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OBTAINING AND STUDY A COMPOSITE WITH POLYESTER RESIN AND LOAD OF CHICKEN EGGSHELL

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Abstract. *This work presents the study and characterization of a composite material obtained from the mixture of polymer matrix of polyester resin and residues of chicken eggshell. The collected shells were dried and broken to obtain three granulometries (fine, medium and coarse). The matrices used were the orthophthalic and terephthalic polyester resins and the percentage of residue was of 100% in mass in relation to the matrix. The test samples were constructed and subjected to tests to characterize them through their thermal, mechanical and physical properties. As expected, the proposed composite presented lower mechanical resistance than the matrix, characterizing the residue as filling load. In this way, all the formulations of the composite were applicable in structures that do not require resistance to great efforts. The apparent density of the composites did not show significant variation in relation to the resin. The higher quality of the composite studied was the reduction of the amount of resin to be used since the residue dust load can reach 100% in relation to the matrix reducing the cost of the material obtained for the manufacture of diverse structures.*

Keywords: *composite materials, residues, eggshells, characterization.*

1. INTRODUCTION

The search to take advantage of the maximum that the raw material can offer has deserved prominence in recent scientific researches to obtain materials with desired mechanical, physical and thermal properties. For this purpose, composites have been used in large scale, mainly those that have residues in their composition. This strand is due to the search to minimize the harmful effects of exposure of these residues in nature (MOTA, 2016).

Still according to Mota (2016), the ecological appeal, the environmental degradation by the deposition of waste materials and immense variety of tailings led to the change in the definition of composite. Before it considered only the mixture of materials that provided greater mechanical resistance and, today, the loads are included as composite forming elements. It should also be pointed out that the removal of these materials from the environment minimizes the harmful effects caused to the planet's sustainability.

The industrialization of eggs (powdered egg, liquid, frozen, etc.) provides economic advantages, extension of product life, transport and conservation facilities, but it generates an expressive number of peels and is considered waste. Knowing that the bark represents 10% of the egg weight, waste generated corresponds to around 5.92 million tons per year worldwide (OLIVEIRA, BENELLI, AMANTE, 2009).

The eggshell is a rich source of minerals, serving as the basis for the development of products in the cosmetics industry, food supplements, bioceramic bases, fertilizers, bone and dental implants and as an anti-tartar agent in toothpastes (MURAKAMI, 2006).

The composite object of study of this work was obtained from the mixture of orthophthalic polyester resin matrix and eggshell residues, for three granulometries. The percent loading of the residue corresponding to 100% by mass chosen, depending on the tests for the diagnosis of the saturation of the mixture.

The characterization of the composite was carried out obeying the pertinent norms and, among others, the thermal properties, the tensile and flexural and impact strength, density, moisture absorption (distilled water and seawater) and aging test were evaluated.

2. MATERIALS AND METHODSTEXT FORMAT

This section summarizes the materials, manufacturing processes and methodology used for determining the mechanical, physical and chemical properties of the composite obtained.

2.1 Materials

The resins and catalyst used in the manufacture of the proposed composites were purchased directly from a Redelease distributor in São Paulo / SP, at a cost of R \$ 25.00 / kg (orthophthalic) and R \$ 21.00 / kg (terephthalic). The chicken eggshells used as cargo in the composite were donated by a Parnamirim / RN bakery.

The choice of resins was based on their low cost combined with easy manipulation of the material. The manufacture of composites with polyester resins does not require the availability of large equipment, working at low temperatures and with a lower risk of accidents at work.

Butanox M50 manufactured by Redelease was the curing agent used with a percentage of 2% by mass in relation to the matrix. The release agent used was the carnauba wax to obtain a better finished surface besides facilitating the removal of the specimens from the mold.

The sample used in the characterization tests were fabricated from plates made into molds conforming to the dimensions specified by the ASTM standards of each test. In the samples fabrication for tensile, flexural and impact tests, cast iron molds with dimensions complying with the relevant standards were used.

For the realization of the experiments were used other materials such as mixers, Baker, digital scale, pliers, spatula and other tools. Distilled water, used in the water absorption test, was produced in a solar distiller manufactured in LMHES / UFRN. The seawater for the same test was collected at the beach of Ponta Negra, Natal / RN. Low linear density polyethylene (LLDP) containers were reused to mix and manipulate materials for the manufacture of composites

2.2 Methods

In order to obtain and process the egg shell load and subsequent manufacture of the composite, procedures described in the flow chart of Figure 1 were adopted.

