

PERMEABILITY AND MECHANICAL PROPERTIES OF PCL FILMS

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This work reports the influence of radiation on the barrier properties of water vapor permeability (WVP), solubility and mechanical properties (puncture test) of poly(ϵ -caprolactone) – PCL films. These films were prepared by casting with 1g of PCL/100mL of acetone. The mechanical properties were determined by the puncture test and the WVT was determined according to ASTM E96-80 [1].

Introduction

Nowadays much attention has been given to biodegradable polymers made from renewable sources, such as polycaprolactone (PCL) [2], which is a biodegradable, biocompatible and non-toxic synthetic aliphatic polyester [3]. Aliphatic polyesters can be made from petroleum and their manufacturing cost is comparable to that of conventional plastic resins, which makes them competitive in commercial markets [2]. The objective of this work was to study the solubility, water vapor permeability and the mechanical properties of.

Experimental

Materials and methods - The following materials were used to prepare film: Acetone (synth) and poly(ϵ -caprolactone) – PCL (solway) and polyvinyl chloride (PVC) film for domestic use (theoto S.A).

Sample preparation - Films of PCL were obtained by casting from solutions of PCL (1.0g) in acetone (100mL) on a glass plate. The solution was heated to 70°C to dry and then a transparent film was formed. Afterwards the PCL films were irradiated in ⁶⁰Co with 700.000 Ci activities, operating at 5.0kGy.h⁻¹ (dose rates of 2; 5 and 10kGy).

Water vapor transmission (WVT) - was determined according to ASTM E96-80 [1], modified by Gontard *et al.* [4]. A container with silica gel was closed with a sample of edible film firmly fixed on top. Then the container was placed in a dessicator with distilled water at a temperature of 25.0°C. The films were weighed daily on a Mettler analytical balance for 10 days. Water vapor transmission (WVT) was calculated according to equation 1:

$$WVT = \frac{w \times x}{A} \quad (1)$$

where WVT is Water Vapor Transmission (g H₂O. mm.cm⁻²), x is the average thickness of the film (0.070 ± 0.012 mm) and A is the permeation area (23,76 cm²). Water vapor transmission rate (WVTR) was calculated according to Eq. (2):

$$WVTR = \frac{w \times x}{t \times A} \quad (2)$$

Where WVTR is water vapor transmission rate (gH₂O mm h⁻¹ cm⁻²), and the term x/t was calculated by linear regression from the points of weight and time, during constant rate period. The tests were carried out in duplicate.

UV-Vis spectroscopy - UV-Vis spectra were obtained on a UV-Vis spectrometer Shimadzu UV-2401 model at a range 200-800nm.

Mechanical properties – Puncture tests were performed in a texturometer TA.XT2i (SMS, Surrey UK), with a probe of diameter of 3.0 10⁻³. The samples were fixed in a mold of 3.3 10⁻² m diameter. The deformation at puncture was calculated in accordance with the expression (3):

$$\frac{\Delta L}{l_0} = \frac{(\sqrt{D^2 + l_0^2} - l_0) \times 100}{t \times A} \quad (3)$$

Where D is displacement of the probe leads until at the rupture taken on the curves of force versus displacement, with the use of the program texture expert 1.15 (stable systems micron) and l₀ is the initial length of the film.

Solubility in water – Samples with the 4.0 10⁻² m wide were immersed in distilled water at room temperature and then were left under constant agitation for 24h. After that, the films were dried to constant weight at

50°C. The solubility of each film can be determined according to the expression (4):

$$\%MS = \frac{M_i - M_f}{M_i} \times 100 \quad (4)$$

Where %MS is percentage of soluble mass, M_i is initial mass of the film and M_f is final mass of the film.

