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# IR Spectroscopy as a Possible Method of Analysing Fibre Structures and Their Changes Under Various Impacts

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## 1. Introduction

Infrared absorption spectroscopy, broadly applied to analyse polymer structures (within the spectral wavenumber range of 400-4000  $\text{cm}^{-1}$ ), is also employed as a method of studying fibre structures and their changes. This is one of the instrumental methods commonly applied in Fibre Physics for the purpose of qualitative and quantitative analyses of fibre orientation, studies of fibre crystalline structure, and selective evaluation of the structure of fibre surface layers, as well as the effects of superficial and volumetric modification of fibre structures.

The experimental basis in the studies are the absorption spectra of IR radiation through adequately prepared fibre samples.

Studies of the share of crystalline material in fibres (crystallinity) employ so-called fibre pellet preparations, with fibres homogeneously dispersed in KBr, and the IR radiation technique. For quantitative evaluation of crystallinity, the ratio taken is that of the intensity of the so-called "crystalline" or "amorphous" absorption band to the intensity of the so-called "internal standard" absorption band.

## 2. Studies of fiber surface layer molecular structure using ATR-IR technique

The Attenuated Total Reflectance method (ATR - *Attenuated Total Reflection*), has found application in spectrophotometry, for studies of the surfaces of materials, including fibers[1].

In ATR-IR spectroscopy, infrared radiation passes through a crystal characterized by high refraction index, which makes it possible for the rays to be reflected many times in its interior (figure 1).

Close contact of the studied sample with the crystal surface is a prerequisite for correct measurement.

To obtain reflection inside the crystal, the angle of incidence of the rays must exceed the so-called „critical” angle  $\theta_c$ . This angle is a function of radiation beam, both in the sample and in the crystal, formula 1:

$$\theta = \arcsin\left(\frac{n_2}{n_1}\right) \quad (1)$$

where:

- $n_2$  is the radiation beam refraction index for the sample,
- $n_1$  is the radiation beam refraction index for the crystal.

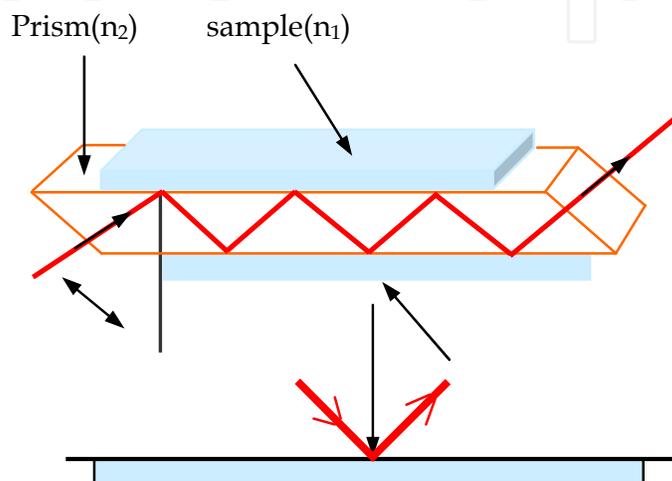


Fig. 1. The Schematic of a path radiation in repeated reflection in the crystal [2].

The radiation entering the crystal may, depending on the type of the investigated sample, penetrate the sample to the depth of to  $2\mu\text{m}$  [3,4] (fig.1). The penetration depth of the „vanishing” wave  $d_p$  is defined as the distance between the crystal and the sample, formula 2 [4].

$$d_p = \frac{\lambda}{2\pi n_1 \left[ \sin^2 \theta - \left( \frac{n_2}{n_1} \right)^2 \right]^{1/2}} \quad (2)$$

where: -  $\lambda$  is the wavelength of IR radiation.

The measurements are performed by means of a FTIR spectrophotometer, equipped with an ATR sampling accessory.

The conditions most commonly applied for the measurements are as follows:

- the range of IR radiation used in the studies for wool, natural silk and polyamide fibers is  $2000\div 600\text{ cm}^{-1}$ , for polyacrylnitrile fibers -  $2500\div 600\text{ cm}^{-1}$ ,
- the mirror movement speed -  $5\text{ mm/s}$ ,
- the radiation wavenumber resolution recorded by the detector -  $4\text{ cm}^{-1}$ ,
- as a rule, at least 32 scans of each sample with IR radiation beam are performed to obtain the absorption spectra.

For the investigated fiber samples, IR absorption spectra are plotted in the  $A=f(1/\lambda)$ , or  $T=f(1/\lambda)$  system. They provide the basis for interpretation of changes in the molecular and supramolecular structures of the surface layer. An example of a spectrum has been presented in figure 2. The absorption bands are corrected by determination of the baseline, and then their absorbance is determined.