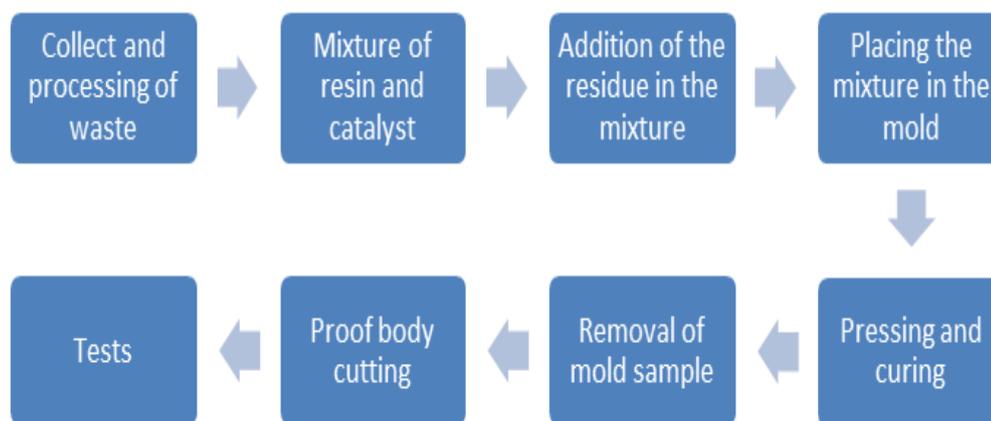


Figure 1. Flow chart of the stages of the samples manufacturing process of the proposed composite.

After collect, the eggshells were put to solar drying for one day, and collected overnight, in a dryer made from scrap manufactured by the LMHES of UFRN, object of Silva's master dissertation (2013).

The grinding of the material was done manually to obtain the coarse (G3) and medium (G2) granulometry, while a domestic blender was used to obtain powder (G1). In this way, specimens fabricated for the three granulometries shown in Figure 2.



Figure 2. Different granulometries of the eggshell residue - G1 (0.5-0.6 mm), G2 (1.2-1.5 mm) and G3 (2-3.3 mm).

Before starting the process of manufacturing the samples, the formulations to be studied were defined. Through saturation tests, the percentage of 100% by mass of residue was chosen in relation to the matrices used. In this way, the composites manufactured varied granulometries (G1, G2 and G3) and matrices (orthophthalic and terephthalic polyester resin).

The process used to manufacture the samples used was molding and cold pressing. The release agent was applied to the mold to ensure uniformity in the coating, in order to facilitate the removal of the plates, obtaining a good finish.

Regardless of the mold to be used, the process for making the composite was the same. After application of the release agent, weighings of the resin, catalyst and residue quantities were carried out on a precision scale.

According to the manufacturer's recommendation, it was necessary to use 2% by mass of catalyst relative to the amount of resin. After addition of the catalyst, the residue was added graded to complete the complete mixing between the matrix and the filler.

Then the resin and residue mixture was placed in the closed mold and taken to the Marcon hydraulic press (maximum capacity of up to 10 tons), where a load of 2 tons was applied until the total solidification of the material, which lasts 12 hours. The pressing process aimed at ensuring the homogeneous distribution of the blend within and reducing the presence of bubbles and other defects within the plate.

After waiting the curing time, the plates were removed from the mold carefully so as not to cause damage to the sample. The composite manufacturing process was completed by cutting the plates using a bench disk saw to obtain the samples to be tested.

2.2.1 Survey of physical properties

To obtain the mechanical properties of the composite, tensile, flexural and impact tests were performed. The tensile and flexure tests at three mechanical points were performed using a Shimadzu AG-X machine, with a capacity of 300 KN. Five samples were used for each formulation where the tensile test follows ASTM-638-10 and three-point flexing, ASTM D-790.

For the Charpy Impact test, an analog pendulum of the VEB WERSTOFFMASCHINEN LEIPZIG machine was used, which indicated the energy value absorbed by the ASTM E 23 material using five samples of dimensions indicated by it.

