



## The effect of electron beam on sheep wool



Mária Porubská<sup>a,\*</sup>, Zuzana Hanzlíková<sup>a</sup>, Jana Braniša<sup>a</sup>, Angela Kleinová<sup>b</sup>, Peter Hybler<sup>c</sup>, Marko Fülöp<sup>c</sup>, Ján Ondruška<sup>d</sup>, Klaudia Jomová<sup>a</sup>

<sup>a</sup> Constantine the Philosopher University in Nitra, Faculty of Natural Sciences, Department of Chemistry, Tr. A. Hlinku 1, 949 74 Nitra, Slovakia

<sup>b</sup> Polymer Institute, Slovak Academy of Sciences, Dúbravská cesta 9, 842 36 Bratislava, Slovakia

<sup>c</sup> University Centre of Electron Accelerators in Trenčín, Slovak Medical University, Limbová 12, 833 03 Bratislava, Slovakia

<sup>d</sup> Constantine the Philosopher University in Nitra, Faculty of Natural Sciences, Department of Physics, Tr. A. Hlinku 1, 949 74 Nitra, Slovakia

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### ABSTRACT

The effect of accelerated electron beam with absorbed doses of 0–400 kGy on wool fibres was investigated. The S-oxidized species, CH groups, secondary structure, temperature and enthalpy of crystal cleavage, strength and elongation of the fibres were monitored. All the properties showed fluctuation with the absorbed dose and are related with change of the secondary structure. Increasing absorbed dose led to progressive predominance of  $\beta$ -sheet over  $\alpha$ -helix conformation. The helical conformation favoured generation of S-sulphonate while the  $\beta$ -sheet suppressed it. Changes in the abundance of –CH– groups indicated a slight networking. High absorbed doses resulted in the polypeptide chain breaking and the formation of shorter fragments with helical conformation. The fibre strength was not changed significantly and the elongation, after an initial increase, monotonously decreased due to further denaturation and chain breaking.

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### 1. Introduction

Nowadays traditional textile exploitation of sheep wool has been considerably replaced with the use of using synthetic fibres. Wool is fully renewable material and deserves attention regarding sustainable development. Therefore present research should be aimed also in studying further and more detailed applications of this remarkable raw material.

The main component of wool fibre is hard keratin, having content of the cysteine residual group. The principal structural units in the native wool are successive turns of the alpha helix. The intrinsic stability of the alpha helix, and thus the fibre, results from intramolecular hydrogen bonds. Both low-sulphur protein and high-sulphur protein exist in wool fibre, which play a key role in determining super-molecular structure of wool fibre. The low sulphur content proteins have a  $\alpha$ -helical crystalline structure, while the other proteins do not have a helical structure [1].

It is believed that in the low-sulphur protein of raw wool fibres, about 50% molecules exhibit alpha-helix structure in microfibrils, and the other 50% of molecules exhibit the form of irregular coils.

Most molecules exist in matrix as irregular coils in the high-sulphur protein, and disulphide bonds link low-sulphur protein molecules to high-sulphur protein molecules. Therefore protofibrils in wool fibre are coupled with matrix via S–S bonds.

Wool has been an object of numerous studies due to its exceptional properties. Many research studies have been focused on improving shrink resistance and dyeability of wool fabric [2] in the textile industry. Potential applications of wool have been examined in composite materials [3,4], in medicine [5] and other fields. Investigations have also focused on the obtaining of keratin [6] as well as keratin powdering [7]. A specific field dealing with superficial structure variations of wool due to physical–chemical modification involves plasma application [8], corona charges [9], microwave [10] and UV radiation [11], etc. These mentioned radiation treatments modify only the wool surface.

Within the last decades development of radiation technologies offers further opportunities to study the wool property modifications. The main feature and advantage of radiation initiation is capability to generate active intermediate products in the solid phase. An electron beam can penetrate the whole volume of the fibres and affect all parts of the fibre substructures. Such treatment does not require the use of any chemicals. It is environmental friendly dry process, which does not involve any solvent and reagent of wet chemical processes.

\* Corresponding author. Tel.: +421 37 6408 655; fax: +421 37 6408 020.  
E-mail address: [mporubska@ukf.sk](mailto:mporubska@ukf.sk) (M. Porubská).

Although the impact of electron beam on synthetic polymers has been described in several papers, e.g. Ref. [12], according to our best knowledge, only one brief report on the electron beam effect on surface wool properties has been published [13]. The elucidation of such modification of whole wool fibre volume could show new alternatives for processing and exploitation of wool since it is a very often undesirable waste today. However, it is necessary to understand some fundamental variations induced by electron beam in wool.

In view of these remarks, the aim of our study is the irradiation of wool by different absorbed doses of electron beam and, in parallel, observation of the wool property variations using FTIR spectroscopy, microcalorimetry and tensile properties.

## 2. Materials and methods

### 2.1. Materials

In the work industrially scoured wool with fibre thickness of 22–27  $\mu\text{m}$  was used in the form of wool combed sliver, supplied by the company Pradiareň vlny JK, Nové Mesto nad Váhom, Slovakia.