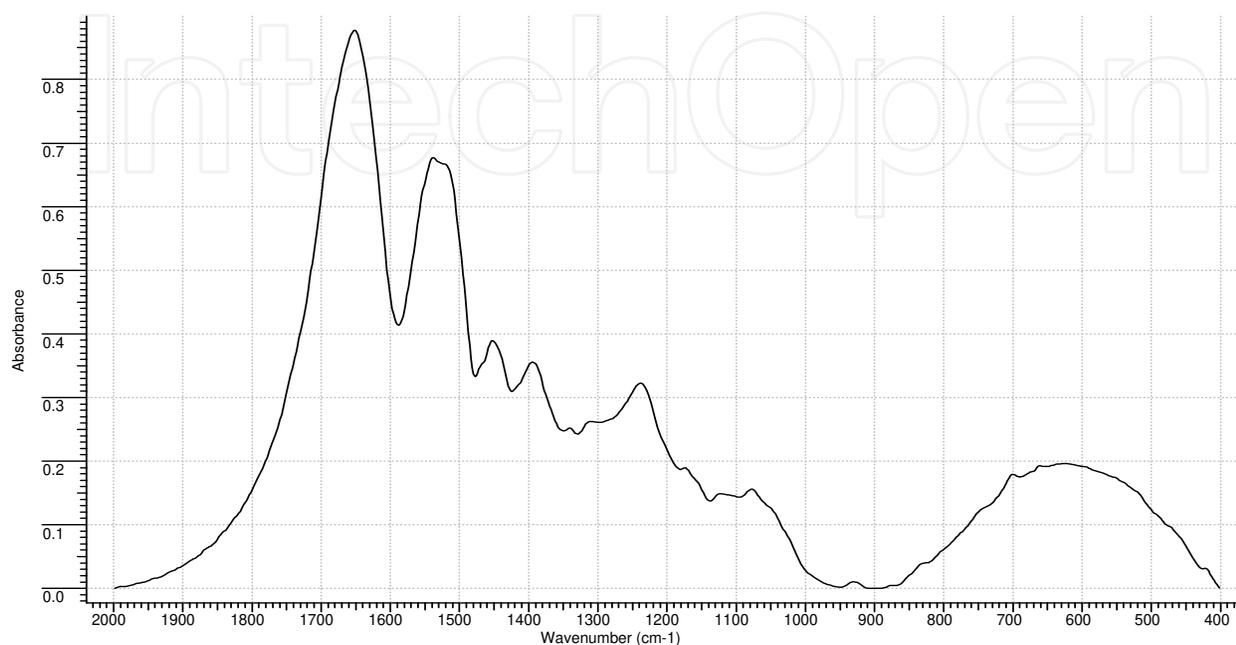


Fig. 2. FT-IR spectrum of wool fibre.

Table 1 presents the correlation between the location of the absorption bands and the type of the chemical groups for wool fibers.

Wavenumber cm <sup>1</sup>	Absorbing group and type of vibration
2950	$\nu_a$ (CH <sub>2</sub> ), $\nu_a$ (CH <sub>3</sub> )
1658	NH <sub>2</sub>
1539	amide II
1393	COO-
1233	CNH
1074	CC, CN <sub>3</sub>
932	CH <sub>3</sub> , CONH

Table 1. The correlation between the location of the absorption bands and the type of the chemical groups for wool fibers under Rau, Urbańczyk [2,5].

### 3. Studies of fiber structure with IR absorption spectroscopy using the transilluminating technique

#### 3.1 Studies of overall fiber structure

Spectroscopic IR transilluminating technique is used for qualitative studies of changes in the overall structure of fibers. Qualitative analysis of the absorption spectra makes it possible to

assess transformations of chemical groups and macromolecules as a result of interactions with external factors.

An IR absorption spectrum represents the changes of radiation intensity, or a value proportional to it, as a function of wavelength  $\lambda$  or another value associated with  $\lambda$ . Absorption of energy portions by intra- and intermolecular bonds, dependent on the frequency of normal vibrations of the chemical group, determines the location and intensity of the corresponding absorption band in the spectrum. Correlation between the location of the band in the absorption spectrum (value of the wavenumber for the band maximum), and the type of the absorbing chemical group is determined experimentally. The dependences of location of the absorption bands on the type of the absorbing groups for fibers are included in the correlation tables and provide the basis for quantitative analysis of the investigated chemical compounds. The correlation of band location with the type of the absorbing chemical group for PAN fibers is presented in table 2 as an example.

The changes in molecular structure manifest themselves by appearance of new absorption bands correlated with the newly formed chemical groups, or by changes in intensity of the absorption bands correlated with the existing chemical groups.

The measurements are performed using a FTIR spectrophotometer.

