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Analysis of Surface Degradation PP and HDPE Banana Fiber by Scanning Electron Microscopy (SEM)

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Abstract
The purpose of this article is to evaluate the surface behavior of polypropylene (PP) and High Density Polyethylene (HDPE) with loads of 5% and 10% in banana fibers in solutions of distilled water, ethanol and sodium chloride (5%) for 15, 30, 45, 60, 90 and 200 days. The samples were sectioned and then weighed for the area measurement of degradation and fluid (mass difference in time function) absorption, according to the environment. The expected results show that the longer the immersion time, the greater the material degradation, regardless of the respective environment. It is also evaluated the material degradation by Scanning Electron Microscopy.

Keywords: Composites, Degradation, Product Design, Scanning Electron Microscope

I. INTRODUCTION

The material innovation that is being experimented suggests an opening of possibilities and resources optimization, and thus the material choice becomes almost unlimited. To designers referring to the product design, this suggests using materials and manufacturing methods as innovation factors. Materials should be seen as stimulation to creativity in their application. The material choice and manufacturing process become a product concept characterizer not limiting themselves to an engineering issue, generating possibilities and innovation related to design. Therefore, it is necessary that interaction between material engineering and design can be guaranteed already in the methodology conception of the products of synergy [1].

In a lot of reports it is observed the products development using cellulose fibers (vegetal residue) of jute, sisal, linen, banana, bamboo, wood, palm and coconut as reinforcement in thermoset or thermoplastic polymers, according to the product characteristic and region [2,3].

The reinforced composites are denominated by the action in which the fiber acts as a fragmented phase in the products present in the high resistance character and weight related rigidity. Therefore, these parameters are expressed in resistance and specific module terms, being the reason between the traction resistance limit and specific weight, and between the elasticity module and specific weight [4,5].

One of the advantages of using polymeric material as polypropylene and polyethylene is their recyclability (Life Cycle). Polymeric composites reinforced with fibers have received a great attention in the last decades due to their strength/tension and module. Recently, the natural fibers have acted as reinforcement to polymeric composites and have become interesting due to their low cost and biodegradability propriety [6].

To select the correct material and after reducing its corrosion the longer as possible, it is essential to know the effects of various factors, such as temperature, chemical composition, heat treatment, superficial characteristics and environments [7].

The natural fibers reinforced composites are applied in many areas, from the automotive industry, packing and even in the civil construction. Some studies have showed also that chemical treatments, done in fibers, improve significantly the fiber/matrix interfacial adherence, above all, guarantee the interesting and inherent characteristics to the natural fiber, contributing the environmental and industrial management by the means of polymer recycling [8,9].

The automotive and aerospace industries have demonstrated an interest in using more natural fibers reinforced composites, for example, in order to reduce vehicle weight, automotive companies have already shifted from steel to aluminum and now are shifting from aluminum to fiber reinforced composites for some applications. This has led to predictions that in the near future plastics and polymer composites will comprise approximately 15% of total automobile weight [10].

However, certain drawbacks, such as poor compatibility with the hydrophobic polymer matrix, the tendency to form aggregates during processing and the low resistance to moisture, greatly reduce the potential of natural fibers to be used as reinforcement for polymers [11,12]. The incompatibility may cause problems in the composite processing and material properties.

The polymers absorb water when submerged in watery means or exposed to humidity, but their absorption depends a lot on the polarity grade, crystallinity, among other factors. Fibers tend to absorb more humidity. So, a natural fiber reinforced polymer becomes the material with the highest grade of water absorption, compared to an isolated polymer [13]. The intensity of the absorption can have modifications and loss of dimensional stability of compounds.

This article has as an objective to evaluate the real superficial characteristics of Polypropylene (PP) and High Density Polyethylene (HDPE), with loads of 5% and 10% of banana fibers in distilled water solutions, ethanol (92,8º), sodium chloride (5%) and biodiesel during the immersion of 15, 30, 45, 60 and 90 days, by optical microscopy and scanning electron microscopy and also to verify the dispersion of fibers in the matrix.

II. MATERIALS AND EXPERIMENTAL PROCEDURE

In this work it was used, for surface characterization and degradation tests, the following composite, HDPE and PP reinforced with 5% and 10% of banana fiber, respectively.

2.1 Superficial preparation of the samples

Many authors have studied the influence of chemical treatments of fibers in mechanical behavior of composites so that these characteristics can be optimized and promote the use of these composites as viable alternatives replacing including the composites that use glass fibers [14-18].

