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EVALUATION OF THE PROPERTIES OF THE EPOXY RESIN BY ADDING LAYERED DOUBLE HIDROXIDES

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Abstract. *Composites have been used, more and more, in different areas of engineering. Therefore, the development of materials with distinct properties and behaviors for varied applications has to be done. In this paper, the dispersion of layered double hydroxides (LDH) will be evaluate in order to improve the epoxy properties with respect to flammability and strength. The new material was tested to verify if these properties were increased, for that, samples were made with LDH varying the percentage of weight of the LDH (0.2, 0.5 and 0.8%). In addition, to control the information, was made a group without LDH. The samples with LDH were made using a specific methodology, which will be describe in this paper. All samples were tested for flammability and strength. With the test results, graphics were made to compare the differences between the materials. In addition, a fractography analysis was performed to identify any possible problem that occurred at the fracture surface.*

Keywords: *Layered Double Hydroxides (LDH), Composites, Mechanical Properties, Nanocomposites, Flame Spread.*

1. INTRODUCTION

The most known composites are constituted by resin, as a matrix, and fiber glass, as structural reinforcement, and they have been each day more used due to their wide use and their properties. When compared with steel, for an example, polymer composites do not have many problems with weather and corrosion as steel (Nasseh, 2011).

Composites can be characterized using the mixtures rules, which says that the Young modulus and the rupture strength is a function of the volume fraction from the matrix and the reinforcement, as show the Eq. (1) and the Eq. (2) (Phulé, 2008).

$$E_c = V_m * E_m + V_f * E_f \quad (1)$$

Where:
E_c = composite modulus of elasticity
V_m = volume fraction of the matrix
E_m = matrix modulus of elasticity
V_f = volume fraction of the reinforcement
E_f = reinforcement modulus of elasticity

$$\sigma_c = \sigma_m * V_m + \sigma_f * V_f \quad (2)$$

σ_c = rupture strength of the composite
σ_m = rupture strength of the matrix
V_m = volume fraction of the matrix
σ_f = rupture strength of the reinforcement
V_f = volume fraction of the reinforcement

Trying to improve the properties of the composites, some researches dispersing particulates materials in the matrix are being performed. The purpose is improving the properties of the matrix, and with that, improve the properties of the composites. Silva (2010), as an example, related an increasing of the Young modulus in 25%, when 3%, in weight, of

Layered Double Hydroxides (LDH) is dispersed in the polystyrene. Botan (2014), related an improvement in 38% in the flexural strength, for the polystyrene, when 2%, in weight, of LDH is dispersed in the matrix. Suave (2008), related an improvement in 22% in the rupture strength, when 0,25 %, in weight, of Carbon Nanotubes (CNT) is dispersed in the epoxy resin.

The LDH are basically materials that are organized on layers composed by two cations, one of them divalent, and the other one, trivalent. Due the positive characteristic, an anionic specie is arranged in the interlamellar domain. The material is composed of two metal hydroxides that form an octahedral structure (Wypych; Satyanarayana, 2005).

The using of the LDH as particulate reinforcement are a differential. This group of material is capable to increase the mechanical properties of the matrix and increase their thermal properties. Nshutti et. Al. (2009) related that nanocomposites with LDH, are more thermally stable than those that had not LDH dispersed, when the samples were submitted a thermogravimetric analysis. This stability is due the loss temperature, in 10% and 50% in weight.

With the purpose to improve the thermal and mechanical behavior of the epoxy resin, this paper has an objective to add LDH in epoxy resin, varying the percentage in particulate material in 0,2%, 0,5% and 0,8%. After the dispersion, some tests were performed to identify improvements in the samples. Besides the tests, some fractography were performed to identify possible errors.

2. EXPERIMENTAL PROCEDURE

2.1 Preparation of the test specimens

The samples were built using a specifically methodology, which was developed by Suave (2008) and adapted for this case. The material utilized were: epoxy resin AR 260 of the brand Barracuda, acetone of the brand Química Fina, hardener AH - 150 of the brand Barracuda and Layered Double Hydroxides of Zinc and Chromium synthetized by Botan (2014) and assigned by State University of Campinas.