Wavenumber , cm <sup>-1</sup>	Absorbing group and type of vibration
2950	$\nu_a$ (CH <sub>2</sub> )
2930	$\nu$ (CH)
2870	$\nu_a$ (CH <sub>2</sub> )
2237	$\nu$ (CN)
1447	$\delta$ (CH <sub>2</sub> )
1362	$\delta$ (CH)
1335	$\gamma_w$ (CH <sub>2</sub> ) + $\nu_a$ (CC)
1310	$\gamma_w$ (CH <sub>2</sub> ) - $\delta$ (CH)
1247	$\gamma_w$ (CH) + $\gamma_w$ (CH <sub>2</sub> ) - $\nu_a$ (C-C)
1115	$\nu_s$ (C-C) - $\delta$ (CH)
1073	$\nu_s$ (C-C), $\gamma_r$ (CH <sub>2</sub> ) - $\delta$ (C-C-CN)
865	$\gamma_r$ (CH <sub>2</sub> )
778	$\nu$ (C-CN) + $\gamma_t$ (CH <sub>2</sub> ), $\nu$ (C-CN) - $\gamma_r$ (CH <sub>2</sub> )
570	$\delta$ (C-C-CN) - $\delta$ (C-C-N)
537	$\delta$ (C-C-CN) - $\delta$ (C-C-N)

Table 2. The IR absorption bands for polyacrylnitrile fibres according Rau, Yamader, Urbańczyk [2,5,6].

The experiments make use of tablet preparations with 1% mass/weight content of the investigated fiber. The tablets are obtained from powdered fiber (segments of length equal

to fiber thickness), dispersed in dried potassium bromide with a homogenizer. The tablets are prepared by hydraulic press moulding. Standard conditions are applied for tablet preparation, i.e.:

- homogenization time - 2 minutes,
- moulding time - 10 minutes,
- forcing pressure - 10 MPa.

Standard conditions for measurements:

- to obtain the IR absorption spectra, transmission technique is used,
- the range of IR radiation used in the studies e.g. for wool, natural silk and polyamide fibers is  $2000 \div 400 \text{ cm}^{-1}$ , for polyacrylnitrile fibers -  $2500 \div 400 \text{ cm}^{-1}$ ,
- the mirror movement speed - 5 mm/s,
- the radiation wavenumber resolution recorded by the detector -  $4 \text{ cm}^{-1}$ ,
- as a rule, at least 32 scans of each sample with IR radiation beam are performed to obtain the absorption spectra.

The spectra obtained in the  $A = f(1/\lambda)$  system provide the basis for determination of absorbance  $A$  (figure 3), the values of which are adopted for the final interpretation of changes in the molecular structure of the studied fibers. An example of a spectrum is presented in figure 4.

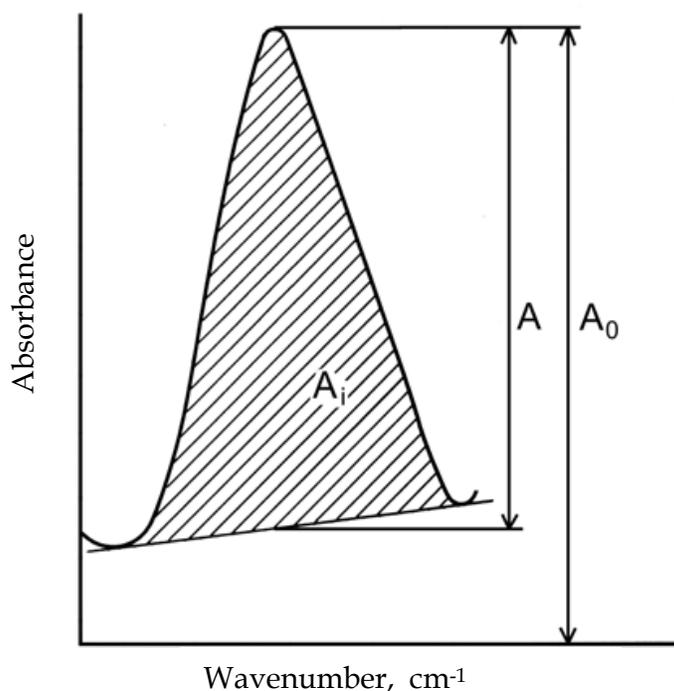


Fig. 3. The method of determining the volume of absorbance  $A$  [4].