2.2.2 Thermal tests

For the determination of thermophysical properties were used the KD2 Pro properties analyzer manufactured by Decagon Devices. Thermal conductivity, volumetric thermal capacity, thermal resistance and diffusivity of the matrices and composite formulations were obtained.

2.2.3 Apparently density

The digital densimeter DSL 910 (with a repeatability of $\pm 0.003\text{g} / \text{cm}^3$) of the Laboratory of Fluid Mechanics - LMF / UFRN was used to obtain the density of the formulations of the proposed composite and of the matrices.

2.2.4 Water absorption

Samples and procedures for the water absorption test followed ASTM 570-98. Three samples of dimensions 25 mm x 25 mm x 8 mm were submitted to each formulation were immersed in two different liquids (distilled water and seawater).

As most of the materials obtained from polyester resin are finished, it has become relevant to apply a resin film to the samples simulating such finishes. These samples obtained from the composite plates for tensile and flexure tests. They were weighed before immersion, after 24 hours and weekly for 60 days until stabilization.

2.2.5 Aging test

Overtime the materials can suffer degradation and cause the compromise of their properties. Degradation can be caused by chemical, physical or mechanical agents or simultaneously. The modifications caused to the material may be irreversible, evidenced by the progressive deterioration of its properties, including the visual aspect. Thus, the aging test should be performed with the purpose of analyzing the influence of the natural conditions of the medium under the composite studied.

Eight samples (one of each formulation and matrix resins) were exposed to natural conditions of aging in the Nucleus of Industrial Technology of UFRN during one year. Each sample was weighed and photographed before and after exposure, in order to compare the change in appearance. The dimensions of the specimens were the same as those used in the tensile and flexure test.

2.2.6 Applications of the proposed composite

Considering the possibility of applications in ornamental pieces, the composite material of orthophthalic polyester resin and thick granulometry (G3) was used in the manufacture of a bathroom sink bench and a bench top in order to test the performance of the material. Figure 3 shows composite applications in the manufacturing phase.



Figure 3. Bench top and bench top made of the proposed composite material.

3. RESULTS AND DISCUSSIONS

The results of the tests used for the characterization of the composite proposed and studied from the terephthalic and orthophthalic polyester matrices and the residues obtained from different granulometry of the eggshell are presented.

3.1 Characterization

3.1.1 Tensile and flexure resistance

The Tab. 1 presents the comparative results between the measurements in the test and the parameters assigned by the manufacturer of the matrices used to obtain the composite.

Table 1. Comparative result of the tensile and flexural resistance measured and supplied by the manufacturer.

| Samples | TR measured | Manufacturer's TR | FR measured | Manufacturer's FR |
|---------------------|-------------|-------------------|-------------|-------------------|
| Orthophthalic Resin | 40.0 | 55.84 | 66.63 | 82.74 |
| Terephthalic Resin | 39.62 | 62.05 | 55.71 | 84.12 |

The tensile resistance (TR) measured were lower than those presented by the manufacturer and showed a reduction of 28.37% for orthophthalic resin and 36.15% for terephthalic resin. This difference may be associated to the problems in the sample manufacturing process, as well as the loss of properties due to the change in the environmental conditions of resin production until its use.

The flexural resistance (FR) showed a decrease of 19.5% when compared to RF of the orthophthalic resin provided by the manufacturer. While the terephthalic resin presented FR 33.8% lower than the one indicated by the manufacturer. As in the tensile test, these results can be justified by the alteration of resin properties as a function of transport, changing environmental conditions and problems in the sample manufacturing process.

For the characterization of the composite, samples were submitted to the tensile test. Tab.2 presents the results during the test and the behaviors obtained.

Table 2. Results of the tensile and flexure resistance test.

| Formulation (orthophthalic) | TR (MPa) | FR (MPa) | Formulation (terephthalic) | TR (MPa) | FR (MPa) |
|-----------------------------|----------|----------|----------------------------|----------|----------|
| Resin | 40.0 | 66.63 | Resin | 39.62 | 55.71 |
| G1 | 37.33 | 14.82 | G1 | 37.41 | 17.47 |
| G2 | 28.51 | 14.46 | G2 | 31.00 | 17.12 |
| G3 | 26.93 | 12.57 | G3 | 26.38 | 13.99 |

The results presented in Tab. 2 showed that as the particle size was increased there was a decrease in the mechanical resistance of the composite.