The equipment used were: Sonificator model USC – 2500 of the brand Unique, magnetic stirrer model HJ-4 of the brand Chang Bioscale and a scale model FA2204 of the brand Bioscale.

The methodology consists to disperse the particles in the acetone, using sonification, and then add the mixture to the epoxy resin. It is necessary follow the resin supplier recommended procedures to add the hardener to the blend, which was made before.

To investigate the influence of the LDH dispersed in the matrix, samples with 0,2%, 0,5% and 0,8%, in weight, was built. A reference group was built and was used as control of the results.

2.2 Tensile test

The tensile testing was performed using the ASTM D638-14 standard. The machine utilized was a universal model for tensile testing. The brand is the EMIC and the model is the DL 10000. The speed was 15 mm/min using a 10 ton load cell.

2.3 Flammability test

The flammability test was performed using the ASTM D635 standard, doing some modifications. The standard says basically, that the samples need stay in contact with a flame provide by a gas, during 30 seconds. If the flame in the sample do not comes out, it is necessary wait until the sample burn up to the sample have 25mm, counting the time of burning. Due the scarcity of samples, the minimum size were reduced to 15mm.

The standard says that the flame should stay in contact with the specimen at a 45 degrees angle. If not possible, the specimen need stay inclined at 45 degrees angle in contact with the flame, which was used in this paper.

2.4 Fractography test

To the fractography tests, were used a Sony camera, model hdr PJ10. The fractography purpose was identify any defect in the fracture area.

3. RESULTS AND DISCUSSION

3.1 Tensile test

The tensile tests results were used to compare the properties of each group of samples. The expected result is, how much more particulate material inside the matrix, higher is the rupture tensile value (Camargo, et. Al., 2009). The Fig. 1, shows the tensile tests results for all the samples.

From the Fig. 1, it is possible identify that the group with 0,8% of LDH, had the lowest result in the tensile test, 37,90 MPa, and has the greatest error value ± 20,39 MPa, with a confidence interval of 95%, wich shows some problems with the samples, due the great variation around the average. The samples with 0,2% of LDH had an inefficient dispersion as show the decrease in the tensile rupture average, comparing the results with the control group.

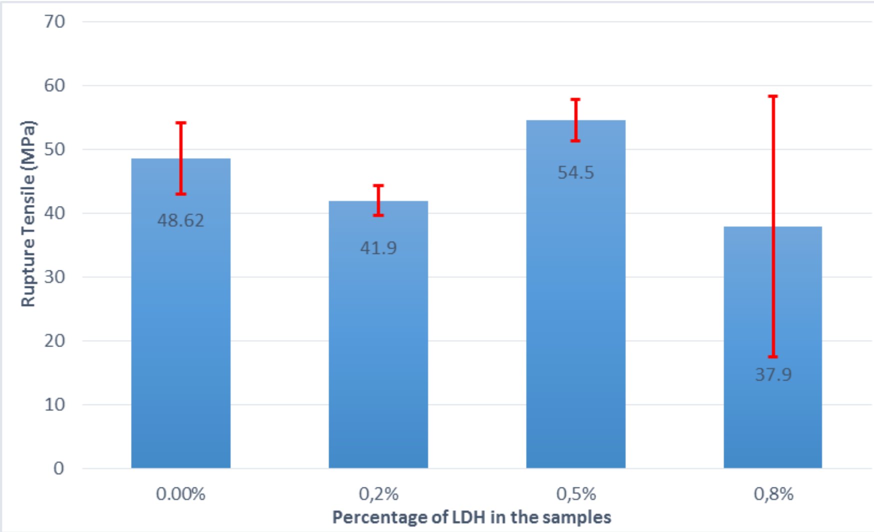

Figure 1- Rupture Tensile results for epoxy resin and nanocomposites.

This average is 41,90 MPa, with an error of ± 2,34 MPa. Song and Youn (2005) notice that, when nanoparticles are added in epoxy matrix the rupture value is increased, provided that an efficient dispersion has been achieved. Inefficient dispersion reduces the properties of the matrix. That mean that the dispersion in the sample with 0,2% of LDH were not efficient.

