the environmental problems generated by its accumulation, were used as reinforcement in particulate polymer composites. X-ray fluorescence spectrometer and uniaxial tensile tests are performed to characterize the constituents and the mechanical properties of the material.

## 2. EXPERIMENTAL PROCEDURE

For the accomplishment of this work was developed a new material with the percentages of 10% (C_10) and 30% (C_30) of limestone (as reinforcement) and resin Orthophthalic Polyester (5061) as matrix. The particles of stone powder (limestone) were obtained in the quarry of the county of Caraúbas-RN. The limestone powder (Fig. 1) was used in the granulometry between # 100 and # 200 and was analyzed by the X-ray fluorescence test to obtain its composition, and thus understand better which causes in the final material.

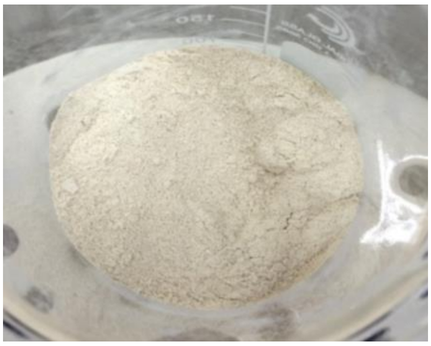


Figure 1. Limestone powder.

Initially manufactured the mold, using glass plates (as shown the Fig. 2) with the finality of fabrication of the proposed materials. The dimensions and amount of the plates are:

- 02 plaques of 40 cm X 02 cm, with 03 mm of thickness;
- 01 plaques of 38 cm X 02 cm, with 03 mm of thickness;
- 01 plaques of 40 cm X 40 cm, with 08 mm of thickness;
- 01 plaques of 35 cm X 40 cm, with 08 mm of thickness.

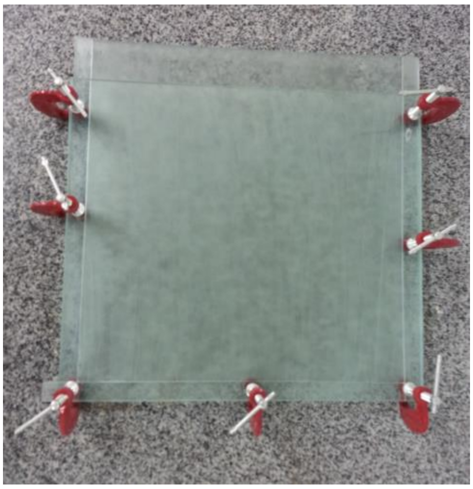


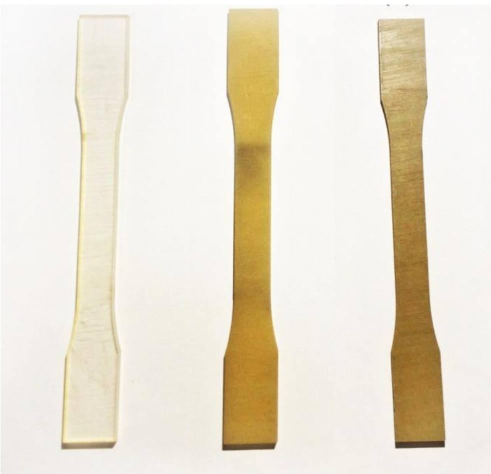
Figure 2. Mold with grampus.

In the manufacture of the proposed composite materials the following procedure was used: The resin was initially placed in a reservoir; the same, it was taken to the vacuum pump for 10 minutes to remove bubbles, and thereafter it was added 0.5% of the catalyst; then, the limestone was added, manually mixed for about two minutes and the air bubbles were again withdrawn from the vacuum pump; After this step, the material was dumped into the mold (Fig. 3) to cure for 24 hours; After curing, the material was demoulded (Fig. 4); Finally, with the obtained material, the laser cutting was done to obtain the dimensions established in standard (ASTM D 638, 2015) of the specimens, to achievement the uniaxial tensile test. It is noteworthy that were made specimens composed only with Orthophthalic Polyester Resin (5061).

Figure 3. Mold with the composite plaque.

Figure 4. Demoulded limestone plate.

