EFFECT OF IONIZING RADIATION ON NANOCOMPOSITES OF HIGH DENSITY POLYETHYLENE WITH PSEUDOBOEHMITE OBTAINED BY SOL-GEL PROCESS

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ABSTRACT

Nanocomposites are polymeric hybrid materials where inorganic substances of nanometric dimensions are dispersed in a polymeric matrix. The fillers present area of raised surface, promoting better dispersion in the polymeric matrix and therefore an improvement of the physical properties of the composite that depends on the homogeneity of the material. The nanocomposites preparation with polymeric matrix allows in many cases to find a relation enters a low cost, due to the use of minor amount of filler, and a raised performance level. Nanocomposites were obtained with pseudoboehmite synthesized by sol-gel process and high density polyethylene with different concentrations of pseudoboehmite. The aim of this work was to study the effects of ionizing radiation on the properties of the nanocomposites obtained. The nanocomposites were prepared by melt intercalation technique and subsequently, the samples were molded by injection, irradiated and submitted to thermal and mechanical tests. The mechanical properties (impact strength and tensile strength), temperature of thermal distortion (HDT) and Vicat softening temperature of the non irradiated and irradiated nanocomposites were determined. The irradiation doses were of 30, 50 and 100kGy in a gamma cell. The results showed an increase in the values of tensile strength; a decrease in the impact strength and an increase in the temperature of thermal distortion (HDT) evidencing the interaction of nanofiller with the polymeric matrix.

1. INTRODUCTION

Polymeric nanocomposites are a hybrid material where inorganic substances with nanometric dimensions are dispersed in a polymeric matrix [1, 2]. Actually, polymeric nanocomposites have attracted great industrial and scientific interest due to attainment of materials with better mechanical, barrier and flammability properties. These improvements can be obtained when small concentrations of the inorganic filler are added to the polymeric matrix and its layers are exfoliated and well dispersed [1, 3]. Modified organically clays can be efficiently exfoliated in polar polymers using adequate conditions of processing [3, 4].

As the fillers present nanometric dimensions, its surface area is very high promoting better interaction with the polymeric matrix and improvement of the composite physical properties.
that depends on the homogeneity of the material [3]. Due to the polyolefin hydrophobic character, they are more used in the nanocomposites attainment, as polyethylene or polypropylene. The production of exfoliated nanocomposites is more difficult, due to the absence of adequate interactions between the nanofiller surface and the matrix, having in many cases, the necessity of the use of compatibilizing agents [5, 6].

The nanocomposites preparation with polymeric matrix allows in many cases to find a relation between a low cost, due to use of minor filler amount, and one raised performance level, that can result of synergy between the components [7]. Alumina and its derivatives, according to Rabello [8], is widely used in processed materials with inferior properties under 250°C, as additive retarding flame, therefore its structure itself decomposes endothermically, contributing for the decrease of the material temperature during the processing. On the other hand, this type of filler promotes the reduction of the polymers mechanical properties [8].

The sol-gel process has been exhaustively studied to obtain possible the attainment of pseudoboehmites (precursory of alumina) in the form of powder and ceramic staple fibers with small dimensions and elevated homogeneity degree, in reduced temperatures. The pseudoboehmite obtained in this process has nanometrics structure and characteristics [9].

The use of pseudoboehmite, produced by sol-gel process as inorganic phase in a high density polyethylene (HDPE) nanocomposite has as main objective to compare the properties of virgin polymer with the incorporation of these nanofiller and the ionizing radiation effect that can be used used as an alternative in the development of new polymeric materials with better mechanical properties [10]. The most important effect caused in polymers by the ionizing radiation is the main chain scission and appearance of crosslinking [11].

The crosslinking occurs by the recombination of radicals formed by irradiation of polymer and three-dimensional networks are formed. The polymeric chains connect themselves through covalent bond generated chemically or by radiation. In this process, there is an increase of the molecular weight, mechanical resistance, viscosity, reduction of the solubility and a change in the temperature of glass transition in amorphous phase. The crosslinking also depends on the dose, dose rate, concentration, irradiation atmosphere among others variables. However the polymer sensibility to parameter changes depends on the type and size of the chains, as well as the polymer morphology [12-13].

In this work was obtained nanocomposites with different pseudoboehmite concentrations obtained by sol-gel process and HDPE with. The nanocomposites were prepared by fusion intercalation and after irradiated in a gamma cell, characterized by thermal and mechanical tests. The aim of this work is to study the effects of ionizing radiation on the properties of nanocomposites with pseudoboehmite.
2. EXPERIMENTAL

2.1. Materials

The pseudoboehmite was synthesized in the Synthesis and Characterization Mackenzie University Laboratory and HDPE (IA-59U3) was supplied by Brasken S.A. The HDPE is a homopolymer of medium melt flow index (7.3g/10min with 2,16kg/190ºC), additivated to resist UV radiation and indicated for the manufacture to injection molding. It is an atoxic product, that attend to legislation of the “Food and Drug Administration” (F.D.A. - United States of America), used for manufacture of boxes to fruits and vegetables, pallets.

2.2. Methods

2.2.1. Pseudoboehmite obtained by sol-gel process

The reagents used to produce the pseudoboehmite were: aluminum nitrate solution [Al(NO$_3$)$_3$.9H$_2$O] 98 w%, ammonium hydroxyde solution [NH$_4$(OH)] 28w% , and poly(vinyl alcohol) (PVAL) solution 8w% [14].