With the results of the test it can be verified that addition of the residue weakened the material, however the smaller grain size showed a behavior closer to that of the matrices. This behavior can be explained by the fact that the smaller particle size facilitates the homogeneity of the mixture, not containing large discontinuities that weaken the material.

The composite proposed in its three granulometries showed a lower mechanical tensile behavior than the matrixes (orthophthalic and terephthalic polyester resin), which indicates the fragility of the material, characterizing the residue as a filler due to its addition to decrease the tensile resistance.

As in TR, there was loss of flexural strength as the grain increased, due to the fact that the discontinuities generated by poor compaction of the grains weaken the material.

These results did not impair the composite in the proposed configurations however this material should not be used in applications with high load requests.

In relation to the formulations with higher mechanical strength, the smaller particle size composites showed better performance. The test also pointed out a difference between the matrixes where the terephthalic resin obtained in average FR performance 16% better than the orthophthalic resin and in the TR this difference drops to 14%.

3.1.2 Charpy Impact test

Tab. 3 presents the results of the energy absorbed in the Charpy impact test carried out with six samples of each formulation and matrix proposed.

Table 3. Results of energy absorbed in Charpy Impact.

| Formulation (orthophthalic) | Energy absorbed (J/cm ²) | Formulation (terephthalic) | Energy absorbed (J/cm ²) |
|-----------------------------|--------------------------------------|----------------------------|--------------------------------------|
| Resin | 0.26 | Resin | 0.3 |
| G1 | 0.22 | G1 | 0.3 |
| G2 | 0.12 | G2 | 0.31 |
| G3 | 0.1 | G3 | 0.34 |

The energy absorption capacity decreases as the grain size increases in the orthophthalic resin. However, the increase of the energy absorbed in the samples of terephthalic resin matrix can be considered irrelevant because the change of granulometry causes increase only in the second decimal place.

3.1.3 Thermal test

Tab. 4 shows the average results of the thermal analysis properties of the matrixes and composite formulations studied.

Table 4. Mean results of the thermal test.

| SAMPLES | C (MJ/m ³ .K) | D (mm ² /s) | K (W/m.K) | °R (°C.cm/W) |
|----------------------------|--------------------------|------------------------|-----------|--------------|
| Orthophthalic Resin | 1.853 | 0.0975 | 0.1785 | 562.45 |
| G1 | 1.028 | 0.1925 | 0.289 | 351.3 |
| G2 | 1.6965 | 0.178 | 0.3015 | 337.85 |
| G3 | 1.707 | 0.209 | 0.36 | 287.55 |
| Terephthalic Resin | 1.357 | 0.1075 | 0.144 | 700.95 |
| G1 | 1.8385 | 0.185 | 0.3385 | 298.1 |
| G2 | 2.169 | 0.1745 | 0.338 | 301.35 |
| G3 | 2.391 | 0.194 | 0.42 | 242.05 |

The behavior of the thermal conductivity of the proposed composite was higher in relation to the matrices used. Thus, it can be concluded that the addition of the eggshell residue adds to the material a greater capacity to conduct heat because the calcium carbonate (greater component of the residue) has a higher thermal conductivity than the polyester resins used.

It was also noted that the thermal conductivity of the composite increased as the grain size increased. This probably happened because the larger grain did not mix homogeneously with the matrices. Thus, when the assay is performed, it is more likely that the sensor will come into closer contact with the residue (higher conductivity) than with the resins (lower conductivity) which increases the conductivity relative to the material. The material proposed in all formulations can't be considered a thermal insulation because it has thermal conductivity higher than 0.21 W/m.K, according to BERGMAN et al. (2014).

As the specific volumetric heat represents the ability of the material to retain heat, the tests show that the terephthalic resin matrix showed increased heat retention as the grain size increase and addition of the residue. However, the orthophthalic resin reduced the amount of heat retained with the addition of the residue. These different behaviors can be explained due to the difference in the polymerization of the resins.

Considering that thermal resistivity is the inverse of conductivity, it is expected that the composite exhibits a decrease in the resistance of the material to conduct heat and this behavior was obtained as seen in Table 3.