The test was performed at the Mechanical Engineering Laboratory of the Federal Rural Semi-Arid University (UFERSA), with base in the standard on ASTM D638 (Standard Test Method for Tensile Properties of Plastics), in the which applied an increasing load of uniaxial tensile in the specimens until its rupture. Being this one of the tests most commonly used in the mechanical components industry due to provide important quantitative properties of the mechanical characteristics of the materials. It should be noted that, for the uniaxial tensile test, specimens were fabricated for the percentages of 10% (C_10) and 30% (C_30) limestone and to analyze the effect of the limestone powder on Orthophthalic Polyester Resin, purely polymeric specimens were also produced (Fig. 5).



(a) (b) (c)

Figure 5. Specimens: (a) Pure resin; (b) C_10; (c) C_30.

## 3. RESULTS AND DISCUSSION

Initially, accomplished quantitative analyzes in the limestone powder, to get the material composition. In next, accomplished the tests of uniaxial traction and with the results plotted the graphic stress x strain of each specimens, as well as the its modulus of elasticity (calculated for 30% of the tensile strength).

### 3.1 Chemical characterization of the limestone for Fluorescence of X-ray - FRX

With the results obtained by fluorescence analysis of X ray that determine the composition in the stone powder it was obtained that the limestone has the listed constituents in the Tab.1.

Table 1. X-ray fluorescence spectroscopy test.

| Constituents | Composition |
|---|---|
| **CaO** | 90.966% |
| **$SiO_2$** | 4.607% |
| **$Fe_2O_3$** | 1.911% |
| **$Al_2O_3$** | 1.718% |
| **$K_2O$** | 0.730% |
| **MnO** | 0.069% |

### 3.2 Mechanical performance in the uniaxial tensile test for the specimens of pure resin

In the mechanical characterization of the developmented materials, it was evaluated initially, as comparative parameter, the mechanical development to traction for the specimens constituted only of resin Poliéster Ortoftálica 5061, in the Fig. 6 is showed the graphic stress x strain for this material.

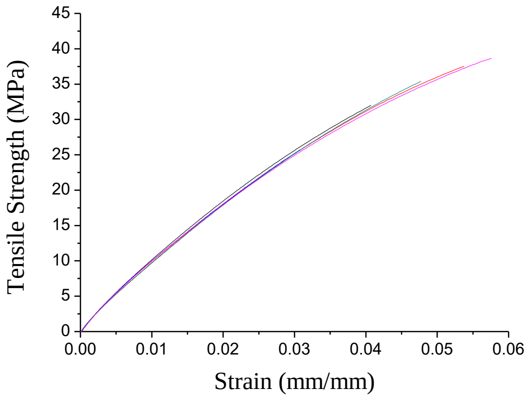



Figure 6. Graphic Stress x Strain for the specimens of pure resin.

The medium values and standard deviation of maximum stress, normal deformation, and modulus of elasticity of the material are shown in the Tab.2.

Table 2. Mechanical properties - The resin of Ortho-terephthalic 5061.

| MECHANICAL PROPERTIES | AVERAGE | STANDARD DEVIATION |
|---|---|---|
| **Tensile Strength (MPa)** | 33.90 | ±5.23 |
| **Strain (mm/mm)** | 0.05 | ±0.01 |
| **Elastic Modulus (GPa)** | 1.13 | ±0.04 |

According to results obtained in the Fig. 6 and in the Tab. 2, realizes that the strain practically did not have standard deviation, already that its value was of 0.01 mm/mm, while that the resistance and the modulus of elasticity shown values with the standard deviation of 5.23 MPa and 0.04 GPa, respectively. Probably, this standard deviation in the resistance happened due to bubbles/imperfections of the specimens, originating of the manufacturing process, that end up acting as stress concentrators, making the material more fragile. While the modulus of elasticity, for being a characteristic intrinsic of the material, did not suffer influence of the shaped bubbles.

The value obtained for the tensile strength is according with the literature, already that Crawford (1998) quotes that the tensile strength of the polyester resin is between the values of 21 MPa to 45 MPa.

### 3.3 Mechanical performance in the uniaxial traction for the specimens of limestone

For the specimens with 10% of limestone in its composition was obtained the graphic, illustrated in the Fig. 7, with the values of Stress x Strain of the material and the Tab. 3 shows its values and standard deviations.

