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INFLUENCE OF THE CHEMICAL TREATMENT OF CURAUÁ (ANANAS ERECTIFOLIUS) VEGETAL FIBERS ON THE MECHANICAL RESISTANCE OF COMPOSITES IN EPOXY RESIN MATRIX

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ABSTRACT:

The use of vegetable fibers as reinforcing agents in polymer composites is being increased in substitution of synthetic fibers such as glass fibers or carbon fibers because of good mechanical properties and low density among other properties. In this work an evaluation of epoxy resin composites and curauá fibers is carried out, whose fiber is extracted from the leaves of a plant from the Amazon region called Curauá (Ananaserectifolius). The objective of this work was to study the interaction between the curauá fiber and the resin aiming at a greater bonding of the fiber in the matrix and with that increase the mechanical properties of the composite. Treatments to improve the fiber's wettability were based on sodium hydroxide by varying the concentration and the exposure time of the fibers in the dissolutions. After treated fibers were observed in the Scanning Electron Microscope (SEM) to compare the results of the treatments in relation to the fibril separation and the surface roughness of the fibers, the results of which were then compared with the results of the tensile test. In order to carry out the work, was constructed a mold composed of several male and female cavities with standard dimensions according to the norms for mechanical tensile tests of composites in polymer matrix and fine parallel longitudinal and transversal slits were machined to place the fibers with precision. As a result of the work the 5% sodium hydroxide dissolution which 4 hours of exposure was determined as the best treatment, whose tensile strength values were high, stating that this fiber could in the future replace the glass fiber.

KEYWORDS: *Composites, Curauá, Resin Epoxy, Polymeric matrix .*

1- INTRODUCTION

The development of ecological materials consists of a need to reduce the natural impacts caused by the uncontrollable extraction of natural resources and the reduction of the impact on the environment. In this way to manufacture for the current market products composed of natural fibers goes in the direction of sustainability [1]. In this work was carried out a study of the use of new composite materials through the Curauá (Ananaserectifolius) fiber in matrix of Epoxy Resin. The combination of these two materials resulted in the manufacture of a compound with good mechanical properties capable of replacing the glass fiber in the future.

The curauá fiber of Amazonian origin presents great potential, because it relates good properties, low cost and low density.

Volkswagen of Brazil [2] was the first automobile manufacturer to use the fiber of Curauá, native plant of the Amazon Forest, in its products, replacing fiberglass. In Fig. 1 we can see the plantation of Curauá in the Amazon region.

Figure 1:Cultivation ofCurauá(Ananaserectifolius)
https://paraguacu.wordpress.com/2010/01/11/volkswagen-sustentavel/

The fiber of Curauá within the vegetal fibers has aroused great interest by the fact of being cultivated in a particularly sensitive area in relation to the environmental problems, (Amazonia), [3]. Curauá fiber is the fiber of higher strength and lower density among all vegetable fibers, with tensile strength around 400 MPa.

In the literature we find several works related to the Curauá fiber [4], and [5], where the microstructural aspects were investigated. According to the authors, the natural adhesion between the filaments constituting the fiber originates voids between filaments when chemically treated. These voids allow the penetration of the liquid matrix and may aid in the adhesion to the polymer matrix, resulting in an effective reinforcement for compounds reinforced with Curauá fibers.

2-MATERIALS AND METHODS

This work was carried out using a Sikadur® 32 epoxy resin matrix, which is an epoxy resin, medium viscosity (fluid) and Curauá fibers that are extracted from the leaves of a plant in the Amazon region called Curauá (Ananaserectifolius) of the bromeliaceae family that is attracting much attention, particularly since 1993, when this fiber was commercially recognized through the automotive industry.

The objective of this work was to study the types of fiber treatment to achieve the separation of fiber fibrils, better fiber and matrix adhesion and thus obtain better mechanical properties. The Curauá fibers used in this research come from the City of Santarém, State of Pará, Brazil and were received from SENAI ofSantarém in natura form, asshown in figure 2.

Curauá fibers have a chemical composition of 73.6% cellulose, 9.9% hemicellulose, 7.5 lignin and 0.9% ash [6]. The curauá leaves are hard and have a 1.5-1.7 m long and 4 cm wide, erect and have flat surfaces. In 2003, Brazil produced 150 tons of Curauá [7]. Previous studies have indicated that Curauá fiber is a promising material for reinforcing thermosets and thermoplastics ([1], [8]). The specific mechanical properties of Curauá fibers are very important because good mechanical properties are combined with low density [9].