The behavior of the thermal diffusivity is influenced by the apparent density and specific heat, so the results obtained by the samples of matrices and composites proposed presents this.

3.1.4 Water absorption

The methods adopted for the absorption test of distilled water and sea water was established by ASTM D570-98. The samples saturated on average with 40 days of immersion. Tab. 5 shows the results obtained in the water absorption test.

Table 5. Average water absorption in the different immersion media.

| SAMPLES | ABS. DISTILLED WATER WITH FILM (%) | ABS. DISTILLED WATER WITHOUT FILM (%) | ABS. SEAWATER (%) |
|----------------------------|------------------------------------|---------------------------------------|-------------------|
| Orthophthalic Resin | 2.457 | 2.7 | 0 |
| G1 | 2.644 | 3.53 | 0.877 |
| G2 | 3.027 | 3.536 | 3.741 |
| G3 | 3.986 | 4.346 | 4.835 |
| Terephthalic Resin | 1.099 | 2.028 | 2.623 |
| G1 | 1.107 | 2.449 | 3.455 |
| G2 | 1.360 | 3.028 | 3.942 |
| G3 | 3.739 | 4.398 | 4.634 |

The absorption in the two media was higher for the composites in the three grades studied when compared to the resins. The composite exhibits this behavior because the addition of eggshell residue increases the porosity of the material when compared to the matrix used.

The percentage increase in moisture absorption can still be observed as the grain size of the residue increases. This behavior is due to the fact that the egg shell is more hydrophilic than the resin and the smaller the grain, the greater the ease the resin presents to seal it.

As expected, the application of the protective film had a positive effect since it decreased the moisture absorption of the samples when compared to those that did not have film and also were immersed in distilled water.

As obtained by Oliveira (2017), the applied film reduced the absorption of distilled water and showed an average reduction of 28.4% in relation to the samples without film.

The samples immersed in seawater presented similar behavior to those immersed in distilled water in which the moisture absorption increased as the grain size of the residue was increased.

All composite formulations showed higher moisture absorption than their matrix resins. When comparing the absorption in distilled water and sea water, it is noted that the samples absorbed more when immersed in salt water. This behavior can be explained by the fact that the salts present in this aqueous medium react more with the residue causing it to absorb more water.

Although there is an increase in the moisture absorption of the composite in relation to the matrix, it can be said that the proposed composite can be applied in aqueous media, considering that the moisture absorption was low and still lower than the results obtained by Oliveira (2015).

3.1.5 Apparently density

By measuring the apparent density of the proposed matrices and composites, the results presented in Tab. 6 were obtained.

Table 6. Densities of the matrices and formulations studied.

| Formulation (orthophthalic) | Apparently density (g/cm³) | Formulation (terephthalic) | Apparently density (g/cm³) |
|--|--|---------------------------------------|--|
| Resin | 1.306 | Resin | 1.279 |
| G1 | 1.772 | G1 | 1.704 |
| G2 | 1.716 | G2 | 1.653 |
| G3 | 1.716 | G3 | 1.597 |

In all formulations studied, there was an increase in the composite bulk density when compared to pure matrices. As the eggshell load presents a higher density than the matrices used, its addition in the composite confers gains in the density value.

Despite this, the density of the composite decreased as the grain size increased. Thus, the lower density composites were G3 (higher particle size). This behavior was obtained because the larger grain presented compaction difficulties when compared to G1. Thus, it is possible that in the samples tested there is a smaller amount of residue as the grain size of the residue increases.

The density can indicate applications of the composite, especially when it is tried to fabricate light and resistant structures. Thus, when comparing the two matrices, the terephthalic resin becomes more viable because of its lower density.

3.1.6 Aging test

Samples were exposed to the environment at the UFRN in Natal-RN, being subjected to climatic variations, with rains and variable solar radiation levels during nine months and ten days. A change of coloration in the samples was observed, more pronounced in the samples of orthophthalic resin, as shown in Image 4 and Tab. 7 shows the sample masses before and after the 9-month and 10-day exposure in Natal.