Treatment and chemical modification of fibers are conducted to improve the adhesion conditions between fibers and the matrix or even to change the fibers characteristics. It is known that some natural fibers are lignocellulosic materials that own many hydroxyl groups along their chains, what confers a great hydrophilicity to the fiber, as the high density polyethylene (HDPE). The hydrophobic matrixes are unmatchable with hydrophilic fibers, the obtained composites with these two materials use to present structural problems caused by their incompatibility. Thu, it has been applied the use of compatibilizing agents and chemical modification processes in both fiber and matrix to improve the final composite properties [19-23].

Banana pseudostalk fibers modified were mixed with HDPE in a thermokinetic mixer, model MH-50H, with speed rate maintained at 5250 rpm, in which fibers were responsible for 20 wt% in composition. After the mixture, composites were dried and ground in mill, model RONE. Composites and pure HDPE were placed in an injector camera at 165ºC and 2ºC min^{-1} heating rate in required dimensions pre-warm mold to obtain tensile specimen (Figure 1).

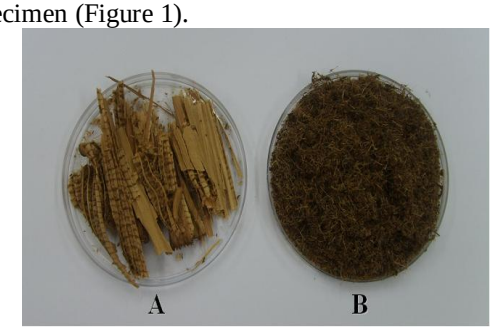

Fig. 1 - Banana pseudostalk fibers in nature (A) and modified (B).

Specimens confection to the immersion tests was done in the Laboratório de Materiais, Texturas e Modelagem at FATEA, with the objectives of preparing them to the degradability tests in different means of immersion and time.

The samples were weighted individually in an analytical balance (Shimadzu AY 220) in Laboratório de Química of Instituto Santa Teresa to determine the material degradation in frequency of immersion time in different means.

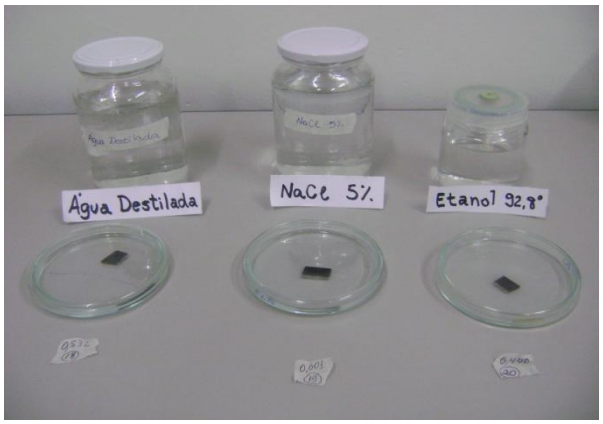

Fig. 2 - Samples immersed in the means distilled water, NaCl and ethanol

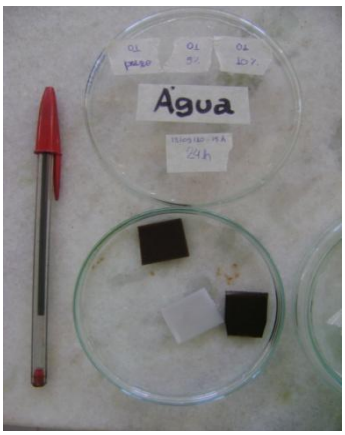

Fig. 3 - Sample immersed in water

The immersion media done for degradation tests were: distilled water, sodium chloride in solution of 5%, ethanol (92.8º) and biodiesel during 15, 30 and 45 days.

2.2. Optical Microscopy and Scanning Electron Microscopy

The micrograph was done in the Laboratório de Materiais, Texturas e Modelagem at FATEA (Figure 4), by means of an optic stereoscopic, by Topcon, with magnification ranging from 20 – 100 times, aiming to evaluate the kinetics of superficial degradation of samples before and after immersion.

Fig. 4 - Stereoscopic Linked to an Image Analysis System

After degradation test in different media and attack time, samples were metalized and then, a scanning electron microscopy was done (Figure 5) using the secondary electron mode with magnification of 50 to 10.000 X aiming to view the topography and morphology of fibers in relation to the matrix of polypropylene and polyethylene.