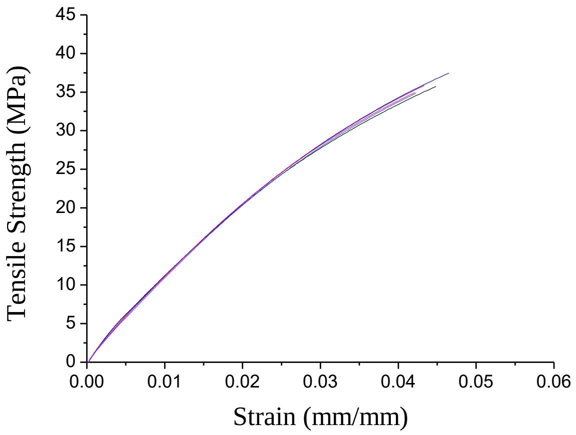



Figure 7. Graphic Stress x Strain for the specimens of 10% of limestone.

Table 3. Mechanical properties - 10% limestone.

| MECHANICAL PROPERTIES | AVERAGE | STANDARD DEVIATION |
|---|---|---|
| **Tensile Strength (MPa)** | 36.01 | ±0.88 |
| **Strain (mm/mm)** | 0.04 | $\pm 1.51 \times 10^{-3}$ |
| **Elastic Modulus (GPa)** | 1.11 | ±0.01 |

With these values, realizes that both the modulus of elasticity, resistance and the strain did not show large standard deviations, showing that the particles filled of uniform shape the spaces in the material. In comparison with the specimens of pure resin, probably the insertion of particles did with that the amount of bubbles decreased, already that some empty spaces were completed for these particles.

For specimens with 30% of limestone in its composition was obtained the graphic, illustrated in the Fig. 8, with the values of stress x strain of the material and the Tab. 4 shows its values and standard deviations.

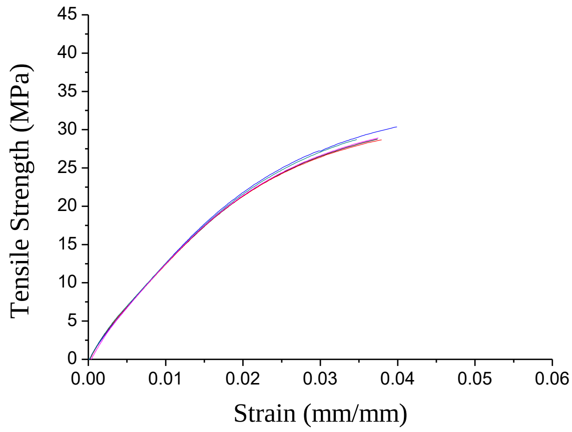



Figure 8. Graphic stress x strain for the composite with 30% of limestone.

Table 4. Mechanical properties - 30% limestone.

| MECHANICAL PROPERTIES | AVERAGE | STANDARD DEVIATION |
|---|---|---|
| **Tensile Strength (MPa)** | 29.09 | ±0.71 |
| **Strain (mm/mm)** | 0.04 | $\pm 1.85 \times 10^{-3}$ |
| **Elastic Modulus (GPa)** | 1.51 | ±0.15 |

By the Fig. 8 and Tab. 4, can be observed a lost accentuated of the linearity in the final of the shape of the curve stress x strain and yet a value larger of modulus of elasticity, in comparison with the limestone 10%. The increase for powder did with that the modulus increased, making thus the material harder.

### 3.4 Influence of the limestone percentage

Comparing the influence of the percentage of stone powder in the composition of the materials, in the fractions of 10% (C_10) and 30% (C_30) of limestone, we have the Fig. 9 that shows the graphic of the average curves of the pure resin, for this analyze, being able thus accomplish a comparative of the mechanical behavior of these materials.

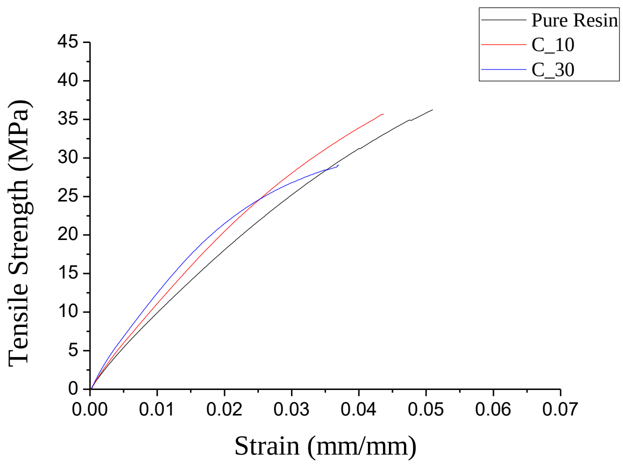
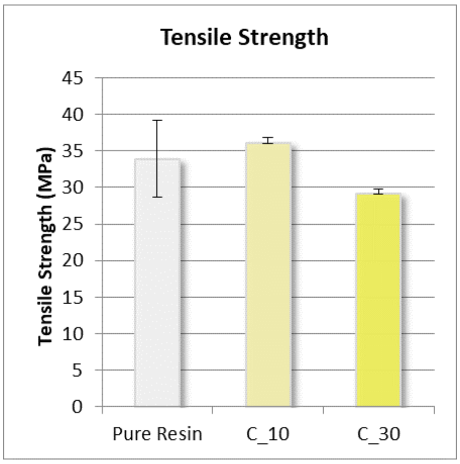