First the fibers were combed and remove dirt and some debris from foliations as shown in figure 3.

In order to study the influence of the chemical treatment of the Curauá fiber on the tensile strength of the composites the fibers were immersed in different dissolutions of sodium hydroxide and different periods of time as shown in table 1 and in figure 4.

Figure 2: Curauá fiber supplied in natura

Figure3: Curauá fiber combed

In order to study the influence of the chemical treatment of the Curauá fiber on the tensile strength of the composites the fibers were immersed in different dissolutions of sodium hydroxide and different periods of time as shown in table 1 and in figure 4.

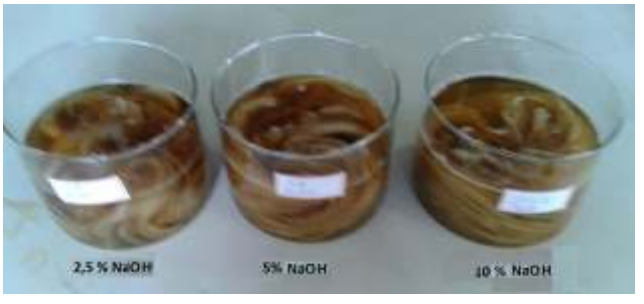


Figure 4: Chemical treatment of the fibers with sodium hydroxide

The experimental design for this study is defined in Table 1. Fiber groups were withdrawn from each dissolution according to the planned immersion times (after 1 hour, 2 hours and 4 hours).

All fiber groups were washed with tap water and thoroughly dried and then dried at room temperature for 48 hours in a closed place protected from the sun and rain. They were then dried in an oven at 60 °C for 24 hours and finally in an oven at 100 °C for 50 minutes to completely eliminate moisture, as shown in figure 5.

Table 1: Chemical treatment of curauá fiber

Test Body No.	CurauáFiber(%)	FiberTreatment
1	30	No
2	30	2,5% NaOH for 1h
3	30	2,5% NaOH for 2h
4	30	2,5% NaOH for 4h
5	30	5% NaOH for 1h
6	30	5% NaOH for 2h
7	30	5% NaOH for 4h
8	30	10% NaOH for 1h
9	30	10% NaOH for 2h
10	30	10% NaOH for 4h
11	0	0

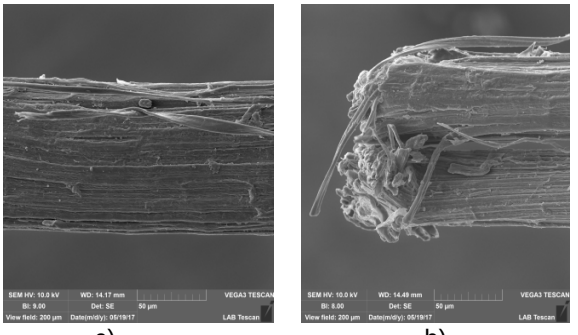
Figure5: Fiber drying

For use in composites it is important to know the thermal degradation of these fibers. As the vegetable fibers are composed of different components, such as:cellulose, hemicelluloses, lignin, etc., their thermal decomposition results in complex reactions. In previous studies four stages of decomposition are seen in the thermogravimetric analysis of Curauá fiber as [10].

The first stage is the evaporation of water at 100°C, approximately 3% weight loss, which indicates the need to dry the fibers in the case of molding of composite materials, where the moisture of the fiber decreases adhesion to the matrix, the second stage is the decomposition of hemicelluloses at 268 °C, the third, the degradation of cellulose at 335 °C and the fourth the degradation of lignin, which occurs slowly, with a maximum decomposition rate at 439 °C. From these thermogravimetric analyzes we know that we should dry the fiber at 100°C for 50 minutes as the last drying process and also that as the curing process of the resin is less than 200°C it will not degrade the fiber.

Before the manufacture of the specimens in order to know the effectiveness of the treatments performed, samples of each group of fibers were observed to the Scanning Electron Microscope (SEM). The microscope used is of the TESCAN mark and VEGA3 model, of the TESCAN Company from Sao Paulo.

In the figure 6Acan be observed a micrograph of the lateral part of an untreated Curauá fiber and in figure 6b the tip of the fiber. In this figure one can observe the fiber fibrils all joined together forming a single fiber bundle.