### 2.2. Sample preparation

The wool was extracted with dichloromethane in Soxhlet using 9 flow-off, then rinsed 2 $\times$  in distilled water, first dried freely and finally dried in a laboratory oven at 60 °C for 4 h. Such wool being still warm was put into a zip PE pockets with dimension of 18  $\times$  25 cm, closed and saved in a dessicator till irradiation.

### 2.3. Sample irradiation

For each irradiation dose, the samples of about 12 g in mass put in PE pockets were placed into carton boxes grouped according to selected absorbed doses. The exposure in air was conducted at the University Centre of Electron Accelerators in Trenčín in linear electron accelerator UELR-5-1S (manufacturer FGUP “NIIIEFA”, Petersburg, Russia) with 5 MeV of installed energy, an electron beam (hereafter, EB) intensity of 200  $\mu\text{A}$ , mean power of 1 kW and mean dose rate of 750 kGy/h. The doses applied were 0-16-25-40-63-100-156-250-400 kGy repeating 100 kGy cycles plus needed supplementing dose, if necessary. Between individual irradiation cycles, the samples were allowed to cool down for 30 min to maintain the temperature below 50 °C.

### 2.4. FTIR spectral analysis

Spectral analysis was carried out 9 days after the irradiation. For the measurements, the fine-cut wool samples were blended with KBr in a small grinder and pressed into discs in a pellet press. The moulded discs were kept in the dessicator till spectral measurement. Infrared spectra were collected using AVATAR 330 Thermo Nicolet FTIR Spectrometer (manufacturer Thermo Nicolet Corporation, Madison, USA) in the range of 400–4000  $\text{cm}^{-1}$  at 4  $\text{cm}^{-1}$  resolution and 3 scan repetitions per analysis.

### 2.5. Thermal analysis

Differential scanning calorimetry (DSC) was performed on 1–2 mm wool cut-offs using Mettler Toledo DSC822<sup>e</sup> device in air atmosphere. For analysis the wool sample, in quantities about 5 mg, was moulded into an aluminium crucible 40  $\mu\text{l}$  in volume. The heating rate was 20 °C/min within the interval from 25 °C to 300 °C with 3 repetitions per analysis.

### 2.6. Tensile properties

Before testing, the samples were conditioned at 21 °C and 63% relative humidity for 48 h. The strength at break and elongation were measured on twenty specimens following ISO 5079: 1999 using the tearing machine Testometric (manufacturer Testometric Company Ltd., Lancashire, United Kingdom) at 21 °C and 63% humidity, clamp speed movement 10 mm/min and working length 10 mm. The thickness was scanned by the digital thickness meter Sylvac (manufacturer Sylvac SA, Crissier, Switzerland).

## 3. Results and discussion

Absorption of electron particle energy in the material is complicated by X-ray productions, liberations of high energy secondary electrons, photo- and Crompton processes. Therefore in radiation chemistry mechanism of chemical reactions is no explicit EB energy dependence. However in this work keeping all remaining process parameters to be constant, the only variable was absorbed dose. Therefore observed variations have been relative to the absorbed dose.

### 3.1. FTIR analysis of sulphur-oxidized products

The disulphide bond S–S is the most reactive part of keratin and, after being initiated in air, gives to arise several sulphur-oxidized species such as S-sulphonate (S-sulph), cysteic acid (CA), cystine monoxide (CMO) and cystine dioxide (CDO). Characteristic IR absorbance wave numbers used to estimate the sulphur-oxidative species are given in Table 1 and they are closed to data from papers [4,14].

Collected FTIR spectra were inverted in the second-order derivative spectra, the related signals were read and divided by the signal for reference Amide III. These absorbance ratios were relative to the corresponding ratios of the non-irradiated sample. EB absorbed doses resulted in some shift almost of all bands towards higher frequencies and the ultimate shift up to 12  $\text{cm}^{-1}$  was observed for 25–40–63–100 and 400 kGy doses. However, based on the second-order derivative treatment, there were not any doubts on the assignment of related functional groups.

The results from FTIR analysis for the wool irradiated by graded absorbed doses represent a relative variation of individual functional groups when compared with the parent wool and are shown in Fig. 1. It can be seen that composition of the oxidized sulphur products was changing the each species considerably. Following the amount of the generated species (S-sulph, CA, CMO and CDO) the region of the absorbed doses can be divided roughly into two parts, 0–220 kGy and 220–400 kGy.

In the range of 0–220 kGy, we observed a fluctuation of all oxidized products of which S-sulph and CMO were shown to be the largest. At the lowest absorbed dose of 16 kGy, the amounts of CA and CDO decreased below the initial level, indicating transformation into other products and temporary increase of CMO. At the next absorbed doses 25–40 kGy the formation of CMO together with CA and CDO were reduced to a minimal level at 40 kGy while

**Table 1**  
Characteristic wave numbers for non-irradiated wool.

Form	Structure	Wave number ( $\text{cm}^{-1}$ )
Cystine dioxide	–SO <sub>2</sub> –S–	1128
Cystine monoxide	–SO–S–	1075
Cysteic acid	–SO <sub>3</sub>	1044
S-sulphonate (Bunte salt)	–S – SO <sub>3</sub>	1022
Amide III as internal reference band	–N–H mixed	1232

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