Condensed Matter Physics

Fernanda Margaça

The Group's main field of research is the development and characterisation of materials with new or improved properties. To this end, radiation is used as a tool to investigate the structure and to induce structural modifications in particular samples. Special polymeric materials have been investigated in collaboration with groups from the Universities of Aveiro and Coimbra, Laboratoire Léon Brillouin (CEA-CNRS-Saclay), KFKI, Budapest, and the Budapest Neutron Centre. During 2007 the main effort was put into the characterisation of hybrid materials and co-polymers prepared by gamma irradiation using the ^{60}Co source of UTR.

The systems studied were: (i) hybrids prepared from mixtures of a polymer (PDMS) and various alkoxides, with emphasis in the elemental and topographic characterization and RBS analysis of various samples prepared using irradiation methods; (ii) development of new copolymers (HEMA grafted on LPDE thin films) suitable for bioapplications. Here, sample preparation conditions were correlated to the grafted material structure and the hydration level achieved by the final product, and toxicity studies were conducted. Tests of Biocompatibility (haemolysis and tromboresistance) are also being carried out. Work on these systems is resumed in an MSc thesis defended in 2007and a PhD thesis completed in 2007.

The Group has also been active in the area of hardware and software instrument development, with emphasis in the design, construction, and testing of systems and components for neutron beam work. Shortage of resources, both human and financial, is preventing the proper development of the activity in this area.

As concerns human resources both the staff and the students were recently significantly reduced. Frederico Carvalho, senior researcher and founder of the group, had reached retirement age in 2006. Although he continues to contribute to current activities the long due implementation of a plan of selected recruitment of scientific personnel has become more pressing.

This situation has been aggravated by the exit of two students in the end of the first half of 2007. David Silva left as he found a better professional position outside of the Institute and the MsC student Susana Gomes left at the end of the FCT grant, after having finished her Master thesis.

As concerns the financial resources, the neutron scattering instruments situation is also particularly difficult. This type of instruments installed at reactors require a set of equipments and components that are, generally expensive items, to be able to tailor the neutron beam, eliminate the undesired background and collect the data, in a suitable and efficient way. This becomes particularly critical when the source has low flux. During 2007 the core of the Portuguese Research Reactor, RPI, has been converted from HEU to LEU. It is predicted that this change will have a significant impact in both the total thermal neutron flux and on the signal to noise ratio. Work will proceed, in the instruments installed at RPI, to determine how far these are affected.

The Group’s work has been supported by funds from FCT, IAEA, ITN and income from services.

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[1] Retired
[2,3] Left in end of June

Novel Way to Control PDMS Cross-linking by γ Irradiation

F.M.A. Margaça, S.R. Gomes, A.N. Falcão, L.M. Ferreira and I.M.M. Salvado

Objectives

The purpose was to investigate the properties of the cross-linked PDMS network obtained from the gamma-irradiation of a mixture of PDMS and the silicium alkoxide TEOS.

Results

Samples were prepared by γ-irradiation of a mixture of polydimethilsiloxane, PDMS, and tetraethylortosilicate, TEOS, in varying concentrations. The obtained materials are monolithic, flexible and transparent. It was confirmed that, for this type of mixture, the mass of the irradiated materials approach that of the PDMS used in the preparation stage as the drying stage proceeds [1]. Thus, most of TEOS evaporated and only a very small part was retained in the dried sample. X-ray diffraction was carried out in dried samples and showed that they are amorphous. The thermal behaviour has also been studied by using Differential Scanning Calorimetry (DSC) and Thermal Gravimetric Analysis (TGA).

Fig.1. Thermograms of xPDMS (1-x) TEOS samples.