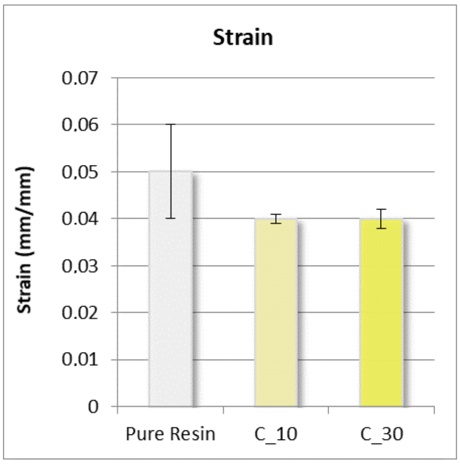

Figure 9. Graphic stress x strain: Pure Resin, C_10 and C_30.

By the graphic of the Fig. 9, can be observed that the material with 10% of limestone presents, in the average, a slight superiority in the that says respect to strength and a small inferiority in the strain in relation to pure resin. However, according with the standard deviations of the tables 2 and 3 can not claim that this is true, the graphic in bars (Fig. 10) shows that both presents breaks in common.

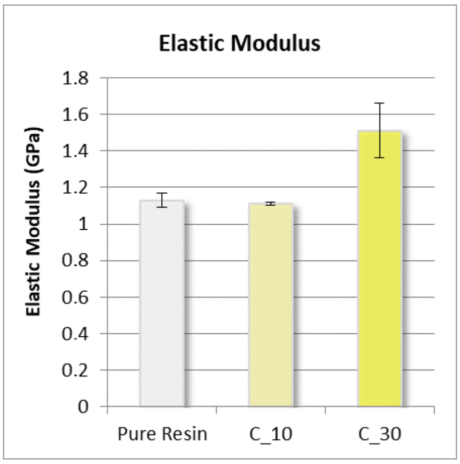
Still realizes that the measure that increased the percentage of the limestone for 30%, happened an improvement in the modulus and a lost in the strength. In a quantitative evaluation have an addition of 33.63% and a lost of 14.19% of the material with 30% of limestone in relation to pure resin, respectively. An interesting result is that for 30% of limestone presents a shape more curved that when compared with the pure resin and 10% of limestone. The Fig. 10 shows the average values comparatives of the three analyzed materials, for each property separately, in form of graphic of bars.




(a)




(b)




(c)

Figure 10. Comparative between the specimens of pure resin, C_10 and C_30: (a) Tensile Strength; (b) Strain; e (c) Elastic Modulus.

## 4. CONCLUSIONS

Thus, it is concluded that the increase in the quantity of the stone powder caused the elastic modulus increased, making thus, the material more rigid, and intensified changes in the behavior on stress-strain diagram as a marked loss the of linearity at the end of the curve.

The material with 10% of limestone presented, on the average, a little superiority with respect to strength and a little inferiority in the deformation relative to the specimen that was made with pure resin. However, according to their standard deviations it can not be said that this is true, since they have intervals in common.

When the percentage of limestone was increased to 30%, there was an improvement in stiffness and loss in the strength. In a quantitative evaluation has an increase of 33.63% in the Stiffness and a loss of 14.19% in the strength, of the material with 30% of limestone in relation to the pure resin, respectively.

Therefore, it is suggested that the waste of stone powder accumulated without use in quarries can be reused in the manufacture of new materials, reducing their quantity and avoiding damages to the environment, since they are advantageous in the mechanical point of view, like adjustment in the properties of the elastic modulus and strain and thus used in different applications.

## 5. ACKNOWLEDGEMENTS

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