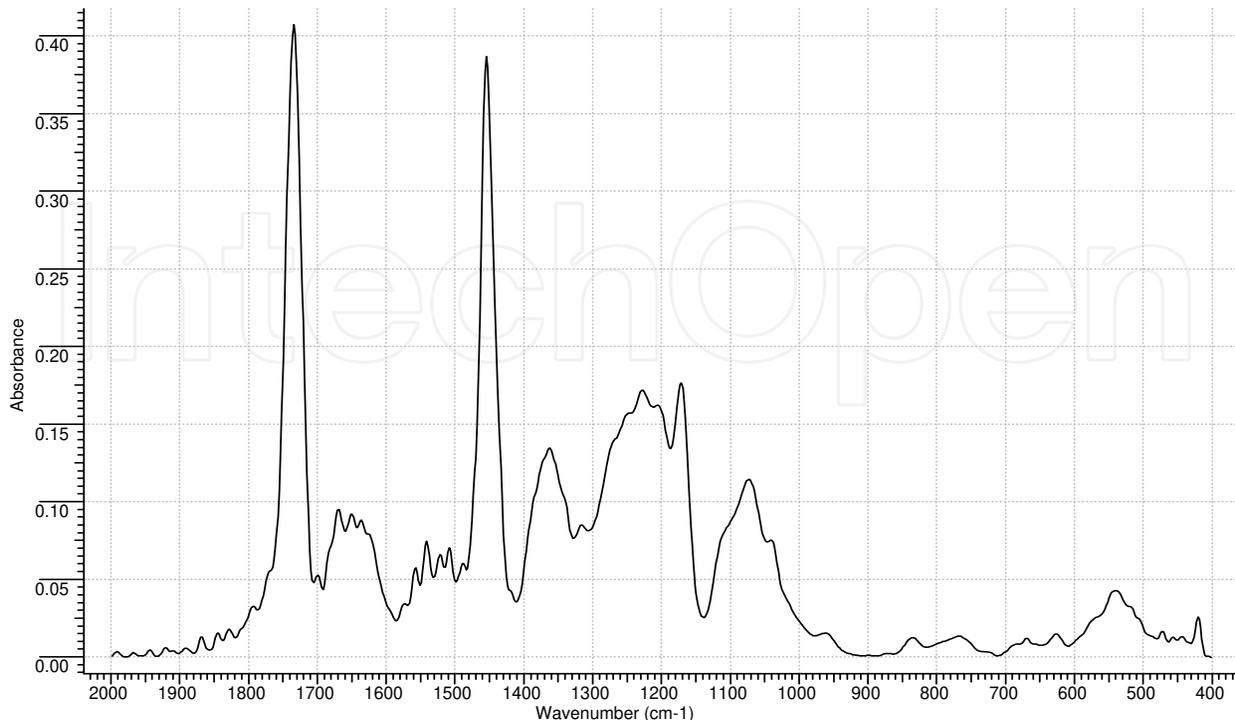


Fig. 4. FT-IR spectrum of PAN fibre „Nitron semi-dull”.

### 3.2 Studies of fiber crystallization grade on the example of PA6 fibers

The assessment of changes in the polyamide fiber structure due to UV irradiation was made [7].

IR analysis was carried out by the transilluminating technique using tablet specimens containing 1% of powdered fiber. IR absorption spectra were recorded within the wavelength range from  $4000\text{ cm}^{-1}$  to  $400\text{ cm}^{-1}$ , in the following systems:  $T = f(1/\lambda)$ ,  $A = f(1/\lambda)$ .

The changes in the crystallinity index values were determined from so-called crystalline absorption bands and the bands of internal standard. The values of fiber crystallinity index  $x_{IR\alpha}$  were calculated using Dechant's formulas [4], namely for modification  $\alpha$   $x_{IR\alpha} = A_{i1030}/A_{i1074}$ , and for modification  $\gamma$   $x_{IR\gamma} = A_{i976}/A_{i1074}$  where:  $x_{IR\alpha}$ ,  $x_{IR\gamma}$  - examined indices of crystallinity for crystallographic forms  $\alpha$  and  $\gamma$  and  $A_{i1030}$ ,  $A_{i976}$ ,  $A_{i1074}$  - integral absorptions of the bands at wave number values  $1030\text{ cm}^{-1}$ ,  $976\text{ cm}^{-1}$  and  $1074\text{ cm}^{-1}$ , respectively.

Table shows the results of assessment of the changes in supermolecular structure of the fiber-forming polyamides based on the changes in crystallinity indices  $x_{IR\alpha}$  and  $x_{IR\gamma}$ . These data characterize the fractions of crystallographic modifications  $\alpha$  and  $\gamma$  in polyamide fibers versus exposure time. So, one can conclude that recrystallisation process takes place in the fibers under the influence of UV radiation, namely so-called crystallographic transformation or the reconstruction of crystallographic lattice from modification alfa ( $\alpha$ ) into gamma ( $\gamma$ ) type modification. In the case of polyamide fiber round “semi-dull”, the fraction of crystallographic form  $\gamma$  increases during the initial

stage of exposure, while the fraction of  $\alpha$  form is decreased. This indicates the crystallographic transformation of more ordered modification  $\alpha$  into modification  $\gamma$  with a lower degree of order. When the exposure time is prolonged the fraction of crystallographic form  $\gamma$  clearly decreases, while the fraction of modification  $\alpha$  decreases only to a slight extent. The most serious decrease in the total crystallinity index  $x_{IR}$  is observed for polyamide fibers round "bright" and round "semi-dull". Clearly lower changes of this index are observed in mat fibers of round cross-section. In these fibers one can observe the clearest process of transformation of crystallographic modification  $\alpha$  into modification  $\gamma$ . The fibers showing triangular cross-sections do not show such drastic changes in crystallinity due to the exposure to UV radiation.

