



Surface modification of polyurethane and silicone for therapeutic medical technics by means of electron beam

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ABSTRACT

Surface modification technologies are gaining growing acceptance for treatment of implant materials to enhance biocompatibility. Our examinations focus on polyetherurethane and silicones, two typical flexible implant materials, which we have modified by non-thermal electron beam processing. Advantages of this method are the adjustable degree of modification as well as the simultaneous sterilizing effects. The polymer surfaces were characterized with regard to wetting behavior, surface energy, chemistry and morphology. The cell adhesion was examined too. The results reveal that the electron beam is a useful tool for surface modification of polymers.

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1. Introduction

Medical products that are used for applications on or in the human body must be made of materials which are fully compatible with the surroundings at the point of application. This puts high requirements on materials, and on polymers in particular, many of which are not sufficiently biocompatible in their as-produced state [1].

In order to enhance the biocompatibility, and in particular the biofunctionality of materials used for implants, increasing use is being made of surface modification techniques [2,3].

The use of electron beam technology for surface modification has, however, been little reported, even though this approach has many advantages. Particular benefits worthy of mention here are the ability to adjust the degree of modification like cleavage of bonds, reticulation and insertion of new chemical groups as well as a simultaneous sterilization of the surface [4]. With careful choice of specific parameters, it is possible to treat sensitive substrates such as biomaterials under gentle conditions [5].

Treatment by low-energy electrons is able to modify the surface of a material without adversely affecting the properties of the base material. The electron beam, mostly secondary electrons, excites and ionizes atoms and polymer molecules on the targeted substrate surface and the penetrated random layer. Atoms and molecules of surrounding media (atmosphere and water) are included in these reactions. Radicals, produced on this way, promote the decomposition and/or crosslinking of the polymer surface as well as implantation of new functional groups, which depends on present media during the

treatment. The resulting network density is affected by availability of unconsumed radicals from the atmosphere which participate in crosslinking reactions and by the quantity of chain scission reactions which are in time-dependence on exposure. Related to experimental conditions all reactions which are initiated and take part in the treatment process are in concurrence to each other. The summary of them delivers the final product modification.

The modification of polyurethane and silicone polymers by electron beam treatment is presented in this paper. The treated surfaces were subsequently characterized for their hydrophilicity, surface energy, chemical composition, and morphology.

In vitro tests using mouse fibroblasts (L929) demonstrated the improved biofunctionality of polyurethane after electron beam treatment. No fibroblasts adhered to either untreated silicone or to silicone treated with an electron beam. However, the material appeared to be protected against damage caused by cells and their enzymes as a result of the modification of the surface molecules. The results show that electron beam technology is a suitable method for functionalizing polymer surfaces for use in therapeutic medical products [6].

2. Experiments

2.1. Electron beam treatment

In general, electrons accelerated with low energy between 100 and 150 kV, are used for the treatment. No radioactivity is present and X-ray shielding is not too expensive. Modern units are available by low cost. One advantage is the exact guidance of applied energy doses in (mostly short) time, amount and penetration depth. The

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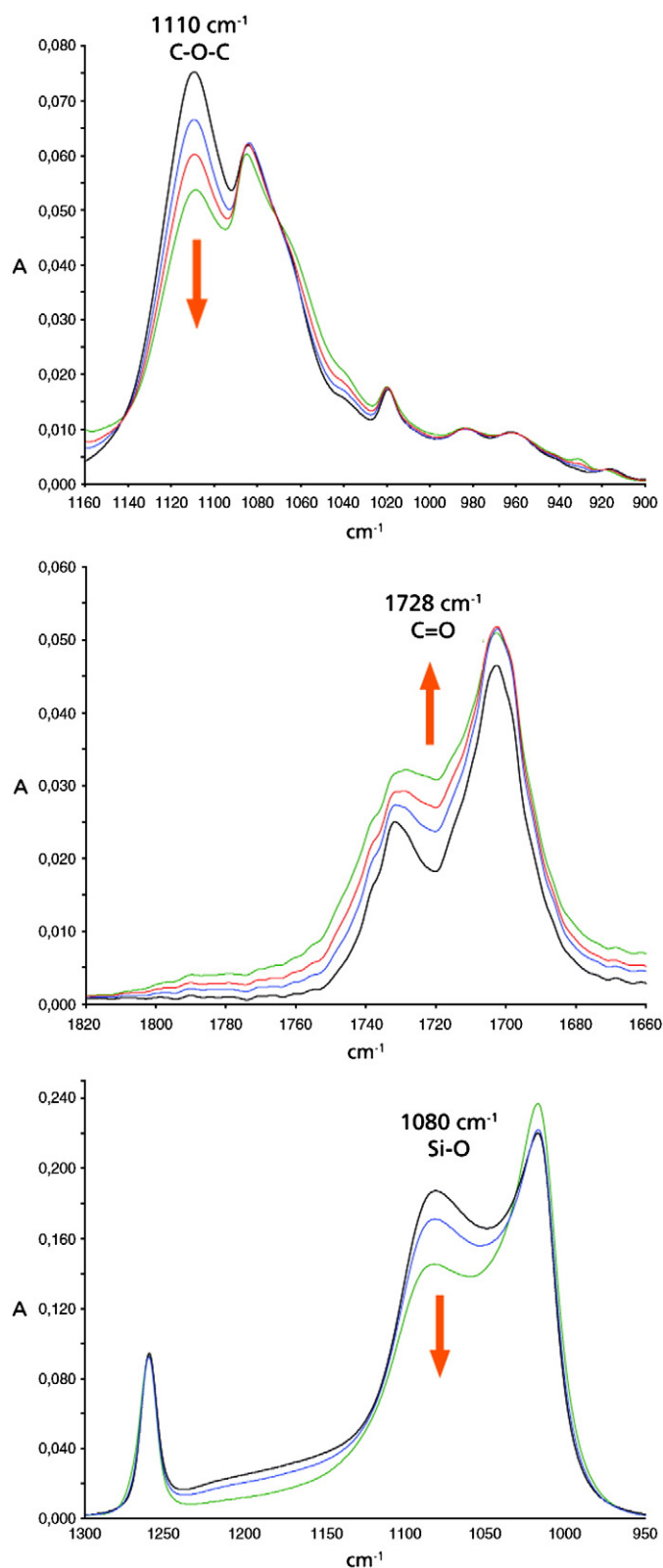


