

Influence of Accelerated Electron-Irradiation on Sorption Properties of Latvian Darkhead Sheep Wool Fibers

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Latvian Darkhead (LD) is a local sheep breed and a genetic resource in Latvia. Preservation and development of the sheep population in the local region is important for the recultivation of fields, it serves as a source for export and local use, as well as waste wool can be applied for developing new products, for example, sorbents for volatile organic pollutants. Therefore, investigation of the sorption properties of the LD sheep wool fibers is under interest. In addition, modification options of the wool for improvement of properties are viewed. Therefore, in the present work, sheep wool fibers as well as accelerated electron-irradiated fibers are analyzed and compared. Fourier transform infrared (FTIR) spectrometry is applied to develop the sorption testing system of volatile organic compounds. An analytical system consisting of a volatile organic compound source, sheep wool filter, and FTIR spectrometry cell is tested and applied for analysis of wool sorption properties for acetone molecules. Registration of the FTIR spectra was performed within the range of 600–4000 cm⁻¹, in the nitrogen flow of about 150 mL/min. FTIR analysis shows, that the accelerated electron irradiated sheep wool fibers absorb acetone of about 33% more than non-irradiated fibers. The obtained results will be used for developing recommendations for filter producers to fabricate filter components containing LD sheep wool fibers.

Keywords: sheep wool, infrared spectrometry, electron irradiation, sorption.

1. INTRODUCTION

Only sheep wool fibers of high quality are utilized by the textile industry, while unprocessed sheep wool fibers often regrettably become waste at loading depots [1]. Non-textile sheep wool has found its place already as an insulating material [2], building additive [3, 4], keratin source [5], or fertilizer [6, 7]. However, still new applications are required, therefore ionizing radiation modifications can be applied [8, 9]. Absorbed doses up to 410 kGy [10–14] have been applied and shown to increase content of free radicals [15]. Among the applied irradiation types are 5 MeV accelerated electrons [16] and gamma rays (cobalt-60 source) [17]. Therefore, an absorbed dose of several hundred kGy can be selected for this work to perform comparable measurements to the previous studies. Previously, the time dependency of the various effects on wool has been investigated, for example, storage conditions [18] and post-irradiation changes [19]. It is proposed that sheep wool fibers can be applied in air filtrating systems [20–22]. However, for the first opinion, if the material has the potential for further applications as a filter, a rapid evaluation is required. Therefore, for the estimation of

volatile organic compounds sorption capabilities in the wool, an approach involving Fourier transform infrared spectra (FTIR) registration is developed and exploited in the present work. As example for testing the FTIR method for sorption of volatile pollutants [23], acetone is selected that occurs in paint, pharmaceutical [24], and polymers processing [25, 26]. The application of recovery of vapors of volatile organic compounds from air is targeted to pollution control, and solvent recovery [27, 28]. Among the known acetone sorption systems are water columns [29], wood barks [30], polymer membranes [31], biochar [32], and activated carbon materials [33]. Sheep wool fibers are proposed for application in water filtrating systems [34] and air filters [35]. Therefore, to estimate the applications of the locally available source for air filtrating units, it is necessary to estimate its sorption behavior.

In the present work, a locally available [36, 37], renewable source, Latvian Darkhead (LD) sheep wool fibers are investigated. Fibers are irradiated with 10 MeV accelerated electrons and both, non-irradiated and irradiated wool, analyzed as potential filtering material for extraction of acetone molecules from gaseous flow of inert gas.

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2. EXPERIMENTAL

Latvian Darkhead (LD) sheep wool fibers, diameter of $25 \pm 3 \mu\text{m}$ [18, 38], were cleaned and prepared for analysis by *Sunakstes vilnas nams* Ltd., Preparation involves washing of the cut wool, drying, and coursing to eliminate soil, dust and other impurities. Wool fibers were irradiated with 10 MeV accelerated electrons up to 500 kGy absorbed dose at room temperature in air at the Institute of Nuclear Chemistry and Technology (Warsaw, Poland) [39].

Chemical bonds in non-irradiated and electron irradiated wool fibers were analyzed by FTIR spectrometry, *Bruker Vertex 70v* spectrometer equipped with attenuated total reflection (ATR) module with a diamond crystal, $\pm 2 \text{cm}^{-1}$, $400\text{--}4000 \text{cm}^{-1}$, 20 scans per spectrum, at least 10 measurements per sample.

Non-irradiated LD sheep wool fibers were exploited for testing the FTIR based sorption system, where the FTIR spectra profiles are used as indicators of the sorption analysis. The FTIR based sorption testing system includes the filter cartridge, volatile organic compound source and measuring cell for gaseous compounds (*Bruker Vertex 70v*, $600\text{--}4000 \text{cm}^{-1}$, $\pm 4 \text{cm}^{-1}$, Liquid Nitrogen-HgCdTe detector). A purge gas, nitrogen flow (*Linde gas* Ltd, 99.99 %) 150mL/min guided through the cartridge to the spectrometer cell for a certain time (1.5 min), then the flow switched through the vapor of interest and FTIR spectra registered. Afterwards, the flow again changed to nitrogen. In the present work, acetone (*Sigma Aldrich*) vapor was used. The scheme of the measuring system is shown in Fig. 1.