The improvement in the mechanical behavior of a nanocomposite is due to restriction movement by the polymeric chain. In this way, adding more particulate material, the expected is an improvement in the mechanical behavior until the solubility limit. If the solubility limit is reached, or the dispersion of the particles were not efficient, the values of tensile rupture will be reduced.

The samples with 0,5% of the LDH had the best results, 54,50 MPa, with an error of ±5,58 MPa, which shows that the dispersion was efficient, although the increase was only 11%, compared to the control group. Suave (2008), reported a 22% increase in the tension rupture when 0,25% of NTC is dispersed in the epoxy matrix. Silva (2010), related that the traction resistance, when 2% of LDH is dispersed in polystyrene, increases by 38%. The increase of 11%, related in this paper to the samples with 0,5% of LDH, is simple and due the way of the LDH were dispersed. Some problems with the delamination happened, which meant that the property was not as expected. Another reason could be the incomplete adhesion between the matrix and the reinforcement, as related by Song and Youn (2005).

3.2 Fractography analysis

The objective of these analysis was to identify crack propagation points, which may have reduced the average rupture tensile.

The Fig. 2 shows the fractography performed in the control group, identifying bubbles, inclusion of some mold pieces or defects generated by the mold, due the mold was built in silicon.

The images with red circles indicate defects found in the samples. After the analysis, in all the samples, a comparative will be realized between the results obtained when all the samples were evaluated and the average of the samples that did not present defects.

The Fig. 3, Fig.4 and Fig. 5 are the fractography of the samples with 0,2%, 0,5% and 0,8% of LDH, respectively. The defects are signalized with a red circle.

In the control group, or without LDH, the samples number 1 and 5 showed bubbles, and the sample number 4 showed a defect caused by the mold.

The group with 0,2% of LDH, the samples number 3 and 4 showed bubbles. The group with 0,5% LDH the same thing happened to the sample number 2. Although, to the group with 0,5% of LDH, the sample number 4 showed a defect caused by the mold.

The most affected group, due the characteristics of the mold, was the group with 0,8% of LDH. The sample number 2 showed defects due the mold. The sample number 3 had inclusion of mold material and the samples numbers 1 and 5 showed bubbles in the fracture surface.

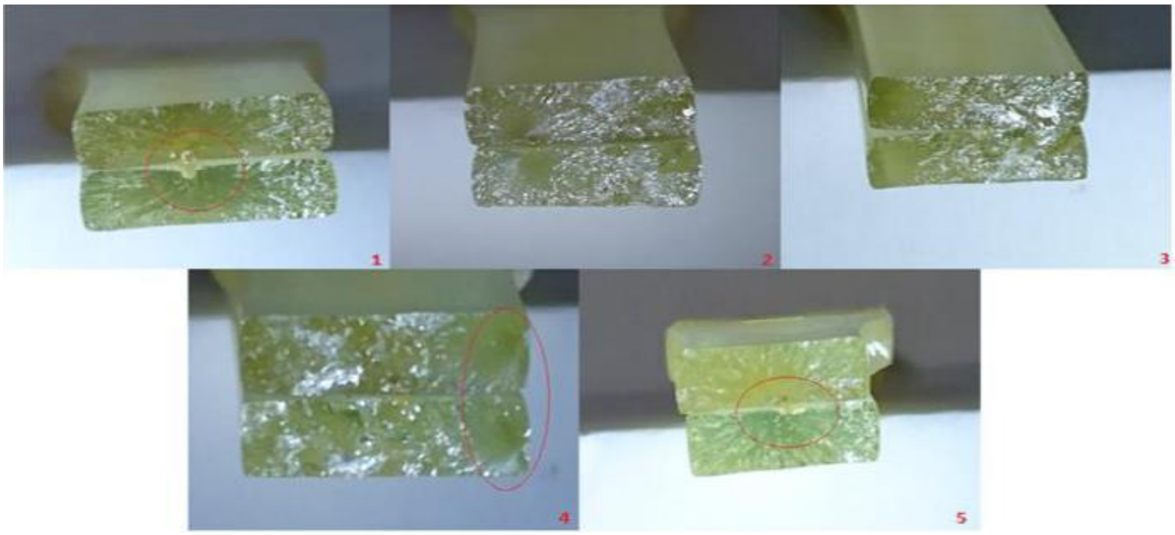

Figure 2 – Fractography of the control group, 0,0% of LDH.