Fiber type	Exposure time (number of summer seasons)	Modification $\alpha$ [-]	Modification $\gamma$ [-]
Polyamide fiber round-"bright"	0	0,27	0,42
	1	0,28	0,33
	2	0,26	0,37
	5	0,25	0,33
Polyamide fiber round-"semi- dull"	0	0,28	0,41
	1	0,24	0,47
	2	0,27	0,34
	5	0,25	0,20
Polyamide fibers round-"dull"	0	0,26	0,32
	1	0,22	0,26
	2	0,17	0,27
	5	0,19	0,35
Polyamide fiber triangle-"bright"	0	0,23	0,29
	1	0,24	0,27
	2	0,25	0,28
	5	0,27	0,28

Table 3. Fractions of crystallographic modifications  $\alpha$ - and  $\gamma$ - in PA6 fibers.

### 3.3 Studies of fiber internal orientation on the example of PA6 fibers

In fibre orientation studies the experimental basis are the absorption spectra of linearly polarised IR radiation applied parallel and perpendicular to the axis of the irradiated fibre. This takes to develop an adequate so-called web preparation being a monofibrous layer of compact and parallel fibres. The study produces two IR absorption spectra with discernible absorption bands correlated with the relevant relative ordered states of macromolecules, so-called "crystalline", "amorphous", and "independent" bands [1,8].

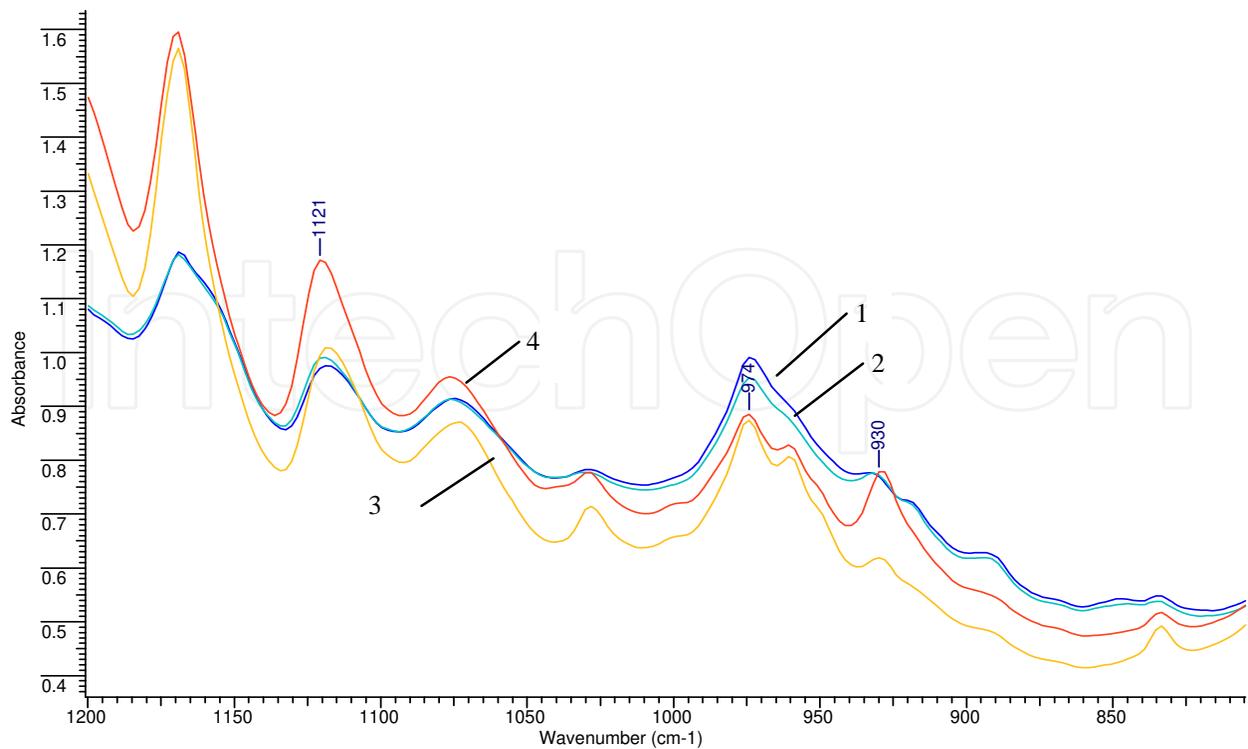


Fig. 5. FT-IR spectra of PA6 fibre: 1) draw ratio  $R=1x$ , parallel direction of IR radiation, 2) draw ratio  $R=1x$ , perpendicular direction of IR radiation, 3) draw ratio  $R=4,7x$ , parallel direction of IR radiation, 4) draw ratio  $R=4,7x$ , perpendicular direction of IR radiation.