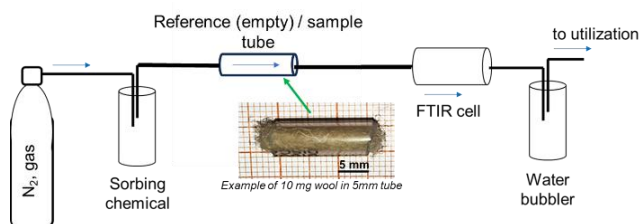


Fig. 1. Scheme of FTIR based sorption testing system for gaseous organic compounds

During the whole analysis (background measurement, insertion of the sorbing chemical, switching back to the background) the FTIR spectra are registered. The estimation of the sorption is performed by analyzing the intensity profile of the selected bond from the acetone spectrum. For the irradiated wool samples, the analysis was performed analogously as for non-irradiated.

3. RESULTS AND DISCUSSION

Chemical bonds of non-irradiated and electron irradiated sheep wool fibers were analyzed by means of FTIR-ATR. Afterwards, the fibers were incorporated into the FTIR based sorption testing system and tested for the sorption of acetone.

3.1. Analysis of fibers

Chemical bonds of the LD sheep wool fibers are analyzed using FTIR-ATR spectrometry. Since the fibrous structure is quite inhomogeneous, the intensities of the

chemical bonds vary and only after summarizing information from several spectra, the overview of the ratios and the compositions of chemical bonds can be characterized. In Fig. 2, it is shown the variation of the spectra within the wool sample of about 1.0 g, where the aliquot for each measurement taken at about 0.1 mg and placed on ATR crystal. Similar variations in the spectra were observed in other samples as well [38]. The main peaks occur in the range of $400\text{--}1700 \text{cm}^{-1}$ and $2850\text{--}3300 \text{cm}^{-1}$. The spectra correspond to the keratin-based [40] chemical structures, containing amide I and II vibrations around 1530 and 1640cm^{-1} [41]. Sulphur containing bonds are in the range of 1020 and 1050cm^{-1} [42]. Oxygen containing bonds in COH groups occur around $1400\text{--}1450 \text{cm}^{-1}$ [43]. Stretching vibrations of $-\text{CH}_2$ and $-\text{CH}_3$ groups are at 2850 and 2920cm^{-1} [44]. Broad peaks around $3200\text{--}3600 \text{cm}^{-1}$ show to presence of primary amines [45].

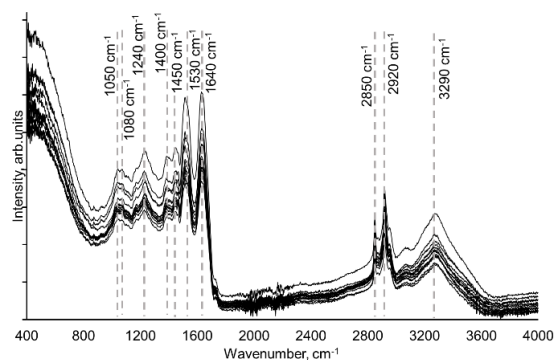


Fig. 2. FTIR-ATR spectra of non-irradiated LD wool fibers

The average spectrum of at least 10 spectra is calculated and compared to the electron irradiated wool spectrum. The average FTIR spectra on non-irradiated wool and electron irradiated wool are shown in Fig. 3. The spectra in Fig. 3 are placed on the shifted y axis for better resolution and visibility of the bonds.

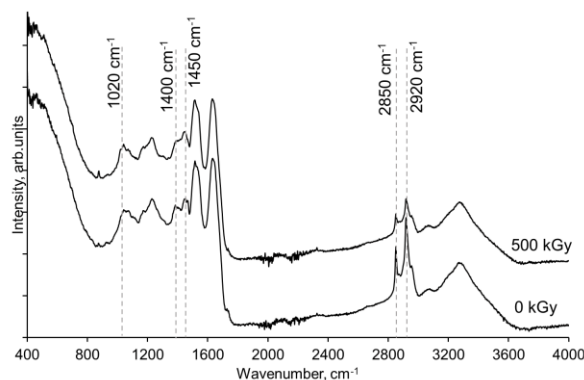


Fig. 3. Averaged FTIR-ATR spectra of non-irradiated (0 kGy) and electron irradiated (500 kGy) LD wool fibers

As can be seen from the obtained FTIR spectra, irradiation with 10 MeV accelerated electrons causes changes in some of the bonds and their intensities. In the electron irradiated wool fibers, the intensities of C-H bonds related signals at 2850 and 2920cm^{-1} have been decreased. In spectra of non-irradiated wool peaks at 2850 and 2920cm^{-1} are comparably higher than the nearby peaks of amine bonds. In irradiated wool intensities of 2850 and

2920 cm^{-1} peaks attributed to C-H bonds, have been decreased. Meanwhile, a slight increase of the signals at 1020, 1400 and 1450 cm^{-1} occurs that may correspond to -C-O- stretching and bending vibrations [46–48]. The changes in the spectra may be due to reorganization of the bonds [49], polymerization [50] or ozone [51] caused oxidation [52] of the functional groups in fibers.