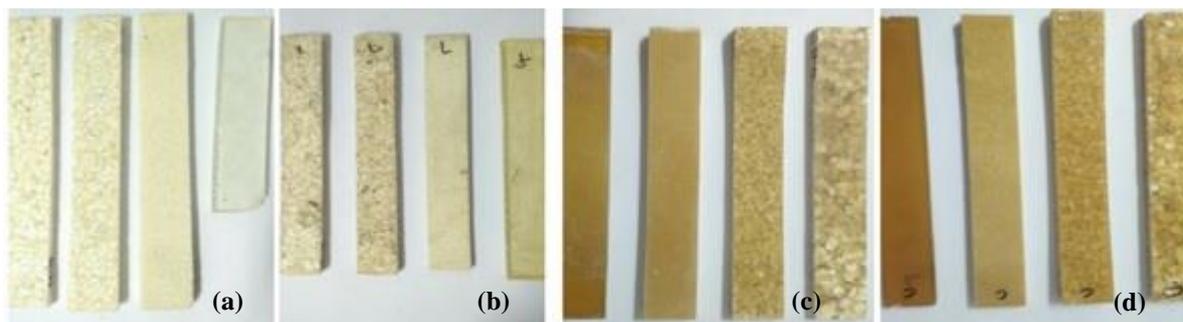


Figure 4. Samples of the composite and resin formulations tested in the aging assay – (a) orthophthalic resin before exposure (a) orthophthalic resin after exposure (c) terephthalic resin before exposure (d) terephthalic resin after exposure.

Table 7. Initial and final masses of samples exposed to the environment.

| Formulation (orthophthalic) | m_i (g) | m_f (g) | Mass loss (%) | Formulation (terephthalic) | m_i (g) | m_f (g) | Mass loss (%) |
|-----------------------------|-----------|-----------|---------------|----------------------------|-----------|-----------|---------------|
| Resin | 43 | 42.8 | 0.47 | Resin | 41.9 | 41.8 | 0.24 |
| G1 | 51.4 | 51.2 | 0.39 | G1 | 65.9 | 5.5 | 0.61 |
| G2 | 65.3 | 64.9 | 0.61 | G2 | 76.4 | 6.1 | 0.39 |
| G3 | 68 | 7.7 | 0.44 | G3 | 72.7 | 2.4 | 0.41 |

The loss of mass of the samples was insignificant, less than 1.0%, for all formulations tested over the exposure period for the two matrices studied. In this way, the proposed composites have shown to be viable for applications in which they can be exposed to the environment where the appearance is not a very important factor.

4. CONCLUSIONS AND SUGGESTIONS

4.1 Conclusions

1. The composite was feasible in the three granulometries and in the orthophthalic and terephthalic matrices studied;
2. The composite may be used to fabricate parts and structures applicable to systems subjected to low mechanical stress due to low tensile and flexural resistances;
3. The residue presented as filling load, due to the mechanical properties of the composite being inferior to the corresponding matrices;
4. The addition of egg shell reduced the amount of resin in the composite composition, minimizing the manufacturing cost;
5. All composite formulations exhibited moisture absorption higher than matrix resins, with increased absorption as the particle size of the eggshell residue increased;
6. Addition of egg shell residue caused increased moisture absorption of the material;
7. Terephthalic matrix composites showed higher tensile and flexural resistances than orthophthalic matrix composites. The formulation of the composite with the highest tensile and flexural resistance was the formulation of terephthalic resin with G1;
8. The capacity of the composites to absorb impact energy decreased as the particle size of the residue was increased, having significant loss only for orthophthalic resin;
9. The mass loss in the aging test was insignificant, but the color change after the test was higher in the composites with orthophthalic resin matrix;
10. The bulk density of the composite material increased with the addition of the egg shell residue;
11. The thermal behavior of the material fell, in relation to the matrices, with the use of the residue, since the conductivity of the residue is higher than the resins. The composite with the best thermal behavior was that of orthophthalic resin and G1, whereas the worst behavior was observed in the formulation with matrix of terephthalic resin and residue with greater granulometry (G3);

12. The best formulation was that of terephthalic resin and G1 because it had the highest mechanical properties and low water absorption.

4.2 Suggestions

1. Test the preparation of the composite with egg shell residue using other polymer matrices;
2. Expose samples of the composite to aging under environmental conditions for more than one year and characterize them before and after the end of the test;
3. Study the acoustic behavior of the proposed composite.

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