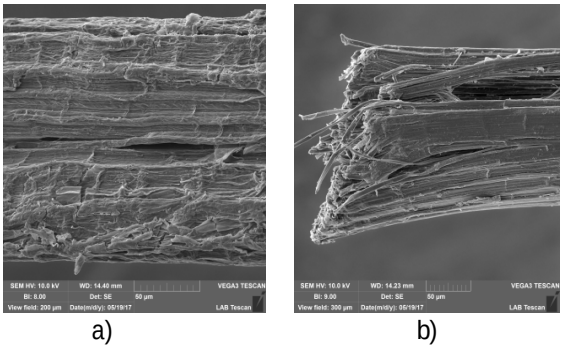


a) b)

Figure 6: Micrograph of an untreated fiber surface, a) lateral side of the fiber, b) - Fiber tip

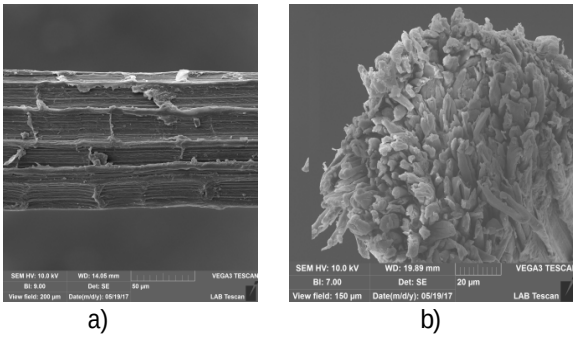
The Figure 7a shows a micrograph of the lateral side of a chemical treated of Curauá fiber in a sodium hydroxide dissolution of 2.5% and two hours exposure and in Figure 7b the fiber tip with the same treatment. In this figure it can be observed that the fiber fibrils are beginning to separate due to the effect of the treatment.

In the figure 8a can be observed a micrograph of the lateral part of a Curauá fiber with chemical treatment in 5% sodium hydroxide dissolution and four hours of exposure to this treatment and figure 8b shows the fiber tip with the same treatment. In this figure can be observed the fiber fibrils that are much more separated due to the effect of the treatment.



a) b)

Figure 7: Micrograph of a fiber surface with chemical treatment in a 2.5% sodium hydroxide solution and two hours exposure, a) Lateral side of the fiber, b) - Tip of the fiber



a) b)

8: Micrograph of a fiber surface with chemical treatment in a sodium hydroxide dissolution of 5% and four hours exposure, a) lateral side of the fiber, b) - Tip of the fiber

From previous studies [1,4, 5, 6] it is known that the dimensions and properties of curauá fiber depend among other factors of the climatic conditions and the soil where it grew. The results of fiber diameter measurements of these studies vary from one author to another. In this work seven samples of sections were

prepared along one of the longer Curauá fibers to determine the average diameter, as can be seen in the micrograph of figure 9. These micrographs were obtained through a scanning electron microscope (SEM) of the brand ZEISS, model LEO 435VP, with an electron beam of 15 kN of the National Institute of Research of the Amazon (INPA).

The representation of all measurements performed to determine the average diameter of a fiber is shown in figure 10. The diameter of the fiber varied from 54 to 127.15 micrometers, obtaining an average diameter of 84 micrometers.

Also, 63 measurements of the diameter of different fibers were made as shown in the micrograph of figure 11 to have an approximate average of the diameter of the fibers, which we are working with.

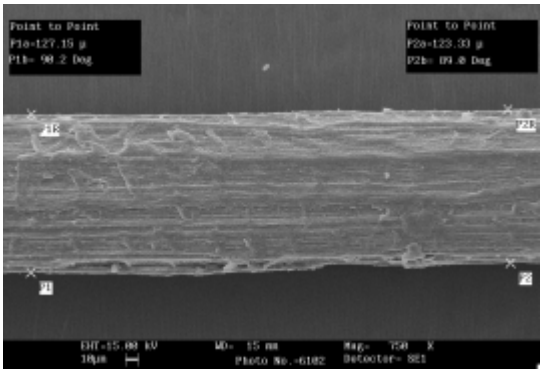


Figure 9: Surface micrograph of a treated fiber with a 5% NaOH dissolution for 1 hour exposure (750 X)

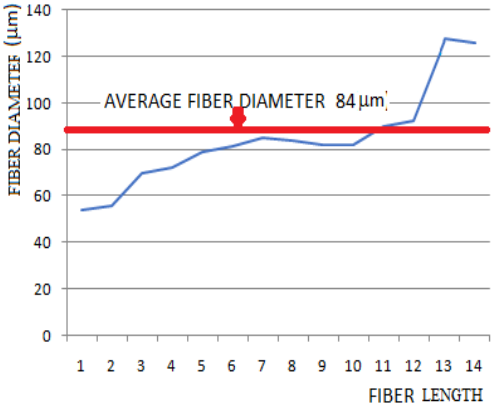


Figure 10: Diameter of the fiber along its length

The figure 12 shows the representation of all the measurements performed in a histogram graph. The diameter of the fiber ranged from 33 to 138.42 micrometers, obtaining an average diameter of 84 micrometers.

