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Biodegradation in Soil of the PHB/Wood Flour (80/20) and PHB/Sisal Fiber (80/20) Tubes

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This paper presents the results of a study that aimed to monitor and evaluate biodegradation in soil, by the mass loss and the mechanical properties of plastic tubes. For this work, there were extruded and injected plastic tubes made of biodegradable polymer poly(hydroxybutyrate) - (PHB) and composite PHB/Wood Flour and PHB/Sisal Fiber, both with 20% fiber. There were used three biodegradation test devices for 30, 60 and 90 days. At the end of each test, the biodegraded samples were taken from soil, washed thoroughly with water, dried and weighed to evaluate the mass loss. Mechanical properties were evaluated using flexural tests before and after biodegradation in soil. Based on the results, it was observed that all the studied tubes lost mass over the biodegradation test, and the tubes of biodegradable composites lost more weight than pure PHB.

Keywords: *Biodegradable polymers, Vegetable fibers, Tubes, Poly(hydroxybutyrate) - PHB, Biodegradation in soil*

1. Introduction

The synthetic polymers have a very large resistance to degradation, which makes the accumulation of these increasing in dumps, in addition, generate a exhaustion of the non-renewable source, such as petroleum. With increasing environmental concern throughout the world and an attempt to reduce the impact caused by polymeric materials new solutions began to be suggested. Some measures began to be taken to try to reduce this excessive amount of waste. Among them are the incineration, recycling, reuse and research directed to the development of biodegradable polymers[1].

Polymers known as Poly(hydroxyalkanoates) - (PHAs) are a family of thermoplastic polymers most widely studied for application as biodegradable materials[2,3]. Among the most studied PHAs is the poly(hydroxybutyrate) - (PHB) homopolymer (Figure 1) that presents thermoplastic properties similar to isotactic polypropylene due to its crystallinity and melting point[4].

The biodegradation process is a biological activity of living organisms to decompose the complex structure of organic compounds to nontoxic products with lower molecular weights. The end products of the biodegradation process can be used as an energy and nutritional source for anabolism of non-producing organisms. The biodegradation of polyhydroxyalkanoates (PHAs) takes place either under anaerobic conditions to produce carbon dioxide (CO_2), water (H_2O) and methane or under aerobic conditions to produce CO_2 and H_2O[5].

The conventional plastic tube used for production of the seedlings is made of polypropylene (PP). The use of plastic tube in the early 80's revolutionized the traditional nurseries of the plastic bag and brought advances in terms of operating income, labor reduction, possibility of automation operations. However, this type of container imposes radical restriction, favoring the appearance of deformation, and these deformations may remain after the nursery stage, reducing the growth of plants in the field. Even with disadvantages the use of polypropylene tubes is widely spread throughout the forest sector. This type of container can reduce 38% consumption of inputs and the period of production the seedlings[6].

The use of biodegradable plastic tubes can reduce the use of bags and PP plastic tubes for some crops. The plastic tube can also reduce planting time, as the seedlings are not taken from them, and are planted directly into the soil. The use of biodegradable tubes could modify the entire chain of production of flower seedlings, since the biodegradable tubes compared to traditional plastic bags take up less space in transport trucks, which would require an adaptation of the structures used today to transport plants ornamentals and flowers[7].

This paper presents the results of a study of the biodegradation in soil the tubes used for planting seedlings.

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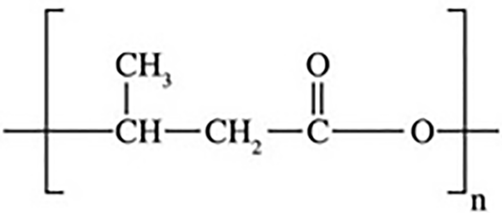

Figure 1. Poly(hydroxybutyrate) chemical structure[3].

For this study was used the polymer Poly(hydroxybutyrate) - PHB pure and two composites, PHB/Wood Flour (80/20) and PHB/Sisal Fiber (80/20).

2. Experimental

2.1. Materials

The biodegradable polymer Poly(hydroxybutyrate) - PHB used in the development of the study was provided by PHB Industrial S/A lote FE-147 with 4% of Valerate. The vegetable fibers used were Wood Flour (mesh 60) and Sisal fiber (3mm in length). The wood flour was provided by Pinhopó Moagem de Madeiras Ltda. and the Sisal fiber by Sisaltec.

