## EFFECT OF ELECTRON BEAM IRRADIATION ON THE BIODEGRADABILITY OF AROMATIC ALIPHATIC COPOLYESTER FILM AND THEIR BLEND WITH CORN STARCH

# Leonardo G. Andrade e Silva<sup>1</sup>, Maira L. Rezende<sup>2</sup>, Derval S. Rosa<sup>2</sup>, Patrícia N. S. Poveda<sup>1</sup>

<sup>1</sup> Instituto de Pesquisas Energéticas e Nucleares, IPEN – CNEN/SP Av. Professor Lineu Prestes 2242 05508-000 São Paulo, SP lgasilva@ipen.br

> <sup>2</sup> Universidade São Francisco Rua Alexandre Rodrigues Barbosa – Centro 13251-900 Itatiba – SP

#### ABSTRACT

Biodegradable and green plastics have been studied in the last years. The aim of this paper is to study the effect of electron beam irradiation on the biodegradability of aromatic aliphatic copolyester film and their blend with corn starch. The samples were irradiated at different doses 10 and 40 kGy in a linear accelerator. The biodegradability of the materials was evaluated by two methods: soil simulated and enzymatic. In the method enzymatic when it was used  $\alpha$ -amylase, the irradiated samples presented faster biodegradation than the references non irradiated. The blend of aromatic aliphatic copolyester (Ecoflex<sup>®</sup>) film in both methods studied.

#### 1. INTRODUCTION

Biodegradable plastics have been considered as a possible solution for the ecological and environmental problems of the huge amount of plastic waste [1].

Some biodegradable polymer materials, for example aliphatic polyesters, are manufactured for commercial purposes. The Ecoflex<sup>®</sup> is aromatic aliphatic copolyester obtained by polymerization of terephthalic acid, adipic acid and 1, 4-butanediol (Fig. 1). This combination (copolyesters containing aliphatic and aromatic monomers) turned out to be the most appropriate characteristics, biodegradability and good material properties [2,3,4].

**Figure 1. Structure of Ecoflex**<sup>®</sup>

The materials Ecoflex<sup>®</sup> and Ecobras<sup>®</sup> are biodegradable plastics and can be used as film products.

Natural polymers, such as starch, are good base materials for producing rapidly degradable plastics, with the advantages that they are inexpensive and are easily digested by microorganisms. Starch is a particularly useful natural polymer because of its low cost and its abundance in nature. The biodegradability of starch in a wide variety of environments makes it applicable for a wide range of products. Starch consists of amylose, a linear alpha-D-(1-4)-glucan, and amylopectin, an alpha-D-(1-4)-glucan that has alpha-D-(1-6) linkages at the branch point [5].

In the present work the radiation the effect of electron beam irradiation on the biodegradability of the aromatic aliphatic copolyester films (Ecoflex<sup>®</sup>) and the blend of aromatic aliphatic copolyester with corn starch (Ecobras<sup>®</sup>) were studied.

## 2. EXPERIMENTAL

### 2.1. Materials

The following polymer films, commonly used as packaging materials were tested:

(A) An aromatic aliphatic copolyester film (Ecoflex<sup>®</sup>), 30  $\mu$ m thickness, produced by BASF. The polymer density (23°C) was 1.25 – 1.27 g/cm<sup>3</sup> (ISO 1183), melt flow rate (2.16 kg/190°C), 2.7 – 4.9 g/10 min (ISO 1133). The film was formed by means of the blowing extrusion with a blowing factor of 2.5.

(B) An aromatic aliphatic copolyester film (Ecoflex<sup>®</sup>), 30  $\mu$ m thickness, produced by BASF. The film was formed by means of the blowing extrusion with a blowing factor of 2.5. Electron beam irradiation at dose of 10 kGy.

(C) An aromatic aliphatic copolyester film (Ecoflex<sup>®</sup>), 30  $\mu$ m thickness, produced by BASF. The film was formed by means of the blowing extrusion with a blowing factor of 2.5. Electron beam irradiation at dose of 40 kGy.