Subsequently, the specimens were made in a mold designed and manufactured to make the specimens for tensile tests according to ASTM D636 (I). In Figure 13 the mold can be seen and in Figure 14 the plate with the cavities with the dimensions of ASTM D636 (I), supported on the lower mold support.

After filling the cavities with the mixed resin and the fibers the superior part of the mold is guided down by columns. It is using a press to overcome the force of the springs and that the males penetrate in the cavities compressing the composite and eliminating possible bubbles or voids. This type of mold allowed applying pressure directly in the composite eliminating the formation of bubbles, helping in the curing process of the resin and also allowed to put the fibers well directed and fixed in such a way that they maintain the position without movement during the curing process of the resin.

Each set of test specimens was left in the mold for 24 hours in a press with 4 tons of pressure to eliminate

air bubbles and to obtain greater compaction of the material.

After extraction from the mold the final curing process was carried out in an oven at 80 °C for 8 hours and then at 125 °C also for 8 hours, according to the manufacturer's recommendations. Figure 15 shows the specimens after the curing process.

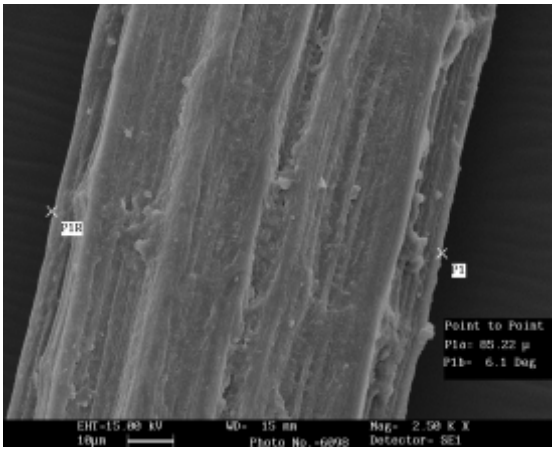


Figure 11: Micrograph of the treated fiber with a dissolution of 5% NaOH during 4 hours of exposure (2500 X)

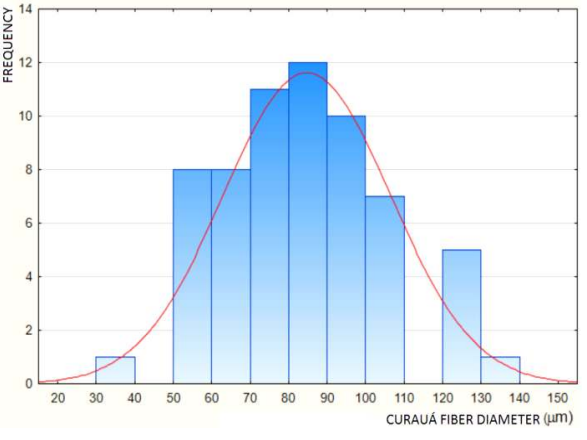


Figure 12: Histogramgraph of fiber diameter measurements

After the curing process, all the specimens were subjected to tensile tests to determine the mechanical strength of each composite and to study the influence of the treatment of the fiber on the tensile strength of the composite.

The tensile tests were performed on an INSTRON Brand Universal Electromechanical Testing Machine, model 5984, with a load cell of 150 KN. The tests were performed at a traction speed of 5mm / min.

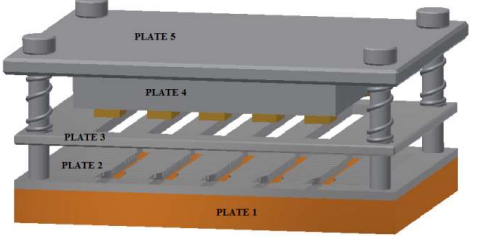


Figure 13: Mold for manufacturing test bodies according to ASTM D636 (I)