This provides the basis for fibre orientation analysis in terms of the ordered state separately for: crystallites, macromolecules in non-crystalline material, and the resultant – as total (fig.5, table 4, 5). The indicator in the quantitative evaluation of fibre orientation is the value of the dichroism quotient  $R$  which expresses the intensity ratio of the dichroic absorption band appropriate for the parallel irradiation of the fibre to the intensity of that absorption band found in the spectrogram for the perpendicular irradiation of the sample. The more the  $R$  value diverges from 1, the higher is the fibre orientation level. The quantitative fibre orientation indicators [1,8] are determined based on certain experimental relationships[1,8].

Wavenumber $\text{cm}^{-1}$	Absorbing group, type of vibration	The type of band	The type of dichroism	Dichroic ratio, $R$	Orientation index $f_{IR}$
1119	$\nu$ (C-C)	o	$\Pi$	1,073	0,024
974	$\nu_0$ , amide IV	a	$\sigma$	0,892	0,075
932	$\nu_0$ (0, $\Pi$ )	c	$\Pi$	2,364	0,313

Table 4. The data for selected dichroic absorption bands: “crystalline”, “amorphous”, and “independent” for initial fibre PA6 draw ratio  $R=1x$ .

Wavenumber cm <sup>-1</sup>	Absorbing group, type of vibration	The type of band	The type of dichroism	Dichroic ratio, R	Orientation index, f <sub>IR</sub>
1119	v (C-C)	o	π	1,313	0,095
974	v <sub>0</sub> amide IV	a	σ	0,692	0,229
932	v <sub>0</sub> (0, π)	c	π	4,889	0,565

Table 5. The data for selected dichroic absorption bands: "crystalline", "amorphous", and "independent" for initial fibre PA6 draw ratio R=4,7x.

#### 4. Studies of changes in fiber structure due to external factors

The IR absorption spectroscopy method used to assess changes in fiber structure is aimed to detect changes in molecular structure and crystalline structure. The measurements may be carried out using the ATR technique, or transilluminating technique. For this purpose, a fibrous sample appropriately positioned in relation to a special prism, or a tablet preparation is used. Both the changes in structure of the fiber volume (transilluminating technique) and the changes of its surface characteristics (ATR technique) are assessed.

##### 4.1 Changes in structure of wool and polyamide fibres

The changes in fiber structure may take place as a result of interactions with many external factors, chemicals, as well as intentional processing aimed at modification of the fiber surface.

For example, assessment may concern the character of fiber-forming material remodeling due to such factors as: exposure to sunlight, heat and humidity, skin secretions – sweat and sebum [8], including:

- changes in fiber-forming material structure in the aspect of its negative effect on the skin due to remodeling of the fiber material, so-called „aging” as a result of various factors, or due to development of mites, for which it constitutes a favorable medium;
- changes of morphological and macroscopic structure – as a mechanical factor causing skin irritation.

The fibers are studied for changes in their structure due to the effect of external factors using IR absorption spectroscopy techniques – ATR and transilluminating technique.

##### 4.1.1 Molecular structure of the wool fibre surface layer and its changes under the influence of the external factors used (example)

The test results of three types of wool fibres with various thicknesses show a clearly different molecular structure of their surface layer. Thinner fibres with a thickness of 15,5 μm or 19,5 μm show similar structures, which results from the comparable absorbance values of absorption bands correlated with the peptide group being characteristic of keratin (fig 6.). A considerably lower absorbance of the band correlated with the peptide group of

the fibre with a higher thickness (25,5  $\mu\text{m}$ ) indicates a lower keratin content in its surface layer. This seems to result from a more developed cuticle layer (greater and thick scales), the structure of which contains a higher quantities of other chemical substances than keratin, e.g. lipids.

