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Nuclear Instruments and Methods in Physics Research B 191 (2002) 675–679

NIM B
Beam Interactions
with Materials & Atoms

www.elsevier.com/locate/nimb

Thermal analysis evaluation of mechanical properties changes promoted by gamma radiation on surgical polymeric textiles

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Abstract

The large number of surgical operations with post-operative infection problems and the appearing of new infectious diseases, contribute to the development of new materials in order to answer the needs of health care services. This development must take into account the modifications promoted by sterilisation methods in materials, namely by gamma radiation. The differential scanning calorimetry (DSC) and thermogravimetry (TGA) techniques show that a nonwoven and a laminate textiles maintain a good molecular cohesion, do not showing high levels of degradation, for gamma radiation dose values lower than 100 kGy in nonwoven and 200 kGy in laminate materials. The tensile strength and the elongation decrease slowly for the nonwoven textile and decrease faster for the laminate textile for 25 and 80 kGy absorbed dose. This paper shows that the DSC and TGA techniques can be helpful for the prevision of mechanical changes occurred in the materials as a consequence of the gamma irradiation. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Polymeric surgical textiles; Thermal analysis; MCNP; Radiosterilisation

1. Introduction

Nowadays, the large number of surgical operations with post-operative infection problems (in Europe, in average between 5% and 10%) and its tendency to increase, as well as the appearing of new infectious-contagious diseases, contribute to accelerate the development of new materials in

order to answer the requirements and specifications of health care services.

Although, the selection of the most appropriate surgical protection is very complex as a consequence of the different parameters that have to be conjugated. Surgical exigencies, type of material and their related properties (mechanical, barrier, comfort, etc.) and the changes promoted by the lethal agent in the materials during the sterilisation process, are parameters that must be taken into account.

Some polymeric based textiles, mainly nonwovens and laminates, are showing to have the

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adequate properties, essentials for the manufacturing of disposable surgical protection clothing [1].

Ionising radiation (gamma and electron beam radiation) is often used for the sterilisation of polymeric based disposable medical devices. Nevertheless this sterilisation can induce some changes in their behaviour and some of them could restrict their use for the defined applications. The mechanical properties (tensile strength and elongation) are commonly regarded as a rule of quality and they must be evaluated before and after sterilisation treatment. These data should be included in the assessment of the quality of a textile material.

This work is part of a study of the influence of ionising radiation (gamma and e-beam radiation) on the properties and performance of disposable materials and products for hospitalar protection. In this paper will be presented the results obtained with two textiles, a nonwoven and a laminate, irradiated with gamma radiation at the Co-60 Portuguese Gamma Facility (UTR) [2].

According to the standards [3], this kind of research requires the irradiation of lots of samples to a maximum acceptable dose for the products.

Knowing materials behaviour profile with radiation, in what concerns to a few properties directly dependent of the molecular organisation, it is possible to foresee what will happen with other essential properties. This is the case of melting temperature (T_m), melting enthalpy (ΔH_m) and the temperature at which the materials begin to degrade (T_{deg}). These properties can be measured by thermal analysis techniques (e.g. differential scanning calorimetry (DSC) and thermogravimetry (TGA)) [4].

Both, DSC and TGA techniques, require only few milligrams of material to perform a thermogram. Then, instead of irradiate a lot of samples in their final form, it is possible to irradiated little samples of material at several doses. These procedures allow a quick knowledge of material behaviour and the identification of the critical doses.

2. Experimental details

FAPOMED SA, Felgueiras, Portugal processed textile samples of nonwoven and laminate mate-

rials. These textiles have the following composition and density:

Nonwoven: (55% cellulose, 45% polyester); $\rho = 0.17$.

Laminate: 1st layer: 0.02 kg low density polyethylene (LDPE) film; 2nd layer: nonwoven (70% viscose, 30% polyester); $\rho = 0.14$.

Eight samples ($0.1 \times 0.1 \text{ m}^2$) of each material were packed in a proper bag, evacuated and sealed.

Two batches of 25 gowns manufactured from each material were packed in the same conditions and were accommodated in pasteboard boxes of $0.4 \times 0.4 \times 0.4 \text{ m}^3$ (standard boxes of UTR).

The samples were irradiated at the UTR. The small ones were irradiated in very well characterised steady positions, with doses of 25, 80, 150, 200, 250, 300, 350 and 400 kGy.

The big samples (gowns) were irradiated in the pasteboard boxes in dynamic process for a final dose of 25 and 80 kGy.

The irradiations control parameters were monitored with PMMA dosimeters (Amber Perspex [1, 30 kGy] and Red Perspex [1, 50 kGy]) [5], being the dose values calculated by Monte Carlo N-Particle Transport Code (MCNP code) [6–8], which allows the planning of the irradiation.

The small samples irradiated with doses between 25 kGy and 400 kGy and a nonirradiated one (blank) were analysed by DSC and TGA techniques. These assays were performed with equipment from the TA Instruments, at the Polymer Characterisation Laboratory of ITN, Sacavém, Portugal.