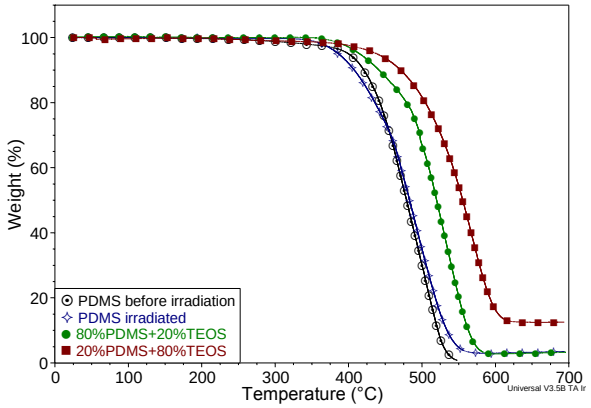
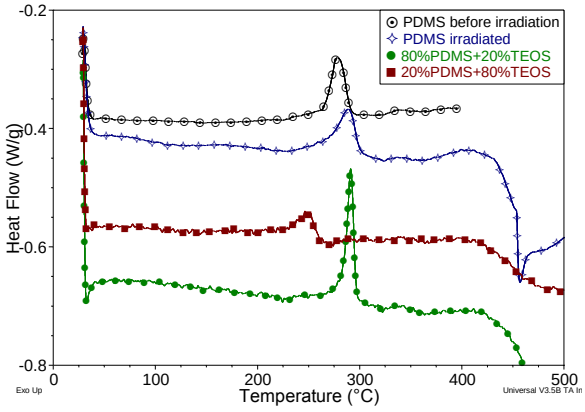
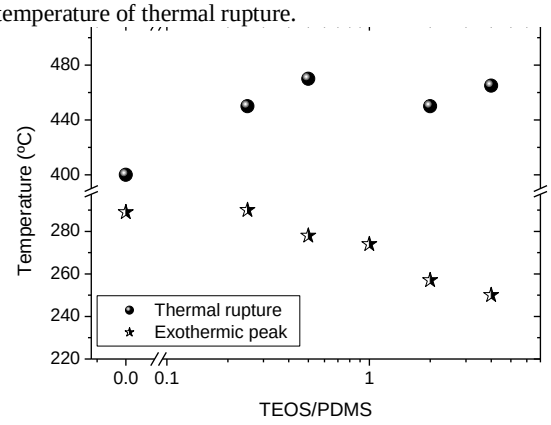


Fig.2. DSC plots for the same set of samples.



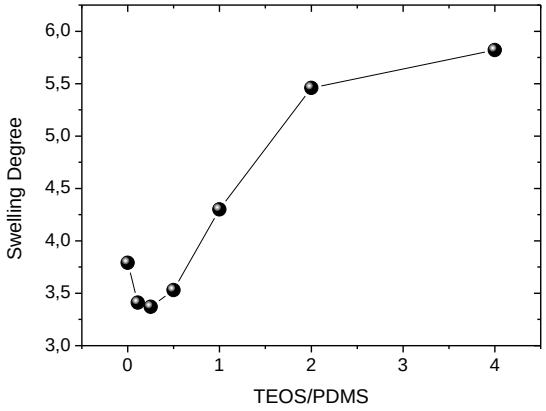
DSC and TGA curves show that the thermal behaviour depends on the presence of TEOS.

Fig.3. Variation of the exothermic peak position and temperature of thermal rupture.



The temperature necessary to promote the thermal rupture increases, by 50 ºC, when TEOS is used. As TEOS evaporates almost completely during the drying stage, the increase in this temperature seems to be related to the increasing strength of the PDMS cross-linked network. The exothermic peak, that is associated to chain ordering, occurs at T < 300 ºC and its value decreases as the TEOS content increases. This shows that TEOS facilitates the spatial ordering of PDMS chains in the sample.

Fig.4.Variation of the swelling degree with TEOS.



Although most of TEOS evaporates after precursors irradiation, its presence has a major impact in thermal properties as well as in the swelling properties of the material prepared by γ-irradiation.

The latter showed that PDMS cross-linked network strongly depends on the TEOS content present during the irradiation process. This provides a new way to control PDMS cross-linking by γ-irradiation.

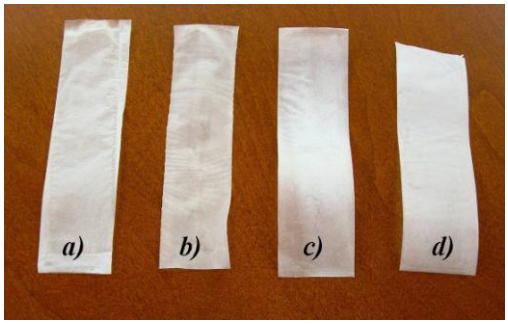
Published Work

S.R. Gomes, F.M.A. Margaça, L.M. Ferreira, I.M. Miranda Salvado, A.N. Falcão, *Nuclear Instruments and Methods B* **265** (2007) 114-117.