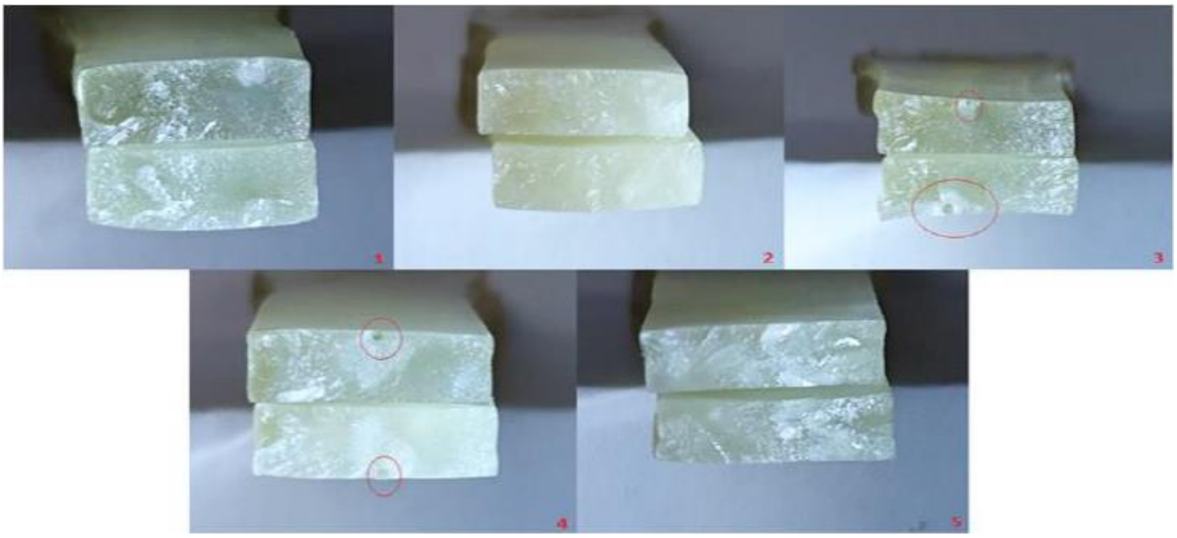

Figure 3 – Fractography of samples with 0,2% of LDH.