DSC and TGA thermograms were obtained under the same experimental conditions: nitrogen flux of 0.01 l/s and with standard heating programs for polymer analysis.

Concerning the big samples, the textiles mechanical properties were measured and statistically analysed at UM – Department of Textile Engineering, Guimarães, Portugal [9], before and after irradiation at 25 and 80 kGy.

For the mechanical tests it was used a pneumatic dynamometer from Hounsfield Equipment (charge cell: 20–1000 N). The tests were realised in agreement with the standard guidelines for medical textiles [3,10].

3. Results and discussion

For the steady and dynamic irradiations the provisions of dose performed by MCNP code show a maximum difference of 2%. The dose uniformity for these irradiations was $U(25 \text{ kGy}) = 1.11$ and $U(80 \text{ kGy}) = 1.18$.

Figs. 1 and 2 show the variation of melting enthalpy (ΔH_m) and melting temperature (T_m) with the absorbed gamma radiation dose, for nonwoven and laminate textiles. Fig. 3 shows the variation of temperature of beginning of degradation (T_{deg}), of nonwoven and laminate textiles, with the absorbed gamma radiation dose.

For evaluation of the relevant mechanical properties purpose, in different irradiation states and fibre material orientation (MD – machine direction; CD – cross direction of the material) 20 measurements of each material were performed.

The statistical study concerned the test of variance (F-Snedecor) and the test of the means (*t*-Student-Fisher), with 0.05 level of significance to the both distributions:

Tables 1 and 2 show the effects of two gamma radiation doses, 25 and 80 kGy, on the mechanical properties, tensile strength and elongation, of nonwoven and laminate materials, respectively, taking nonirradiated material as reference.

Figs. 1 and 2 suggest that the materials maintain a good molecular cohesion in the range of

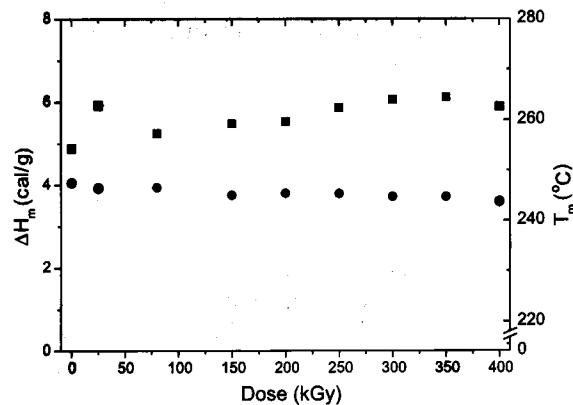


Fig. 1. Variation of melting enthalpy (ΔH_m : ■) and of melting temperature (T_m : ●), with the absorbed gamma radiation dose, for nonwoven material.

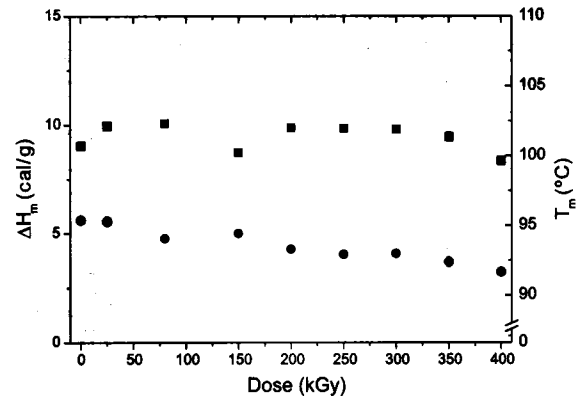


Fig. 2. Variation of melting enthalpy (ΔH_m : ■) and of melting temperature (T_m : ●), with the absorbed gamma radiation dose, for laminate material.

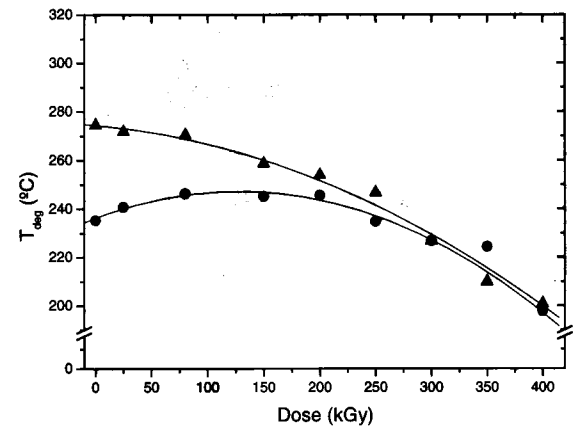


Fig. 3. Variation of degradation temperature (T_{deg}), of nonwoven (▲) and laminate (●), with the absorbed gamma radiation dose.

dose values considered. Fig. 3 do not show the existence in the materials of high levels of degradation, for doses values lower than 100 kGy for nonwoven and 200 kGy for laminate material.

