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Degradation of Polyester-Based and Vegetable Fiber Polymeric Composites

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ABSTRACT

In recent decades, polymeric materials have been considered to be a cause of environmental problems worldwide. An alternative to address this problem is the use of biodegradable polymers, which are materials that are degraded through the action of microorganisms and that are becoming a new global trend. In this study, composites were prepared using a biodegradable matrix based on polyester and cornstarch (Ecobras™) with vegetable fibers of coconut and corn stover, having two types of polymerization degree, namely, film and injection-molded samples. Degradability studies were conducted in simulated soil, following the ASTM G 160-03 protocol, through the monitoring of mass loss and morphology observations of Ecobras™ free from vegetable fibers and of composites combining film-grade Ecobras™/coconut fiber and injection molding-grade Ecobras™/corn stover, with vegetable fiber contents of 1%, 5% and 10% compared to the polymer matrix. The degradability tests showed that that all samples experienced significant mass losses. It was possible to confirm the mass loss and observe the presence of microorganisms attached to the samples after 17 weeks of degradation.
Keywords: Ecobras™, Green Coconut Fiber, Cornstover, Degradation

I. INTRODUCTION

Due to their properties, versatility and price, polymers have many applications in the device market. However, the spread of petrochemical-based polymeric materials increases the use of non-renewable resources and leads to the accumulation of large amounts of non-biodegradable waste. Pollution caused by non-biodegradable polymers has become a global concern. These materials pollute rivers, lakes, and soil, causing a severe negative impact on the environment [1-3].

To reduce the environmental impact, readily degradable materials should be developed from renewable sources [4-5]. An example for minimizing the impact on the environment is the use of natural fibers in biodegradable polymer matrices.

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Ecobras™ is a synthetic polymer based on polyester and cornstarch. Ecobras™ is a biodegradable and compostable plastic produced by BASF and Corn Products that contains 50% vegetable starch from corn and Ecoflex® (polybutylene adipate co-terephthalate (PBAT)). Ecoflex® is, in turn, obtained from the combination of 1,4-butanediol, adipic acid and terephthalic acid. Ecobras™ has an average molecular weight of 66,500 g.mol^{-1} and is most commonly used in film processing. For use in processing by extrusion or injection molding, a polymer with a higher molecular weight may be necessary. Chain extension provides a simple approach for generating this high molecular weight material, at least for test applications [6-7]. Ecobras™ was developed to possess biodegradability along with good mechanical and processing properties. This material has a significantly lower viscosity than Ecoflex® because the corn starch used in manufacturing Ecobras™ is modified with maleic anhydride, a plasticizer with a low molecular weight, and when incorporated in the form of granules, the unstructured synthetic polymer matrix causes a reduction in viscosity [8-9].

The objective of the present study is to evaluate the biodegradation of Ecobras™ composites with cornstover and coconut fibers in simulated soil.

II. METHODS AND MATERIAL

1. Preparation of the composites

This study used a matrix of the composite commercial polymer Ecobras™ with two different processing specifications, film-grade Ecobras™ and injection molding-grade Ecobras™, kindly provided by BASF and Corn Products.

The film-grade Ecobras™ samples were processed at a temperature of approximately 115°C for 8 minutes using a rotor speed of 60 rpm, followed by compression using a hydraulic press at 115°C for 10 minutes to form specimens 0.5 mm thick and 30 mm in diameter.

Injection molding-grade Ecobras™ samples were prepared at a processing temperature of 140°C, a rotor speed of 100 rpm and a total mixing time of 8 minutes. The samples were then injected into a semi-industrial molding machine at a temperature of 135°C to obtain specimens 20 mm long, 12 mm wide and 2 mm thick.

The materials used for the formation of film-grade samples were film-grade Ecobras™ free of coconut fiber and film-grade Ecobras™ with coconut fiber provided by Green Coconut Project, Rio de Janeiro, Brazil.

The materials used for injection molding-grade samples were injection molding-grade Ecobras™ free of corn plant material and injection molding-grade Ecobras™ with milled corn stover provided by EMBRAPA Corn and Sorghum, Sete Lagoas/Minas Gerais, Brazil.

For the preparation of the composite, the ratios of polymer (m)/natural fiber (m) for both coconut fiber and corn straw were 99/1, 95/5 and 90/10.