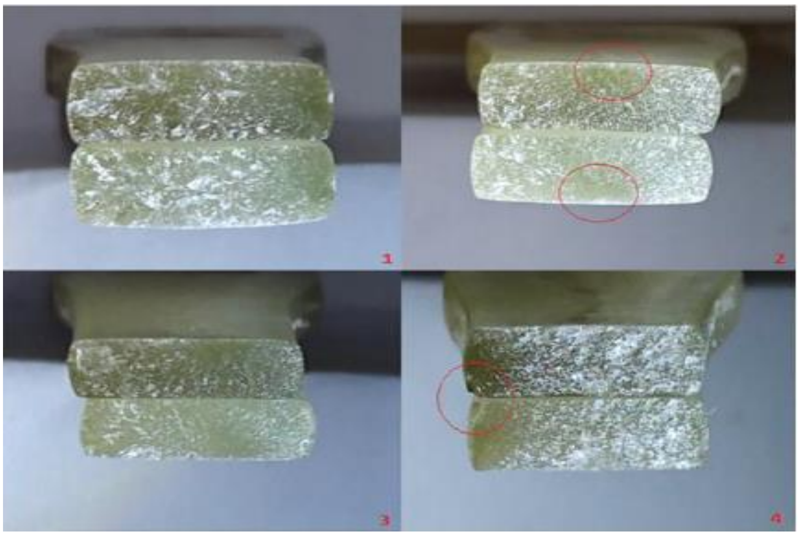

Figure 4 - Fractography of samples with 0,5% of LDH

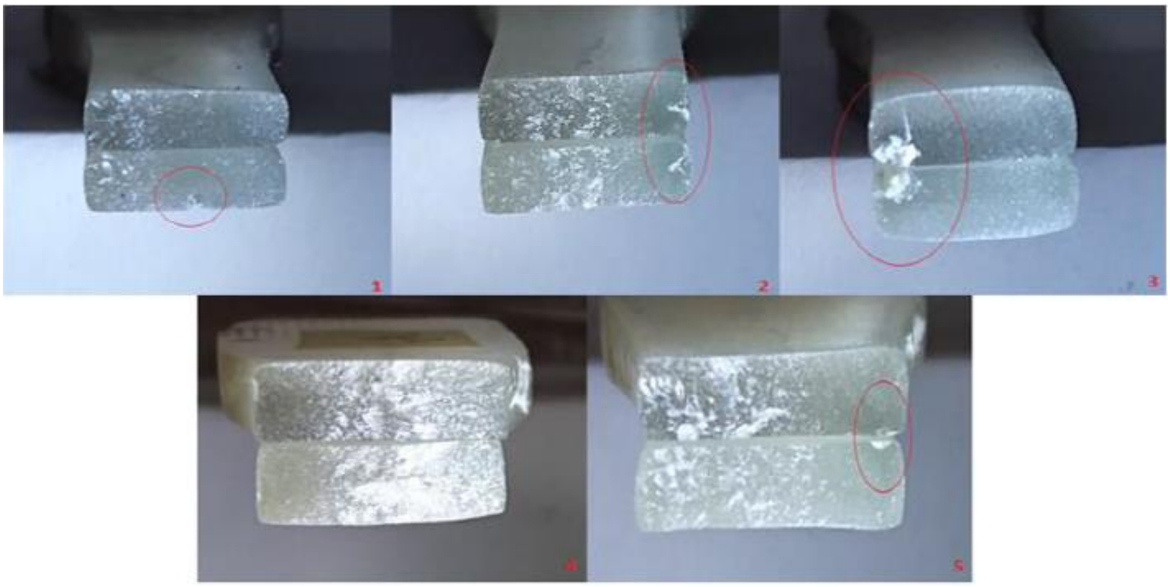

Figure 5 - Fractography of samples with 0,8% of LDH

The Fig. 6 shows the graphic comparing the results. The orange bars are the results of the samples that did not show defects, while the blue bars show the values to all the samples, with and without defects and bubbles. It is possible to see that the all the results had an improvement, when the samples with defects were discarded.

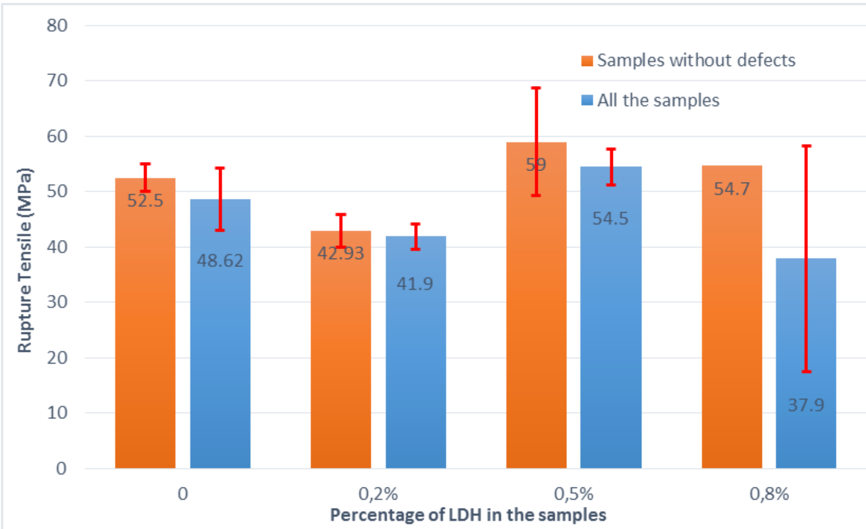

Figure 6 - Comparative of tensile rupture between all the samples and samples without defects.