Newly formed -CO bonds may act as capture centers for acetone molecules. In biochar, the acetone sorption mechanisms are based on large surface area and diffusion [53]. Surface condensation and hydrogen bond interactions are reported among the possible sorption mechanisms [54].

3.2. FTIR based sorption testing system for estimation of wool sorption properties

By applying FTIR based sorption testing system, the sorption properties of acetone in the non-irradiated and electron irradiated sheep wool fibers are evaluated. The measuring technique consists of two main stages, first where the measurement is performed with an empty filter cartridge. Nitrogen gas flow is guided through a cartridge and then acetone is introduced into the system. The FTIR spectra are recorded over a certain time and then the system is switched back to nitrogen flow. An example of measurement with acetone and an empty filter cartridge is shown in Fig. 4. A vibrational band with the maximal intensity for the reference spectrum, at 1753 cm^{-1} , assigned to the -C=O group in the ketone [55,56], specific to the introduced reagent, is selected for analyzing the sorption efficiency.

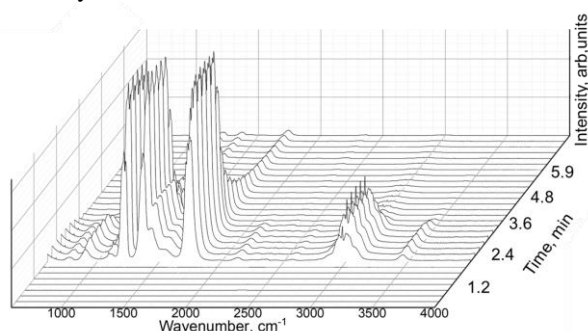


Fig. 4. FTIR spectra of acetone test through empty filter cartridge

The sorption profile of the acetone band at 1753 cm^{-1} on reference test, non-irradiated wool and electron irradiated wool is in Fig. 5.

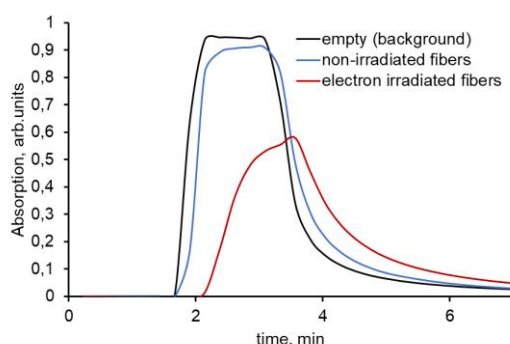


Fig. 5. Absorption profiles of 1753 cm^{-1} acetone stretching signal of acetone in the empty testing system and with the non-irradiated and electron irradiated sheep wool fibers

Normalizing the intensities to 1 and integrating the total band intensities to 100 %, it can be calculated that non-irradiated wool sorbs ~ 1 % of acetone molecules, while electron irradiated wool already ~33 % of the total amount of acetone. Electron irradiation created -C=O bonds, that were observed in FTIR-ATR spectra, can undergo physisorption interactions with sorbing molecules [57] causing the delayed signal in the FTIR spectra.

Therefore, the modifications of LD sheep wool with accelerated electrons can be applied to improve the sorption properties of the fibers. The changes in the sorption profile correspond to the changes in the chemical bond composition. Respectively – irradiation causes oxidation of the functional groups of the fiber structure, which further works as sorbing sites for -C=O functional groups containing molecules. Therefore, electron modified LD sheep wool fibers can be used as components in the air filtrating systems aimed for use in factories and other places, where the presence of acetone vapor is expected. In such a way the available, renewable source, LD sheep wool has a perspective application in air filtrating systems.

4. CONCLUSIONS

An absorbed dose of 500 kGy of accelerated electrons induces changes in the chemical bonds of LD sheep wool fibers, causing a decrease in intensities of -CH signals and the formation of -CO containing sites. Changes in the chemical bonds are followed by increased sorption capability of acetone molecules that are detected by the FTIR method. As result, the FTIR method can be applied as a rapid sorption estimation method. As well as determined acetone sorption can be further used as a beneficiary effect for applying electron irradiated sheep wool fibers as components in the air filtrating systems.

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