2. Degradation evaluation

The degradation evaluation used was in accordance to ASTM procedures [10]. Samples were removed at 2 to 17 weeks during the degradation tests, and for each week one vessel was prepared with the composite free of fiber, and placed in a chamber at 30±2°C. Each sample was cleaned and weighed to evaluate the mass loss and morphological structure of each sample. The experiments were performed in triplicate.

2.1. Mass loss assessment

All samples were taken from each system according to the period of the simulated soil test. The samples were cleaned and weighed to obtain a constant final mass at the end of the degradation process in simulated soil.

The mass loss percentage was analyzed at 2 to 17 weeks after the withdrawal of samples of composite and of Ecobras™ free of natural fiber from each degradation test in simulated soil.

2.2. Morphological analysis

Samples were dried by critical point CO_2 and metalized by sputtering with a thin layer of gold. The microorganisms were observed by scanning electron microscopy at original magnifications of 1,500 to 10,000 with an accelerating voltage of 15kV.

III. RESULTS AND DISCUSSION

1. Mass loss assessment

The mass losses from Ecobras™ free of natural fiber and from the composite were analyzed at 2 to 17 weeks. There was a significant mass loss from both composites and Ecobras™ free of natural fiber. These mass losses can be attributed to the degradation of Ecobras™ and the biodegradation of the natural fibers. It could be observed that the initial mass loss were high, with each sample exhibiting a significant mass loss in the first two weeks. The high content of starch in the Ecobras™ and its composites could have highly contributed to this initially higher degradation [2,11].

The mean percentages of mass loss of composites and Ecobras™ free from natural fibers are in disagreement with the literature, which reports that mass loss values decrease over the course of weeks of biodegradation testing [12-14]. In the present work a stabilization of the biodegradation could be observed from the 2nd to the 17th week of test.

Note that as the fiber content increases in the composition of the composite, a decrease in the mass loss of the material is observed in relation to the fiber-free polymer, that can be explained by the presence of natural fibers, which show a decrease in the rate of biodegradation of the natural fiber samples due to hindering the action of microorganisms in the Ecobras™ polymer matrix [11, 15-17].

In comparing the mass loss of the film-grade EcobrasTM and injection molding-grade Ecobras™, it is important to mention that the increase in molecular weight depends strongly on the processing temperature. Thus, samples of injection molding-grade Ecobras™ showed an increase in the difficulty of degrading the material due to its higher molecular weight and thicker specimens than the film-grade Ecobras™ [7].

2. Morphological analysis

In the scanning electron microscopy evaluations of composite samples and Ecobras™ free of natural fibers, it is possible not only to observe the changes in morphology as a result of each sample biodegradation process but also to assess the adherence of the polymer matrix and natural fiber in the composites.
It was observed that the surfaces of Ecobras™ samples free of natural fibers before the burial test in simulated soil are smooth, without gaps or holes in the surface of the material. However, over the weeks of degradability testing, the Ecobras™ samples free of natural fibers developed voids or holes in the material surface undergoing a noticeable change in morphology. This is in accordance to the results reported by [18].

Coconut fiber and the corn stover were incorporated into the polymer matrix in all composites. In the samples of polymer matrix with coconut fiber or corn stover, the degradation behavior was very similar to that of the polymer free of natural fibers.

Therefore, it could be observed that in the following weeks, the surface morphology of each material changed during the degradation test. One can also investigate the presence of natural fibers separated from the matrix during the degradation test because the natural fibers were fully engaged in the Ecobras™ polymer matrix throughout the sample processing, but during the simulated soil testing, the coconut fiber and corn stover began to separate from the polymer matrix. This phenomenon was because biodegradation occurs preferentially in the polymer, whereas for the coconut fiber and corn stover, the decoupling matrix requires more time to degrade.

It could be observed the presence of microorganisms both in film-grade Ecobras™ free of coconut fiber and in injection molding-grade Ecobras™ free of corn stover. This result indicated that the material is biodegradable, as described in the literature [19-20].

IV. CONCLUSION

The Ecobras™ and its composites with cornstove and coconut fibers lost more than half of its mass during the period of the test, indicating that the biodegradation process took place, particularly in the first two weeks.

The observation of microbial biofilms on the surface of Ecobras™ and its composites confirm the involvement of microorganisms in the degradation process of these materials. This is also emphasized by the observation of microbial cells at the end of the test, confirming that they are still metabolically active at the expenses of the polymeric materials. Therefore, we can assume that Ecobras™ and its composites are biodegradable.

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