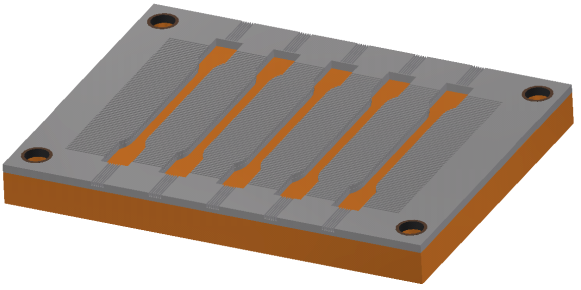


Figure 14: Cavity plate with the dimensions of ASTM D636 (I), on the lower mold support

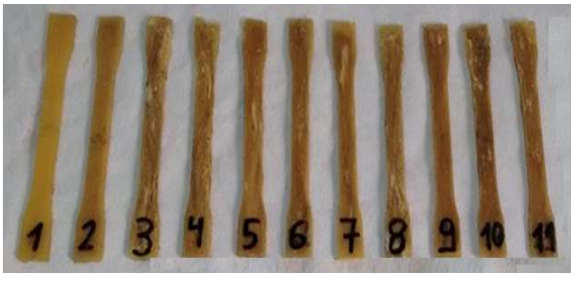


Figure 15: Proof bodies after the curing process.

In figure 16 can be observed the traction machine used at the moment of starting the traction force and in Figure 17 the same machine at the moment when the test piece fails.Figure 18 shows the traction graphs generated by the machine

3- RESULTS AND DISCUSSION

The morphology of the fibers after the treatment showed in figures 6 to 8 that the treatment with sodium hydroxide was able to open the cellulose fibrils and to increase the surface roughness, providing a greater adhesion between the fiber and the matrix and increasing the resistance to traction of the composite.

Figure 19 shows a micrograph of the fracture zone of a test piece made with a mold without pressure, with curing process only by the action of gravity, in this micrograph can be observed many internal bubbles that caused the Fracture at this point.

Figure 16: Electromechanical Universal Testing Machine mark INSTRON, model 5984 at the moment of starting the tensile force

Figure 17: Electromechanical Universal Testing Machine Mark INSTRON, Model 5984 at the time when the test body fails

In the figure 20 we can observe the micrograph of the fracture zone of a test piece made with the manufactured mold, with pressure maintained during curing process, in this micrograph it can be observed the absence of internal bubbles. This micrograph corresponds to the test body whose fibers were maintained for 4 hours in dissolution of 5% sodium hydroxide. Thus, it can be observed also how it had a good wettability of the fiber allowing a good adhesion with the matrix and increasing the mechanical resistance.

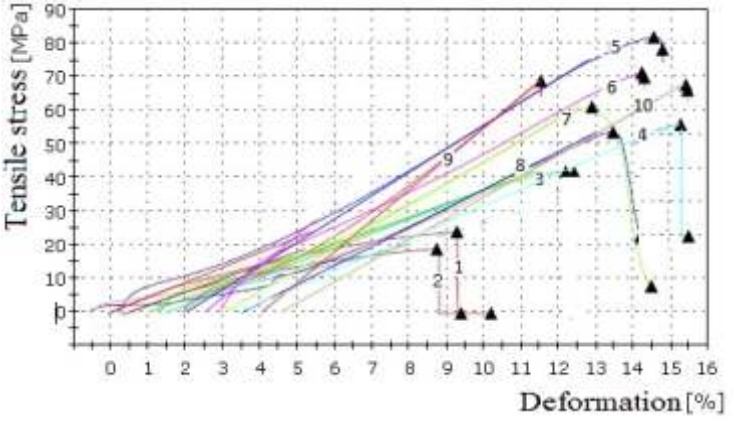


Figure 18: Traction graphs generated by the machine

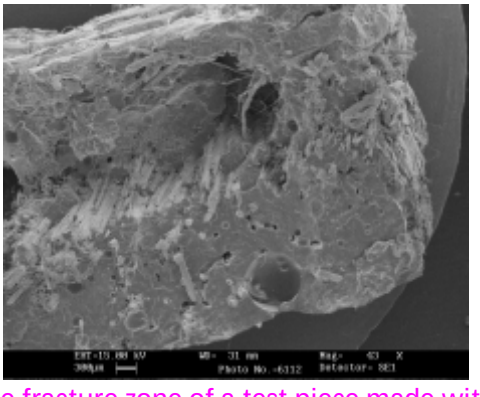


Figure 19: Micrograph of the fracture zone of a test piece made with a mold without pressure