Nonwoven is a cellulose based material. As it is known, cellulose is a preferential degrading material under gamma influence [11]. Being cellulose the backbone of the textile is expected to see a rapid degradation of the material with the increasing dose (see Fig. 3). Nevertheless, polyester is a preferential cross-linking material [11]. The

Table 1
Effects on the tensile strength and elongation of nonwoven material, with a dose of 25 and 80 kGy (fibre direction dependence)

Gamma radiation dose	Mechanical property (average values)							
	F_{\max} (N)		E_{\max} (%)		F_{break} (N)		E_{break} (%)	
	MD	CD	MD	CD	MD	CD	MD	CD
0 kGy	139.70	41.23	24.69	83.95	131.88	40.41	25.61	84.73
25 kGy	135.99	38.61	24.87	81.35	130.80	38.22	25.80	81.98
F	2.80	1.35	1.03	1.27	3.72	3.80	1.42	1.11
<i>t</i>	2.92	5.35	0.56	2.97	0.50	3.61	0.54	2.88
Modifications	S	S	NS	S	S	S	NS	S
80 kGy	136.03	41.75	23.77	82.55	119.90	39.91	25.28	83.45
F	4.31	2.52	2.48	1.33	3.63	1.79	1.09	1.65
<i>t</i>	1.35	0.66	1.91	1.29	2.64	0.48	0.70	1.14
Modifications	S	NS	NS	NS	S	NS	NS	NS

S: Significant; NS: Non-significant.

Table 2
Effects on the tensile strength and elongation of laminate material, with a dose of 25 and 80 kGy (fibre direction dependence)

Gamma radiation dose	Mechanical property (average values)							
	F_{\max} (N)		E_{\max} (%)		F_{break} (N)		E_{break} (%)	
	MD	CD	MD	CD	MD	CD	MD	CD
0 kGy	59.56	9.18	8.94	19.06	57.64	7.07	9.45	22.76
25 kGy	51.68	8.43	7.22	14.56	51.01	7.09	7.54	21.21
F	3.96	2.05	3.46	4.64	5.93	1.29	3.06	2.50
<i>t</i>	12.29	3.05	9.06	4.78	8.34	0.05	7.54	1.41
Modifications	S	S	S	S	S	NS	S	S
80 kGy	51.35	11.13	6.57	14.29	47.15	10.47	6.99	16.15
F	1.11	1.64	1.74	16.65	3.32	1.47	2.42	10.33
<i>t</i>	8.13	5.94	8.82	3.87	6.00	10.66	7.37	4.79
Modifications	S	S	S	S	S	S	S	S

S: Significant; NS: Non-significant.

increase of reticulation promoted in polyester acts in the sense of the increase of mechanical resistance and the degradation occurred in cellulose acts in opposite sense. So, under gamma irradiation, will be expected that the internal mechanical resistance of the textile does not vary substantially (see Fig. 1).

In what concerns with tensile strength and elongation is expected that the tensile strength will decrease slowly as dose increase, as so the elongation (depending on the fibres orientation), as can be observed in Table 1. In effect, according to these data the maximum variation for tensile strength is

10% (for F_{break} , MD direction and 80 kGy conditions) and 3% for the elongation (for E_{break} , CD direction and 25 kGy conditions).

Laminate is a double layer textile. Both layers have preferential cross-linking polymers in their composition: LDPE in the first layer and polyester in the second. Viscose is a cellulose derivative and is a preferential degrading polymer [11]. The behavior of the second layer under gamma irradiation is expected to be similar to the nonwoven cellulose based textile under the same conditions. But in the laminate textile, the first layer will increase the initial resistance of the material as a

result of the LDPE reticulation. So, the viscose degradation effects are opposite to the polyester and LDPE reticulation effects. Due to this the textile will suffer a small improvement of its thermal resistance till a dose near 200 kGy, from where LDPE and polyester begin to degrade too (see Fig. 3). After that dose value, is expected the loss of textile internal mechanical resistance (see Figs. 2 and 3).

In the case of tensile strength and elongation is expected that tensile strength will decrease as dose is increasing (depending on the 2nd layer fibres orientation) and the elongation will be more seriously reduced as it was in nonwoven.

In effect, according to the data shown in Table 2 the maximum variation for tensile strength is 22% (for F_{break} , MD direction and 80 kGy conditions) and 36% for the elongation (for E_{max} , MD direction and 80 kGy conditions).

Even that available data suggest that both textiles maintain their mechanical and thermal stability in the range of dose values usually applied to medical devices sterilisation (15–40 kGy), this study must be complemented with data from other essential materials properties (e.g. barrier properties).

4. Conclusion

Gamma radiation can promote changes in the molecular structure of the materials, leading to an improvement or degradation of their properties. Thermal techniques as DSC and TGA, may be used for evaluation of some properties that are directly dependent of materials molecular organisation. In this way it is possible to use thermal analysis to predict the behaviour of other essential properties, for instance mechanical properties

changes occurred in studied textiles due the gamma irradiation.

Acknowledgements

The work was developed under contract ADI/Steritex Project.

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n dependence)

h)

CD

84.73
81.98
1.11
2.88

S

83.45
1.65
1.14

NS

n dependence)

(%)

CD

22.76
21.21
2.50
1.41

S

16.15
10.33
4.79

S

d 80 kGy condi-
n (for E_{break} , CD
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polymers in their
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