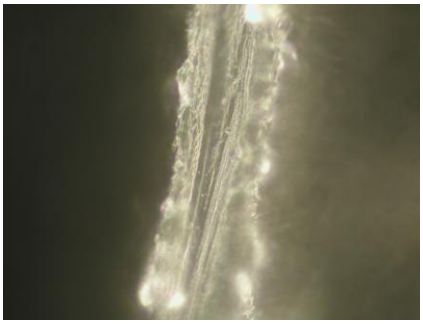
Hemocompatibility Evaluation of LDPE Based Copolymeric Films Obtained by Gamma Irradiation

L.M.Ferreira, A.N. Falcão, M.H. Gil

Using 2-hydroxyethyl methacrylate (HEMA) as grafting monomer, we have optimized the preparation of new grafted copolymeric Low Density Polyethylene (LDPE) based films by gamma radiation, using a ^{60}Co source. The physical and chemical properties observed in these new copolymeric films show that they are very promising materials for multiple bioapplications. In this way we started the hemocompatibility evaluation *in vitro* of the films according to standards ISO 10993-4 and 10993-5. Two types of blood interactions are being studied: hemolysis and thrombogenicity. This work is being conducted in collaboration with the *Laboratório Nacional de Investigação Veterinária* (LNIV).
According to ASTM F 756-00 standard, materials can be classified according to their hemolytic index. When this parameter is between 0 and 2%, the material is classified as "nonhemolytic". If hemolysis reaches values between 2 and 5%, the material is classified as "slightly hemolytic". However, when materials induce hemolysis values upper than 10%, they are classified as "hemolytic". Although it is not possible to define an absolute scale for this effect, by definition a blood compatible material must be "nonhemolytic". The results obtained for the new PE-g-HEMA films reach a maximum of 2% of hemolytic effect, which classify them as "nonhemolytic" and therefore suitable for direct contact with blood. A detailed assessment of thrombogenicity is still being done.
Future work involves the conclusion of thrombogenicity study and the evaluation of the general citotoxicity of the films. The final aim of the research under way is the registration of a National Patent for this new polymeric material.



PE-g-HEMA grafted films *a)* 7,5 kGy, 237%; *b)* 9 kGy, 403%; *c)* 10,5kGy, 166%; *d)* 12 kGy, 163%



PE-g-HEMA cross-section view

Polymer Characterization Laboratory

The *Polymer Characterization Laboratory* (LCP) is a multifunctional unit located at the Physics Department. Initially created in straight collaboration with IAEA, as a tool of the Portuguese Cobalt-60 Facility (UTR) for quality control of the industrial irradiation services of polymer made devices (electric insulators, medical devices, etc), gradually began to increase its R&D activity as a result of the increasingly collaboration with the Dep. of Chemical Engineering, Faculty of Sciences and Technology of Coimbra University. The installation of new experimental resources for materials testing is in course. This new laboratory, *Laboratory of Materials Mechanical Tests,* is being equipped with a *Universal Mechanical Assays Machine* and a *Thermal Press*.

Neutron Spectrometers at the Portuguese Research Reactor

F.M.A. Margaça, A.N. Falcão, J.S. Neves, C.M.M. Cruz, D.M.P.S. Silva, F.G. Carvalho

The situation of the diffractometers ETV and DIDE remained unchanged during 2007. As concerns the small angle neutron scattering instrument, EPA, test work on the measurement of signal-to-noise ratios was halted due to the malfunctioning of the water pump in the circulation system of the neutron diffuser in the beam tube D2. The pump was dismantled and proved to be irreparable. It was replaced by a new one that had to be acquired. Meanwhile the RPI core has been converted from HEU to LEU. It is predicted that this change will have a significant impact in both the total thermal neutron flux and on the signal to noise ratio. Work to determine how far these are affected is underway.