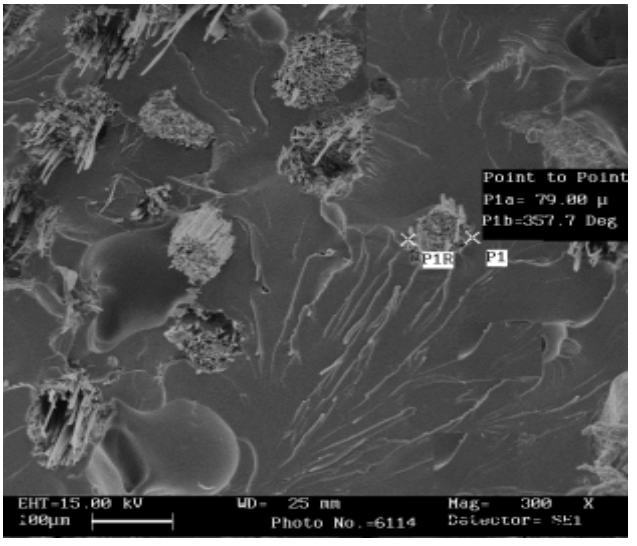


Figure 20: Micrograph of the fracture zone of a test body made with a mold with pressure during curing process

The Table 2 shows the results of tensile strength of each specimen. It can be seen from these results that the treatment with sodium hydroxide improved the strength of the composite, which corresponds to the morphological study of the treated fiber. Of all the treatments as can be observed in the morphological part and in the Table 2, the treatment with sodium hydroxide of greater effectiveness is done with dissolution of 5% and a time of exposure of 4 hours.

Treatments with sodium hydroxide with a lower concentration of 5% and exposure times less than 4 hours did not separate well the cellulose fibrils and, consequently, the greater adherence was not achieved. Treatments with sodium hydroxide with a higher concentration of 5% or longer exposure time of 4 hours compromised the fiber resistance and, consequently, greater resistance of the composite, as can be seen in figure 21.

Table 2: Results of tensile strength of each test body

Test Bodynº	CurauáFiber(%)	FiberTreatment	Tensile strength (Mpa)
1	30	No	44,72
2	30	2,5% NaOH for 1h	32,8
3	30	2,5% NaOH for 2h	71,67
4	30	2,5% NaOH for 4h	73,36
5	30	5% NaOH for 1h	45,44
6	30	5% NaOH for 2h	49,36
7	30	5% NaOH for 4h	78,37
8	30	10% NaOH for 1h	75,58
9	30	10% NaOH for 2h	65,95
10	30	10% NaOH for 4h	61,08
11	0	0	23,87

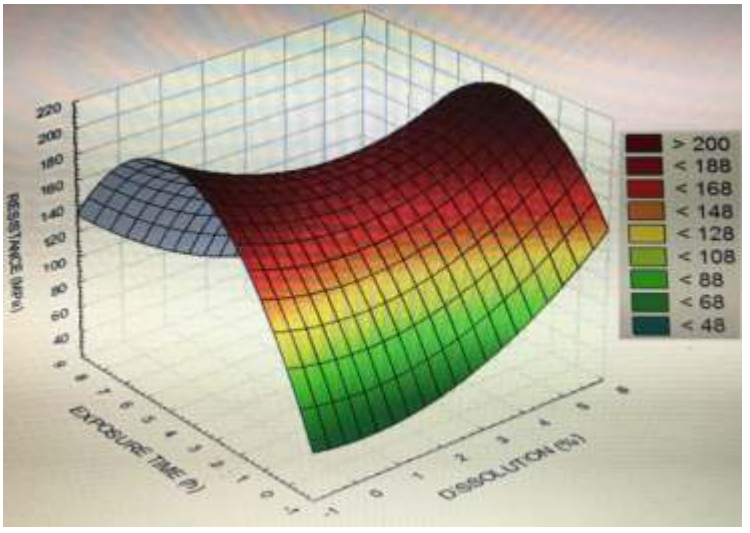


Fig. 21: 3D Surface Plot of tensile stress (Mpa) against %of dissolution and exposure time (h)

4- CONCLUSIONS

The result showed that using a sodium hydroxide solution of 5% for 4 hours, the composite strength was increased, mainly due to the increase in the surface roughness of the fibers and the separation of the fibrils which increase the contact and ensure a good bonding between the Matrix and the fiber, thus increasing the resistance.

The results show that the composite studied with the Curauá Amazon fiber can even replace the composites with synthetic fibers, providing an improvement to the environment, helping in sustainability and opening new sources of employment for the poorest populations of the Amazon region.

5- ACKNOWLEDGEMENTS:

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