Fig. 5 - Scanning Electron Microscopy – SEM

The Scanning Electron Microscopy process after the inlay was done in the metallography laboratory at the Engineering school of Lorena.

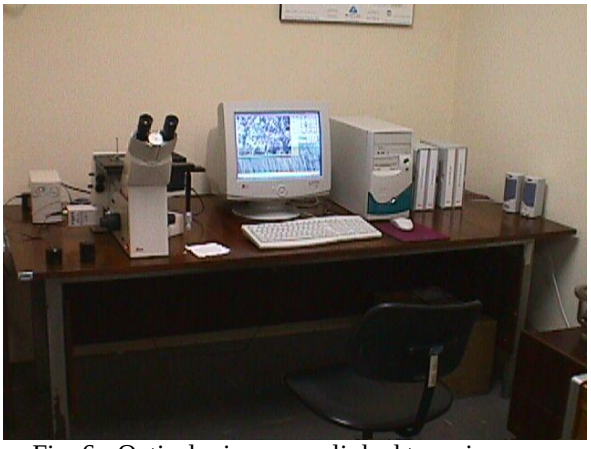

Fig. 6 - Optical microscopy linked to an image analysis system

The surface preparation stage began with the cold-inlay (Serifix-Struers resin), and then the sanding with sanders made of silicon carbide, followed as #220, 320, 400, 600, 2400 and 4000. The polishing was done with application on the cloth (OP-Chem) impregnated with colloidal silica (OP-U). The microscopy analysis was done in clear field with magnifications ranging 50 to 1000 times with the aid of an Optical Microscopy type 451DRM- Leica.

III. RESULTS AND DISCUSSION

3.1. Material Analysis

The matrix material chosen for this paper was the high density polyethylene (HDPE) and the polypropylene (PP), due to their attractive characteristics, such as low cost, processing easiness and recyclability and also for presenting good impact resistance. Besides, this polymer is an important engineering thermoplastic, widely used in industrial applications.

The mechanical properties analysis of a material is essential to its selection to the project of a product. Therefore, evaluating a composite reinforced with vegetal fibers, in this case banana fiber, it can work as comparison basis of this composite with the pure polymer, and evaluate its advantages and disadvantages, and where it can be applied.

Therefore, assessing a composite reinforced with natural fibers, where banana fiber for both the PP and HDPE, can serve as a basis for comparing the composite with the pure polymer before its advantages and disadvantages in their application.

Figures 7-10 show the graphs of absorption of different components of degradation by immersion carried out up to a constant weight in composites reinforced with banana fibers.

The moisture absorption to plastics in general is low, while the lignocellulosic materials have a tendency to absorb moisture because they are composed mainly of cellulose, lignin and hemicellulose that absorb moisture (hydrophilic) between 6 and 14%. Lignocellulosic materials

change their dimensions when they absorb moisture, because the cell walls of these materials have hydroxyl groups that interact with water through hydrogen bonds.

This relationship was based on the calculation of reagents absorption, according to equation 1.

$$\Delta M(\%) = \frac{Mf - Mi}{Mi} \times 100, \quad \text{Eq. 1}$$

Where: ΔM is the water absorption; Mi and Mf are masses of samples before and after immersion in different media.

Below are the graphics, showing the material degradation related to mass (g) to time (days):