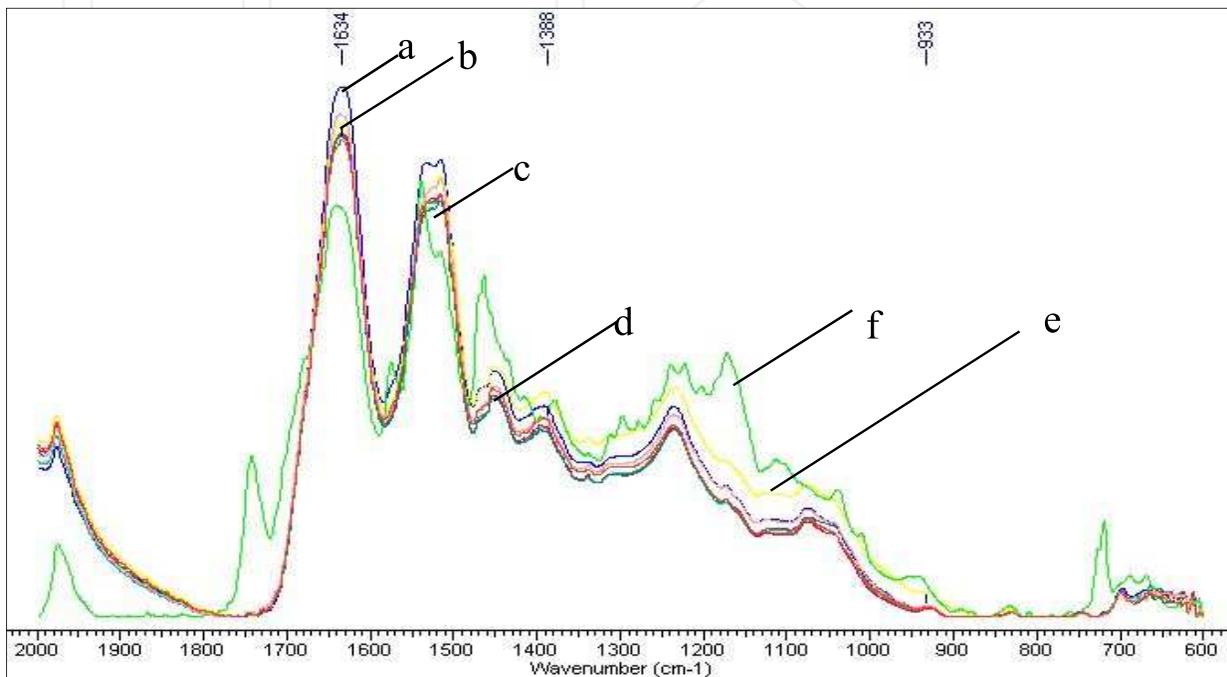


Fig. 6. FT-IR spectra of wool fibre after exposure to external factors: a) initial fibre, b) fibre and alkaline sweat, c) fibre and acid sweat, d) fibre and temp. 37,5, humidity 65%, e) fibre and temp. 37,5, humidity 25%, f) fibre and sebum, g) fibre and UV radiation, 2 seasons

The effect of the external factor used on the keratin depolymerisation is minimal as shown by a low differentiation of value absorbance  $A$  in relation to the absorbance value of the absorption band of  $-\text{CO}-\text{NH}-$  group in the initial fibre. The strongest effect on the keratin molecular structure is exerted by acidic perspiration and heat at RH 65%. In the case of thinner fibres, the decrease in the absorbance value of the absorption band correlated with peptide group is not always accompanied by the increase in the number of  $-\text{COOH}$  and  $\text{NH}_2$  groups. Thus the molecular restructuring process seems to be more complex and concerns first of all the groups occurring in the substituents of  $\alpha$ -amino acids radicals of a acidic and basic character probably resulting from the change in their energetic state as a result of changes in intermolecular interactions due to e.g. the decomposition or formation of new intermolecular bonds as well as the accompanying conformational changes and even the electric state transition (change in the range of isoelectric point) [9].

In the case of thicker fibres, the number of acid and base groups is clearly increased with a slight increase in the number of  $-\text{NH}-\text{CO}-$  groups as indicated by the increased intensity of the corresponding absorption band (increase in the value of absorbance  $A$ ). The impossibility of the unmistakable interpretation of changes in the molecular structure of

wool fibres may however allow one to conclude that these changes take place. Thus it can be assumed that regardless of their character they can influence the interactions between fibre and skin considering e.g. the change in the range of isoelectric point being characteristic of polypeptide fibres, thus the appearance of a more or less specified beneficial interaction resulting from the amphoteric character of keratin.

#### 4.1.2 Molecular structure of the polyamide fibre surface layer and its changes caused by the external factors used

Changes in the polyamide fibre surface layer take place in a different way than those in the remaining fibres. The interpretation principles assumed disappoint in the case of these fibres despite the relatively uncomplicated chemical structure of polyamide (fig. 7).