Fig. 1. ATR-FTIR spectra of PUR in the wavelength range 3600–1000 cm^{-1} (both above) and of silicone in the wavelength range 1300–900 cm^{-1} (below).

quantity of absorbed energy per unit of mass of treated material is expressed in Gray (Gy): 1 Gy = 1 Joule/kg [7].

Compared to most other modification methods, the electron beam not only functionalizes the surface but also simultaneously sterilizes it [4].

For the biofunctionalization, the electron beam treatment is applied at atmospheric pressure. The accelerated electron beam passes from the high vacuum generation chamber through a beam exit window, a thin metal foil, to atmosphere in an X-ray shielded reaction chamber.

For the treatment of the polyurethane and silicone samples, the electron emitter was set in our experiments to an acceleration voltage of 150 kV and an electron current outlet of 10 mA. Hereby the effective penetration depth into common polymer surfaces and boundary layers is about 100 μm [8,9].

The treatment was carried out in an air atmosphere at normal pressure. Samples were linearly carried below the electron beam and exposed to irradiation. The total dose applied to the samples amounted to up to 6000 kGy. High doses were realized by subsequent irradiation steps in single doses of 100 kGy with pauses of 1 min to prevent overheating of the polymer material [10].

2.2. Polymer characterization

The contact angle measurements were also carried out using the contact angle test unit (OCA20) and SCA22 software (version 3.12.11) from Dataphysics Instruments GmbH (Filderstadt). The “sessile drop” method was used. Via an electronically controlled injection module, a 1 μl droplet of the test liquid (deionized water or ethylene glycol) was applied to the test surface at an ambient temperature of ca. 21 $^{\circ}\text{C}$ in air atmosphere. After manual determination of the baseline of the photographed droplet, the contact angle was measured automatically by the image recognition software in the unit. Usually the method according to Owens–Wendt–Rabel–Kaelble (OWRK) is applied for calculation and was used here [11,12].

The ATR-FTIR spectra were taken on a Spectrum 2000 instrument (Perkin Elmer GmbH) using a compatible ATR module with germanium crystal (Specac Inc.). The spectra were recorded in the region from 700 cm^{-1} to 4000 cm^{-1} at a resolution of 4 cm^{-1} .

Examinations by X-ray photoelectron spectroscopy (XPS) were made at UHV XPS/SPM System of Omicron Nanotechnology GmbH. This established method uses an X-ray beam, directed to the sample surface. The impact energy of photoelectrons is absorbed by core electrons of the elements. If the energy high enough resp. is equal to binding energy (unique for every element), the electrons emit out of surface. By analysis of their energy and intensity the presence and composition of chemical elements on the surface can be detected: especially here the C 1s, Si 2p or O 1s lines [13,14].

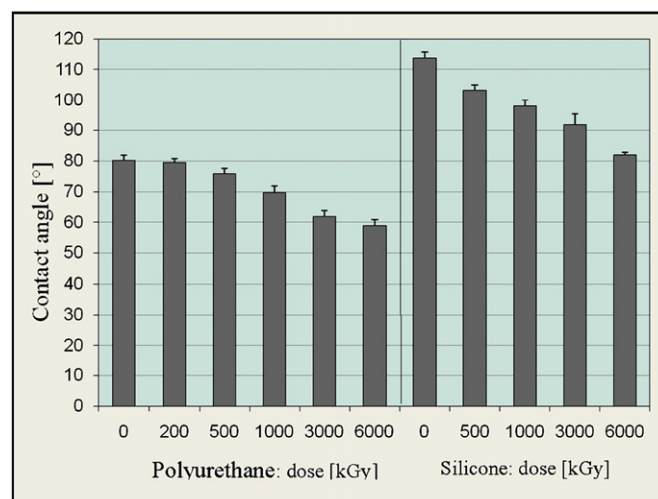


Fig. 2. Contact angle with water of polyurethane and silicone as a function of the electron beam dose.

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