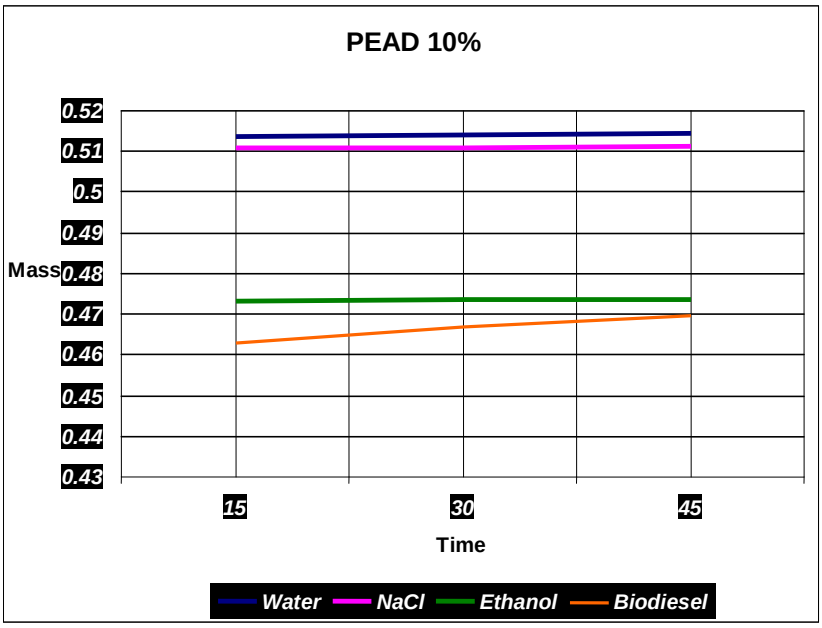

Fig.7 - Absorption of different reagents in function of time, obtained to the reinforced composites of HDPE 10% with modified cellulose fibers

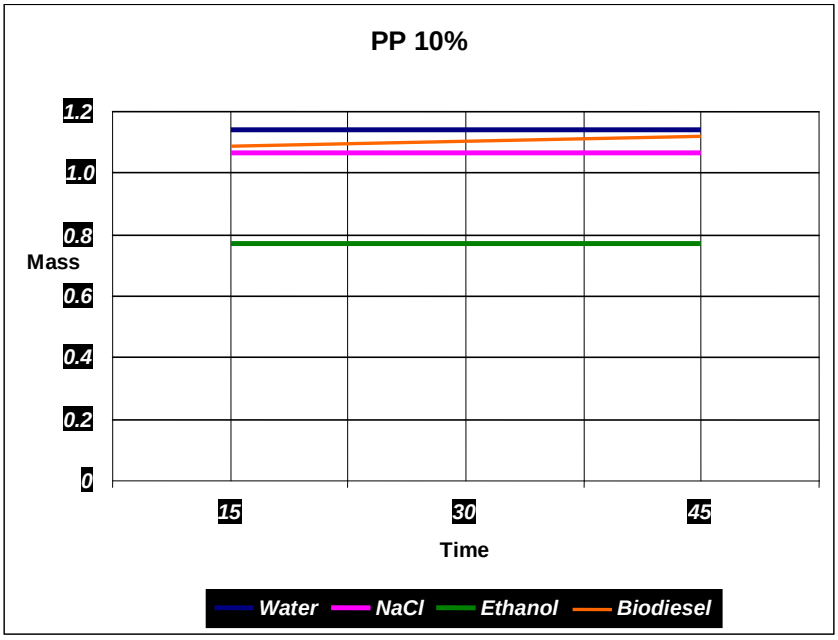

Fig. 8 - Absorption of different reagents in function of time, obtained to the reinforced composites of PP 10% with modified cellulose fibers

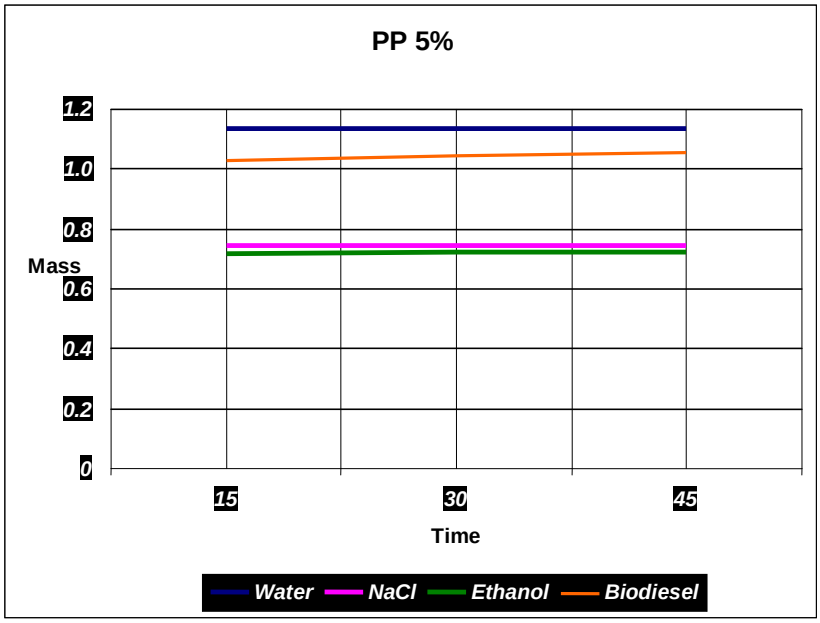

Fig. 9 - Absorption of different reagents in function of time, obtained to the reinforced composites of PP 5% with modified cellulose fibers