2.2. Biodegradation in Soil

The biodegradation test was carried out in soil according to ASTM D 6003 and ASTM G 160 standards[8,9]. The soil used is an organic compound humidified using poultry feces and plant-origin organic materials, produced by Provaso Indústria e Comércio de Fertilizantes Orgânicos S/S Ltda., with the following specifications: Nitrogen (minimum) of 1%; Minimum organic material of 40%; Maximum humidity of 40%; pH 6; C/N maximum of 18/1. For this test, three biodegradation devices were prepared for 30, 60 and 90 days, each device containing 5 tubes of each material (Figure 2). At the end of each test, the degraded samples were carefully washed with water, dried and cooled at room temperature and characterized.

2.3. Characterization

Visual Analysis: After being withdrawn, the samples were washed, dried and visually analyzed for registration and comparison of the materials and the different periods. All test specimens were photographed.

Mass Loss Measurements: For the calculation of mass loss, the samples were weighed before and after the biodegradation test. The calculation was performed using the final mass of dry specimen, as follows (Equation 1):

$$\text{Mass Loss}\ (\%) = \frac{W_0 - W_1}{W_0} \times 100\% \quad (1)$$

Where W_0 e W_1 were initial mass and final mass, respectively.

Flexural Test: The flexural tests were performed on a Universal Testing Machine named EMIC 30kN, in compliance with standard ASTM D 790 M-02[10], with a distance between the grips (flexure three point) of the 60mm, chosen according to the size of the tubes, 5 mm/min speed and a 12,5N load cell.

The tests were performed in tubes before and after the biodegradation in soil test.

Figure 2. Preparation of the devices for biodegradation in soil tests.

3. Results and Discussion

For the manufacture of the tubes, the pure PHB and its composites were extruded and injected. For the extrusion molding, it was used a twin-screw extruder co-rotational made by Extrusão Brasil, with L/D = 40. The injection molding of the pure polymer and its composites used a Romi injection (Prática 130). Figure 3 illustrates the injected plastic tubes.

Figure 3. Photos of tubes (a) PHB, (b) PHB/Wood flour (80/20) and (c) PHB/Sisal fiber (80/20).

3.1. Biodegradation in Soil

3.1.1. Visual Analysis

The appearance of the tested tubes (before and after 30, 60 and 90 days in contact with soil) can be visualized in Figure 4.

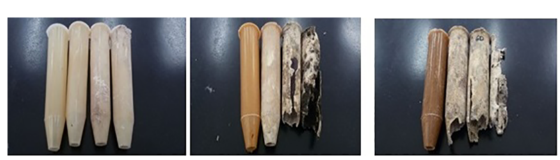

Figure 4. (a) pure PHB; (b) PHB/Wood flour (80/20); (c) PHB/Sisal fiber (80/20).

Analyses were performed by visual observation of the tubes subjected to the test before and after the end of each period. Note in Figure 4 that after 90 days of contact with the soil, the composite tubes cannot be removed entirely from the soil.

Note that the tubes containing wood flour and Sisal fiber are more susceptible to degradation process, although it is possible to see changes in color for tubes with pure PHB polymer over time in contact with the soil.

According Calegari, composites, derived from renewable resources materials are interesting from an ecological point of view. However, the characteristics that make them less harmful to the environment may restrict their applications because they have limited mechanical strength and sensitivity to humidity[11]. For the application in tubes, both limited mechanical and sensitivity to moisture would not be a problem.

3.1.2. Mass Loss

Table 1 shows the average mass loss and the standard deviation for each plastic tube submitted to the test, which was performed with five tubes for each material. For the composites, after 90 days of biodegradation in soil, it was not possible the mass loss calculation because of the material that was lost in the soil, not allowing to identify which sample was, thus, making the mass loss calculation unreliable.

Through the results obtained by the mass loss of the materials under study, it was noted that the materials are biodegradable, both pure and composites. The same does not happen with non-biodegradable polymers such as polypropylene (PP)[12].

In Table 1 we can see that the increase of time in contact with the soil increases the mass loss amount of plastic tube, but not in a proportional way. Note that if 30 days PHB/Sisal Fiber (80/20) had a greater weight loss compared to other materials, 60 days PHB/Wood flour (80/20) had more pronounced degradation compared to the PHB/Sisal fibers (80/20). After 90 days it was only possible to calculate the mass loss of PHB (100), because the reinforced polymer biodegraded much, losing material in the soil, not being possible to identify which sample was.

The difference in the process of biodegradation of the composites can be explained by the fact that the microorganisms act differently according to each type of modifier present in the material[13]. The size of the particles is an important factor in the biodegradation kinetics, being that the larger the contact surface, the greater the speed of biodegradation[14].