The average value to the tensile rupture, in the control group, changed from 48,62 MPa, with ± 2,43 MPa of error, to 52,5 MPa, with ±5,57 MPa of error. The value to the tensile rupture, to the samples with 0,2% of LDH, changed to 42,93 MPa, with an error of ± 2,34 MPa. The new average for the group with 0,5% of LDH is 59 MPa, with an error of ± 9,73 MPa. The group with 0,8% of LDH, only one of all the samples showed no defects. The maximum value to this sample was 54,7 MPa. As it is only a sample there is no way to calculate the error.

3.3 Flammability analysis

The LDH can be used to reduce the spread flames, as reported by Botan (2014). To identify that behavior, a flammability test was performed in all the samples. In the Fig. 7 is showed a graphic with the results, for all the samples with a confidence interval of 95%. The results are presented in the same pattern, burned volume/ time (mm^3/s).

Is possible to see that the reduce of the spread flames was not so big as expected, and the samples with 0,8% of LDH had the best results, when is compared with other groups. In addition, was possible to identify that how much more LDH in the samples, more efficient is the reduction of the spread flames.

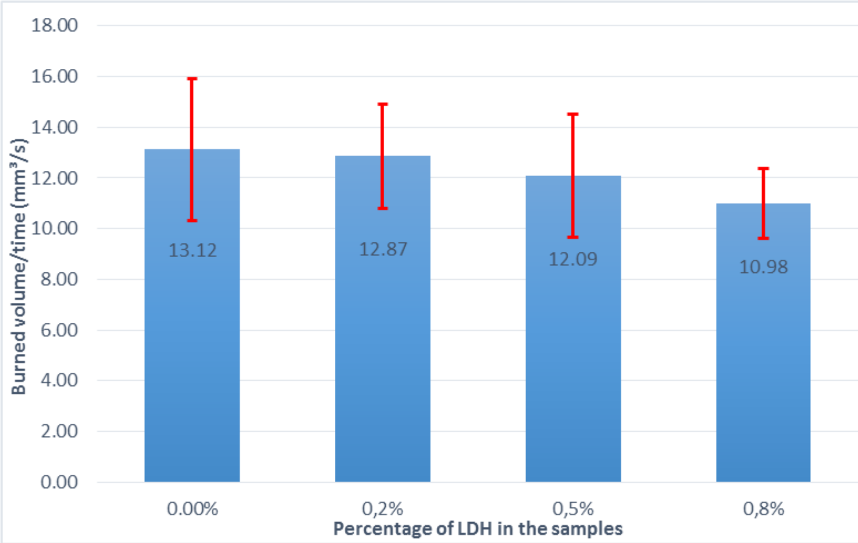

Figure 7 – Flammability analysis.

The delay, in the flame spread, can be explained by two theories. The first one is that the LDH prevents the movement of the polymer chains, as related by Camargo (2009). In addition, the second one is the LDH transfers their thermal stability to the matrix, what explain why the samples with more LDH had a better behavior in the flame retardancy (Botan, 2014).

4. CONCLUSION

The dispersion of LDH in epoxy resin was important to reduce the spread flames. The burn rates show that, without the LDH the polymer had a biggest probability to burn faster than with LDH. What means that the dispersion efficiency is not so relevant as to the mechanical properties.

To the tensile rupture, the rate of dispersion of LDH in the matrix is highly important, as was showed in the results, especially when observes the results in the samples with 0.2% of LDH. Although, the defects that were founded, makes hard to say that the efficiency in the dispersion is important or not, to the improvement in the mechanical behavior.

The fractography and the traction test performed, were not sufficient to identify if the dispersion of the LDH was efficient or not. To identify if the dispersion was efficient, should be performed a scanning electron microscopy, which did not happen due the lack of recourse.

The inclusion of bubbles or the defects cause by the mold, were factors that were not expected in the planning of experiments. To avoid the defects that the mold caused, that should be built with another material different from the silicon, that would not wear out with the use. The bubbles could be prevented using a vacuum mechanism, during the hardening process.

In summary, the LDH's were beneficial when added to the epoxy resin, for both tests, the tensile strength and the flammability.

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