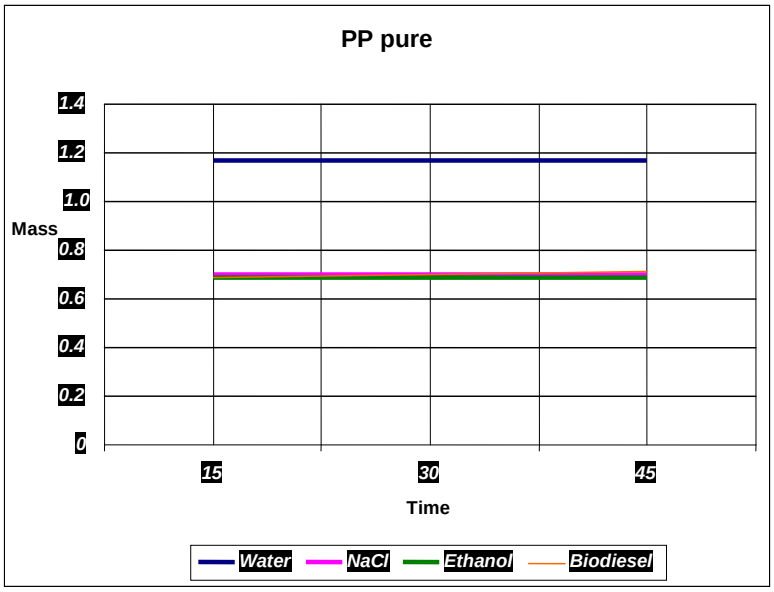

Fig. 10 - Gain mass value with pure PP in different reagents.

Results in degradation phase show that the longer the immersion time and increasing degradation of the material, regardless of medium, the better to view difficulties in adhering fibers in relation with polymer matrix and application of textures on their surface.

The distribution and fiber length in the matrix is a very important parameter to be determined, for the fibers length can alter meaningfully the mechanical performance, as well other composite properties [4,24]. In composites with discontinuous fibers, the load in the fiber is function of its length, and its extreme points are stress concentrators, that induce shear stress in the interface. Besides, some of the possible damages in the composites are associated to the flaw of link between fiber and matrix and break of the fiber. So, in order to evaluate these parameters the optical microscopy techniques and scanning electron microscopy are the most adequate.

3.2. Optical Microscopy and Scanning Electron Microscope

The SEM technique is used to evaluate the adhesion between the fiber and the matrix of fractured surface of composites. The optical microscopy is a technique that provides information about aspects such as length and fiber distribution in the matrix.

In Figure 8, it is observed by optical micrography the random scatter in distribution and size of fibers contributing to the embrittlement (strength loss) and ease in degradation of its surface.

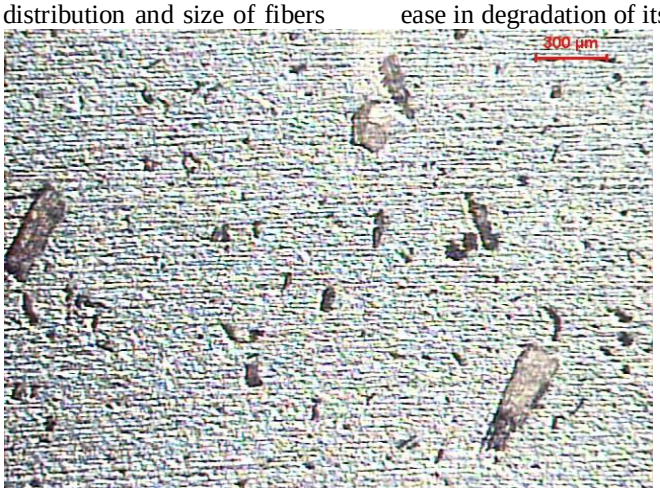

Fig. 8 - Micrography of PP with dispersion with 10% of banana fiber

Analyzing the materials of high density polyethylene and polypropylene with 5% and 10% of banana fiber, the most affected was the polypropylene in biodiesel and ethanol, during 45 calendar days immersed. It is displayed below some images done by SEM, showing the material degradation.