(D) A blend of aromatic aliphatic copolyester with corn starch (Ecobras<sup>®</sup>), 90 $\mu$ m thickness, produced by BASF. The polymer density (23°C) was 1.32 g/cm<sup>3</sup> (ISO 1183), melt flow rate (2.16 kg/190°C), 2.7 – 4.9 g/10 min (ISO 1133). The film was formed by means of the blowing extrusion with a blowing factor of 2.5.

(E) A blend of aromatic aliphatic copolyester with corn starch (Ecobras<sup>®</sup>), 90  $\mu$ m thickness, produced by BASF. The film was formed by means of the blowing extrusion with a blowing factor of 2.5. Electron beam irradiation at dose of 10 kGy.

(F) A blend of aromatic aliphatic copolyester with corn starch (Ecobras<sup>®</sup>), 90  $\mu$ m thickness, produced by BASF. The film was formed by means of the blowing extrusion with a blowing factor of 2.5. Electron beam irradiation at dose of 40 kGy.

To produce granules of Ecobras<sup>®</sup> it was used an extruder machine with thread diameter 45mm and relation length/diameter (L/D) 30:1.

The equipment to blowing extrusion Ecoflex<sup>®</sup> films was an extruder machine with thread diameter 40 mm, relation length/diameter (L/D) 30:1, die opening 1.0 mm and die diameter 100 mm.

To Ecobras<sup>®</sup> films, it was used an extruder machine with thread diameter 45 mm, relation length/diameter (L/D) 30:1, die opening 1.0 mm and die diameter 100 mm.

In the biodegradation tests was used  $\alpha$ -amylase supplied by Novozymes.

## **2.2. Irradiation of samples**

Irradiation of the samples were carried out in air at room temperature using 1.5 MeV electron beam of Dynamitron linear accelerator DC 1500/25 - JOB 188 model at different irradiation doses 10 and 40 kGy at a dose rate of  $11.22 \text{ kGys}^{-1}$ .

## 2.3 Biodegradation tests

## **2.3.1.** Ageing in medium with *α*-amylase

Each sample was placed in a vial filled with 5 mL of 0.05 M phosphate buffer, pH 7.0, containing 1 mg of enzyme, according to the Novozymes protocol. The vials were placed in a water bath at 37°C and at every 48 h the samples were removed, washed with distilled water, wiped dry, and weighed before being returned to the incubation medium. The controls consisted of 5 mL of phosphate buffer without enzyme [6].

## 2.3.2. Ageing in Simulated Soil

The biodegradation tests were done in soil compost containing 23% loamy silt, 23% organic matter (cow manure), 23% sand and 31% distilled water (all w/w). Calcium hydroxide was added to obtain a soil compost of pH 11. The films were weighed and buried in the soil compost, in triplicate. The biodegradability was monitored by calculating the weight loss after periods in soil compost. The buried samples were retrieved, washed with distilled water and dried in open air, before being weighed. The films were buried again in their respective trays after weighing them [7].

## 2.3.3. Simulated Soil analysis

After the preparation of the simulated soil it was analyzed pH, humidity content (%), organic matter (%), total carbon (%) and total nitrogen (%).

## 2.3.3.1. pH analysis

To determine the pH of the simulated soil, it was weighed 2.5 g of soil and 25 ml of distilled water was added. After 30 minutes of agitation in magnetic agitator the mixture 3 pH readings were accomplished while the mixture was being agitated, by using a pHmeter marks Orion, model 520A.

#### 2.3.3.2. Determination of the organic matter (OM) and total carbon content

Organic matter loss method was used by ignition to 550°C. After the humidity determination, the sample contained in porcelain dish was taken to the muffle furnace, and heated up slowly to 550°C, for one more hour. Soon afterwards, the porcelain dish was cooled in desiccator. The result of organic matter was obtained through the Equation 1. The total carbon content was calculated dividing the organic matter values for the factor 1.8.

$$OM = 100x (G_2 - G_3)/M_1$$
(1)

which:

 $G_2$  = crucible mass + sample dries to 100-110°C, in grams;  $G_3$  = crucible mass + ashes to 550°C, in gram and  $M_1$  = initial mass of the samples, in grams.