In the tested conditions, the composites had greater ease of biodegradation in soil. The optimal time for the plastic tube to remain in the soil will depend on the kind of seedlings which is planted, each type has the ideal time for the root to gain strength and adapt to the soil[6].

Depending on the planted seedlings, the tubes with reinforcement may be more appropriate because they do not require so much time for their roots to gain strength and due to their faster biodegradation, but for plants that require a longer time to gain strength and root, the plastic tube of pure PHB would be the most suitable because it has a slower biodegradation. Therefore, the usage of each plastic tube will depend on the seedlings to be planted.

The three materials under study are suitable for planting seedlings, everything will depend on the kind of seedlings and the time needed for them to gain strength in the soil.

3.1.3. Flexural Test

In the flexural test there were evaluated the flexural modulus. Five experiments were performed for each of the compositions under study (neat PHB, PHB/Wood flour and PHB/Sisal Fiber), and with the values obtained were calculated the arithmetic means and the estimated standard deviation. Table 2 shows these results.

From the results shown in Table 2 it is observed that the pure PHB, before being subjected to the biodegradation test, has a modulus of elasticity around 8GPa. Note that adding 20% of reinforcement in PHB, the modulus of elasticity increases to 8,8GPa and 10,4GPa with the addition of Wood flour and Sisal fibers, respectively.

By comparing the PHB values before and after the degradation test, there is a reduction of value in modulus of elasticity. After 30 and 60 days of biodegradation the values of modules were almost equal, however, after 90 days the modulus value halved. Indicating that the polymer has a slow onset of biodegradation, but becomes accelerated after a period of time.

By comparing the values of the modulus of elasticity of the PHB/Wood flour (80/20) composite before and after being subjected to the biodegradation test notices a great reduction in modulus of elasticity. At the beginning the module was 8,78GPa and after 60 days fell to 1,79GPa, after 90 days of experiment it was not possible to perform the flexural test, because the material was very brittle.

For the composite PHB/Sisal Fiber (80/20) it was observed a large reduction in modulus of elasticity after the degradation test. Before being subjected to biodegradation the module was 10,41GPa with 30 days fell to 1,32GPa, a very high reduction. After 60 and 90 days it was not possible to carry out the flexural test because the materials they were very brittle.

Table 1. Mass Loss.

Materials	Mass Loss (%)		
	30 days	60 days	90 days
PHB	1,45 ± 0,52	4,68 ± 2,23	9,77 ± 2,77
PHB/Wood flour (80/20)	2,05 ± 0,76	25,55 ± 4,05	-
PHB/Sisal fiber (80/20)	11,00 ± 4,37	25,02 ± 8,23	-

Table 2. Results of the flexural test.

Materials	Elastic modulus in flexure (GPa)
PHB	8,11 ± 1,07
PHB 30 days	6,61 ± 1,26
PHB 60 days	6,98 ± 1,26
PHB 90 days	4,13 ± 2,40
PHB/Wood flour (80/20)	8,78 ± 1,20
PHB/ Wood flour (80/20) 30 days	6,17 ± 0,69
PHB/ Wood flour (80/20) 60 days	1,79 ± 0,39
PHB/ Wood flour (80/20) 90 days	-
PHB/Sisal Fiber (80/20)	10,41 ± 1,08
PHB/Sisal Fiber (80/20) 30 days	1,32 ± 0,63
PHB/Sisal Fiber (80/20) 60 days	-
PHB/Sisal Fiber (80/20) 90 days	-

After the biodegradation test, note that the modulus of elasticity of all tubes tested have been reduced, and in the tubes with addition of fibers the biodegradation was more pronounced.

4. Conclusions

In this study it was noted the viability of processing of incorporating vegetables and fibrous reinforcements on a biodegradable matrix by extrusion and injection of poly(hydroxybutyrate) - PHB and PHB/Wood Flour (80/20) and PHB/Sisal fiber (80/20) composites.

Although the materials used in this work are biodegradable, it has been checked a greater mass loss in the composite materials than in the pure polymer, since the water absorption is higher due to the presence of fibers, resulting in greater speed of biodegradation of the plastic tube in contact with the environment.

The ideal plastic tube for plantation of the seedlings has a direct relation to the seedlings to be planted, because each has a specific time for its root to gain strength in the soil permanently, therefore all materials under study are suitable for planting seedlings.

5. Acknowledgments

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