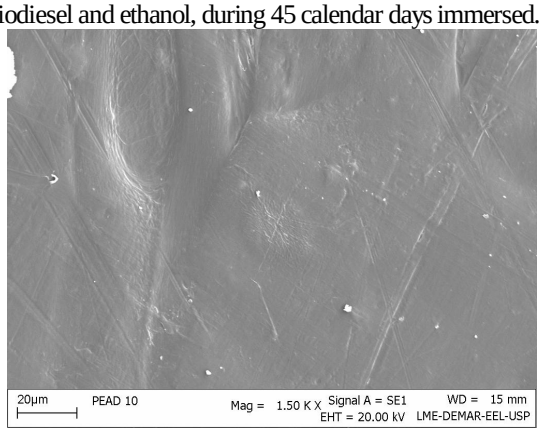

Fig. 10 - HDPE with 10% banana fiber without immersion.

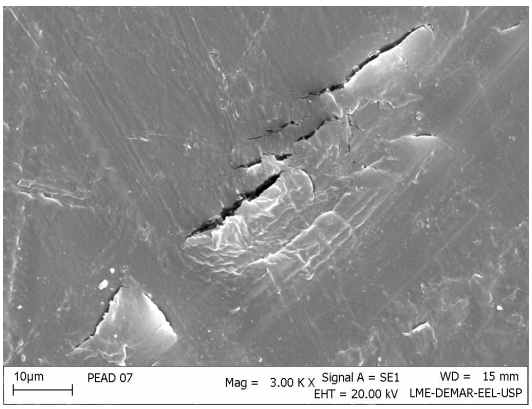

Fig. 11 - HDPE with 10% banana fiber immersed in Biodiesel

Figure 10 shows the HDPE with 10% banana fiber without immersion process and results in a surface attack-free. In contrast, Figure 11 shows the degraded surface with biodiesel for 45 days and result includes formation of microcracks.

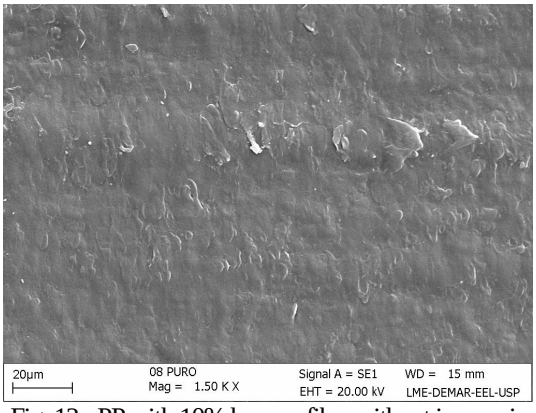

Fig. 12 - PP with 10% banana fiber without immersion.

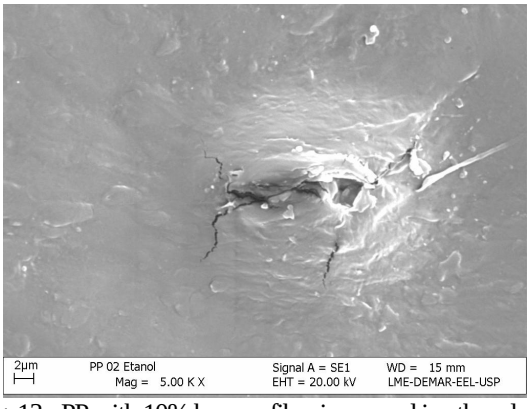

Fig. 13 - PP with 10% banana fiber immersed in ethanol

Figure 12 shows the PP with 10% banana fiber without immersion process and results in a surface free of attack. In figure 13, the surface behavior of PP with 10% of banana fiber in immersion process of 45 days showed degradation

points to the almost cylindrical morphology with growth trends through the cracks present.

The degradation is enhanced through the immersion process in all situations and especially for biodiesel, according to the micrographs via scanning electron microscopy and the graphical immersion.

IV. CONCLUSION

Regarding the results in degradation and submission of specimens in different media and time, it is concluded that absorption tests carried out on HDPE with 10% banana fiber showed an increase in degradation rate between biodiesel and ethanol followed by deionized water. The PP had a similar behavior in the reagents sodium chloride, ethanol, biodiesel and water with a more aggressive attack. In general, the HDPE has a higher absorption rate than polypropylene for different degradation modes. In scanning electron microscopy, the immersion process via Biodiesel for 45 days showed the PP and HDPE microcracks and points located on degradation ratio of fiber and matrix composite.

V. ACKNOWLEDGEMENTS

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