Results and discussion

PCL films were colorless, transparent and 0,07mm thick. The results of water vapor transmission rate (WVTR) on the PCL are shown in Table 1. Films were irradiated at low doses of 2,5 and 10 kGy, commonly used in food irradiation. For comparison, an example of PVC film was included. WVT of polyester films, at 25°C, varied linearly with the dose, with very good correlation coefficient ($R^2=0.98$) Figure 1.

Table 1 – Water vapor transmission rate (WVTR) of the films

Film	Dose (kGy)	WVTR ($\text{gH}_2\text{O} \cdot \text{mm} \cdot \text{h}^{-1} \cdot \text{cm}^{-2}$)
PCL	0	3.82×10^{-9}
PCL	2	3.71×10^{-9}
PCL	5	3.05×10^{-9}
PCL	10	5.31×10^{-9}
PVC	-	0.19×10^{-9}

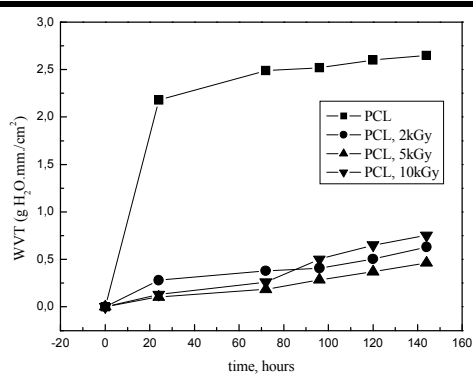


Figure 1 – Barrier properties of PCL films (0, 2, 5 and 10 kGy). WVP = water vapor permeability.

The influence of dose on mechanical properties of PCL films is showed in figures 2 and 3. In the doses of 2 and 5 kGy, the puncture force increased with the increasing of dose.

After irradiation, the barrier properties [water vapour permeability (WVP)] and mechanical properties of the films were improved due to chemical reactions among polymer molecules. The UV-Vis spectrum of PCL films (10kGy) showed an increase of the absorption intensity in the range of 260-340 nm, related to carbonyl groups. Campos and Franchetti [3] also observed a significant increasing of the absorbance intensity of carbonyl groups (260-340nm) and the

presence of three sharp bands related to the absorption of different types of carbonyl groups of biotreated PCL films, suggesting the degradation at this level dose.

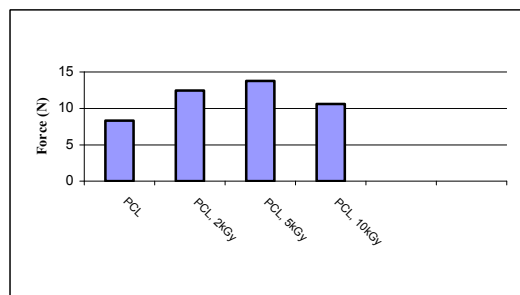


Figure 3 – Puncture force of the films

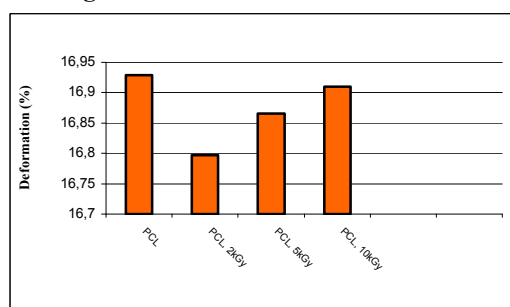


Figure 4 – Puncture deformation of the films

Conclusions

All in all PCL films produced by casting process are transparent enough, with good mechanical properties and potential application for food packaging. Their mechanical characteristics, which are similar to those of synthetic films used in supermarkets (poly vinylchloride (PVC)) are influenced by irradiation dose: An increase in radiation dose resulted in a considerable increase in the puncture force of the films, whereas WVTR can be significantly reduced by low doses of gamma radiation.

Acknowledgements

The authors are thankful to the Fundação de Amparo a Pesquisa do estado de São Paulo (FAPESP) and the Conselho Nacional de Pesquisas (CNPQ) for financial support.

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