Aluminum nitrate Al(NO$_3$)$_3$ (370g) was mixed with poly(vinyl alcohol) (200g) during thirty minutes, using a magnetic mixer (Fig. 1-A) finally [NH$_4$(OH)] (312g) cooled at -9ºC was dripped on the obtained mixture. The initial temperature of mixture was 26ºC and at the end - 6ºC.

After total precipitation and aging per 1 week in low and controlled temperature at 12ºC (Fig. 1-B), the solution was filtered in Buchner funnel and washed with 1L of distilled water followed with 500mL of propanone (Fig. 1-C).

Figure 1. (A) Aluminum nitrate and poly(vinyl alcohol) mixture; (B) Precipitated pseudoboehmite solution before filtration process; (C) Precipitated pseudoboehmite after aging and filtration.
2.2.2. Nanocomposites Preparation

Two HDPE/pseudoboehmite nanocomposites in w1% and w3% of pseudoboehmite was prepared by melt intercalation technique.

Initially the pseudoboehmite and the HDPE were mixed manually and the nanocomposites were prepared by hot melting in a calender of parallel coils (Mecanoplast mark, C400-3 model) (warm 155°C). The nanocomposites in the deformed plate form were obtained. The plates obtained were grinded in a mill (Seibt mark, model 270).

Due to good dispersion the nanocomposites were directly molded by injection, using injector Romi Primax model 65R, with 134cm³ of the maximum capacity of injection and screw of 35mm, to obtain the specimens in accordance with the standards.

2.2.3. Samples irradiation

The samples were exposed to gamma rays from Cobalt-60 at doses: 30, 50 and 100kGy.

2.2.4. Nanocomposites characterization

The samples were characterized by: tensile strength (ASTM D-638-01); impact Izod strength (ASTM D-256); thermal distortion temperature-HDT (ASTM D-648) and soften Vicat point (ASTM D-1525-81).

3. RESULTS AND DISCUSSION

3.1. Tensile Strength

The tensile strength was determined for HDPE and nanocomposites reinforced with 1 and 3% of pseudoboehmite, irradiated and non irradiated samples. Fig. 2 and 3 present the results of tensile strength.
The results show that:
- The presence of pseudoboehmite promotes a significant increase in the tensile strength and, consequently a decrease in the deformation of the system;
- The increase in the tensile strength and the decrease in the elongation of the nanocomposites obtained indicate that nanofiller reinforce the polymeric material;
- The HDPE tensile strength increased with the radiation dose. The explanation of this increase, proceeding from the exposition of the materials to the radiation, can be attributed to
the two main processes [15-16]: increase in the crystallinity and formation of crosslinkings in the crystalline and amorphous regions;

- The ionizing radiation increases the resistance of the composite containing pseudoboehmite until the dose of 50kGy and decrease occurs in the dose 100kGy.

- Probably the nanofiller presence intervenes with the crystallinity of the material becoming the material more resistant to radiation;

- The elongation of the nanocomposites decrease with the increase of dose radiation.

3.2. Impact Strength Izod

Fig. 4 presents the results of impact strength for HDPE and HDPE/pseudoboehmite nanocomposites.

![Image of bar graph showing impact strength comparison](image)

**Figure 4.** Impact Strength of HDPE and the nanocomposites reinforced with 1 and 3% of pseudoboehmite, irradiated and non irradiated samples.

The results show that:

- The impact strength values decrease with the pseudoboehmite concentration in the nanocomposites;

- To HDPE the impact strength decrease with the radiation dose, while to nanocomposites HDPE/pseudoboehmite the impact strength decrease with the irradiation until 50kGy. To 100kGy dose the impact strength of the nanocomposite increase;
- This results are in accordance with tensile strength tests.

3.2. Thermal Distortion Temperature-HDT and Soften Vicat Point

Fig. 5 and 6 present the results of HDT and soften Vicat point test.

![HDT and Soften Vicat Point](image.png)

**Figure 5.** HDT of HDPE and the nanocomposites reinforced with 1 and 3% of pseudoboehmite, irradiated and non irradiated samples.

**Figure 5.** Soften Vicat point of HDPE and the nanocomposites reinforced with 1 and 3% of pseudoboehmite, irradiated and non irradiated.
The results show that:

- The addition of pseudoboehmite promote an increase in the HDT of the nanocomposites, while the soften Vicat point, practically do not altered;

- The increase in the HDT of the nanocomposites obtained indicates that nanofiller, probably, increase the crystallinity degree.

- The HDT increase with the radiation dose, while the soften Vicat point, practically do not altered.

4. CONCLUSIONS

The results had allowed the following conclusions:

- The radiation promotes changes in the macromolecular structure of polyethylene and, consequently, in its properties;

- The increase of the radiation dose corresponds to increase in the tensile strength and a decrease in the impact strength;

- The increase of the radiation dose promotes an increase in the thermal stability of the polyethylene;

- These results can be attributed to the crosslinking in the polymeric matrix;

- The presence of pseudoboehmite promotes an increase in the thermal stability and tensile strength; and promotes a decrease in the elongation and impact strength of the obtained nanocomposites, indicating that the nanofiller increases its crystallinity degree;

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