#### 2.3.3.3. Total nitrogen content

The total nitrogen content was determined by using the Raney alloys method in agreement with the norm ABNT-MB-1167.

#### **3. RESULTS AND DISCUSSION**

In Fig. 2 are shown the results of mass retention of  $\text{Ecoflex}^{\$}$  and  $\text{Ecobras}^{\$}$  films non irradiated and irradiated at 10 and 40 kGy, in presence of the  $\alpha$ -amylase enzyme.

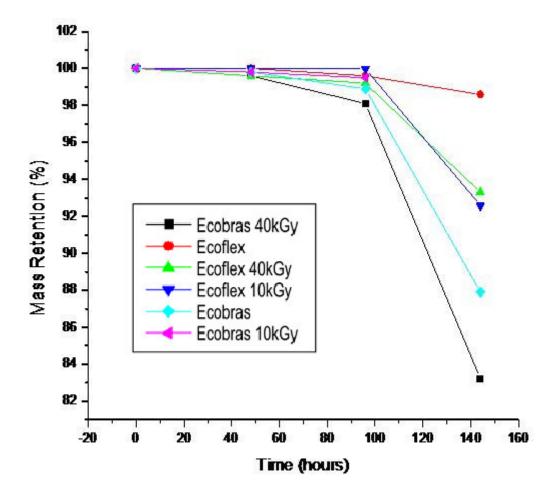


Figure 2. Mass retention of Ecoflex<sup>®</sup> and Ecobras<sup>®</sup> films non irradiated and irradiated at 10 and 40 kGy, in presence of the α-amylase enzyme.

The Ecoflex<sup>®</sup> film samples presented lesser weight loss than Ecobras<sup>®</sup> films. The Ecobras<sup>®</sup> film samples irradiated at 10 kGy had its curve discontinued after 100 hours. This behavior represents a stressed loss of weight. The irradiated samples had a bigger loss of weight than the non irradiated samples.

In Fig. 3 are shown the results of mass retention of Ecoflex<sup>®</sup> and Ecobras<sup>®</sup> films non irradiated and irradiated at 10 and 40 kGy, during ageing in simulated soil.

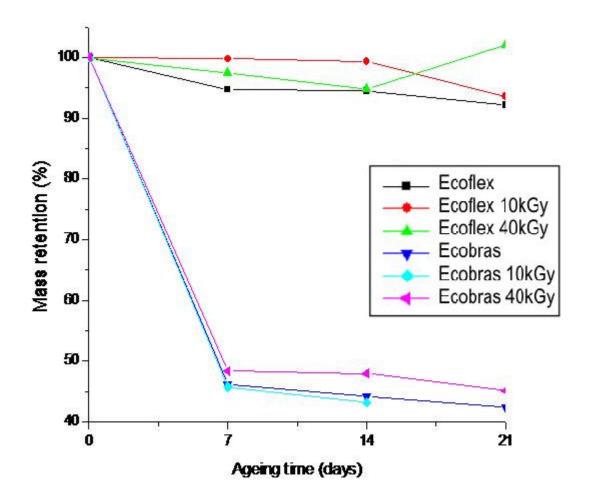


Figure 3. Mass retention of Ecoflex<sup>®</sup> and Ecobras<sup>®</sup> films non irradiated and irradiated at 10 and 40 kGy, during ageing in simulated soil.

The Ecoflex<sup>®</sup> and Ecobras<sup>®</sup> films non irradiated and irradiated at 10 and 40 kGy in the ageing in simulated soil in both cases they did not have any difference among themselves. The Ecobras<sup>®</sup> film samples had a loss of weight bigger than 50% after 7 days.

#### **3. CONCLUSIONS**

In methods of  $\alpha$ -amylase, the irradiated samples presented faster biodegradation than the references non irradiated. The irradiated Ecobras<sup>®</sup> film presented a bigger biodegradability than the Ecoflex<sup>®</sup> film in both methods studied.

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