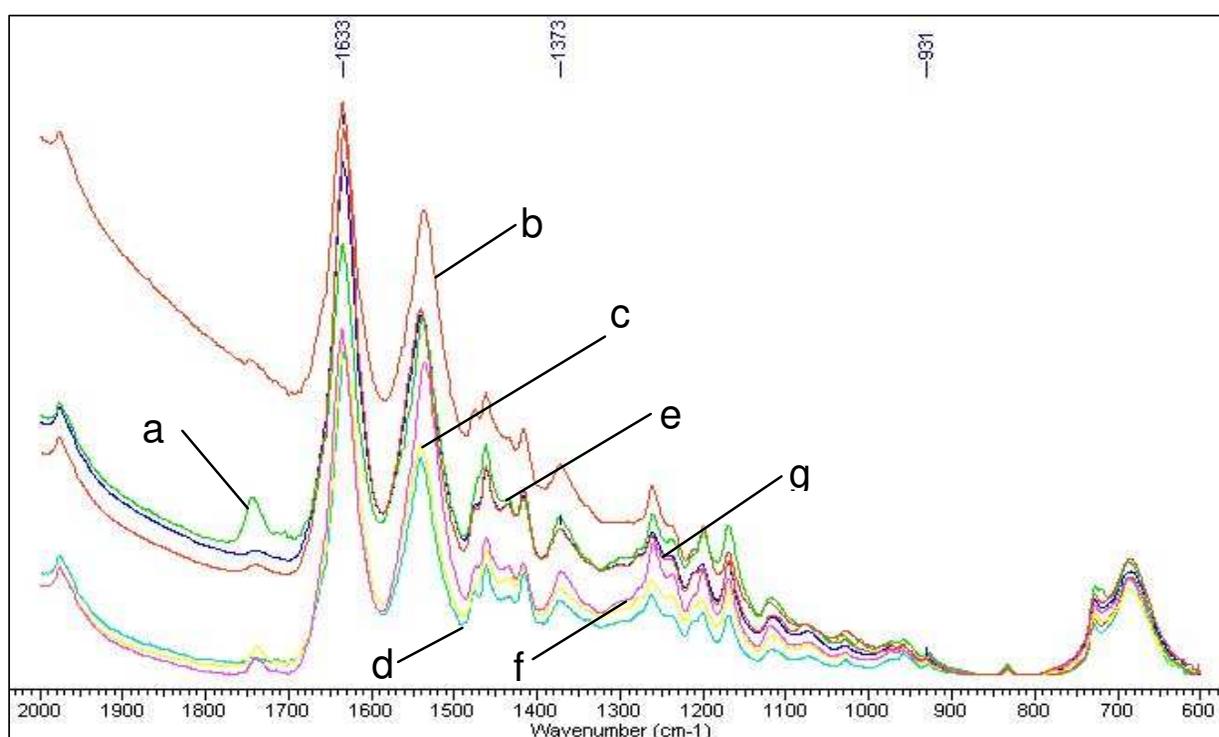


Fig. 7. FT-IR spectra of polyamide fibre after exposure to external factors: a) initial fibre, b) fibre and acid sweat, c) fibre and alkaline sweat, d) fibre and temp. 37,5, humidity 25%, e) fibre and temp. 37,5, humidity 65%, f) fibre and sebum, g) fibre and UV radiation, 2 seasons.

This different behaviour of polyamide fibres under the influence of the factors used seems to result from the occurrence and superimposition of the parameters of the fibre physical microstructure, especially the high degree of crystallisation on the fibre molecular structure. The effects of interaction will be different in relation to the fibre amorphous and crystalline materials. The decrease in the absorbance values of all the absorption bands tested in relation to bands A of the initial fibre in the case of the action of alkaline perspiration can be connected with a strong “etching” of the amorphous surface layer by

alkaline perspiration. Acidic perspiration showing swelling effects on the polyamide material [11] will cause the restructuring of the crystalline material into a non-crystalline matter, releasing characteristic groups from intermolecular interactions, which will be seen in the increase in the intensity of bands correlated with these groups. The assumed mechanism of the effect on restructuring the PA6 fibre surface layer concerns heat and moisture. One may assume that when moisture is lower the effect resolves itself into the removal of low-molecular fractions from the fibre surface, while within the range of high moisture the crystalline matter is restructured with releasing the groups from intermolecular interactions.

#### 4.2 Assessment of surface wool fibres as a result of a biopolymer deposition

Considering the possibility of skin irritation [12, 13] due to a physical mechanism (friction of the fibers against the skin surface), the fibers were subjected to surface modification using a bioactive polymer – chitosan – as a modifier. The modification of wool fibers was carried out by subjecting them to water bath with 5% chitosan content, pH = 5,0 and processing temperature of 80°C for differentiated periods of time of 10 min and 30 min. The studies were conducted on intact fibers, and fibers which had undergone preliminary enzymatic processing with two different enzyme concentrations - 1% and 3% [12].

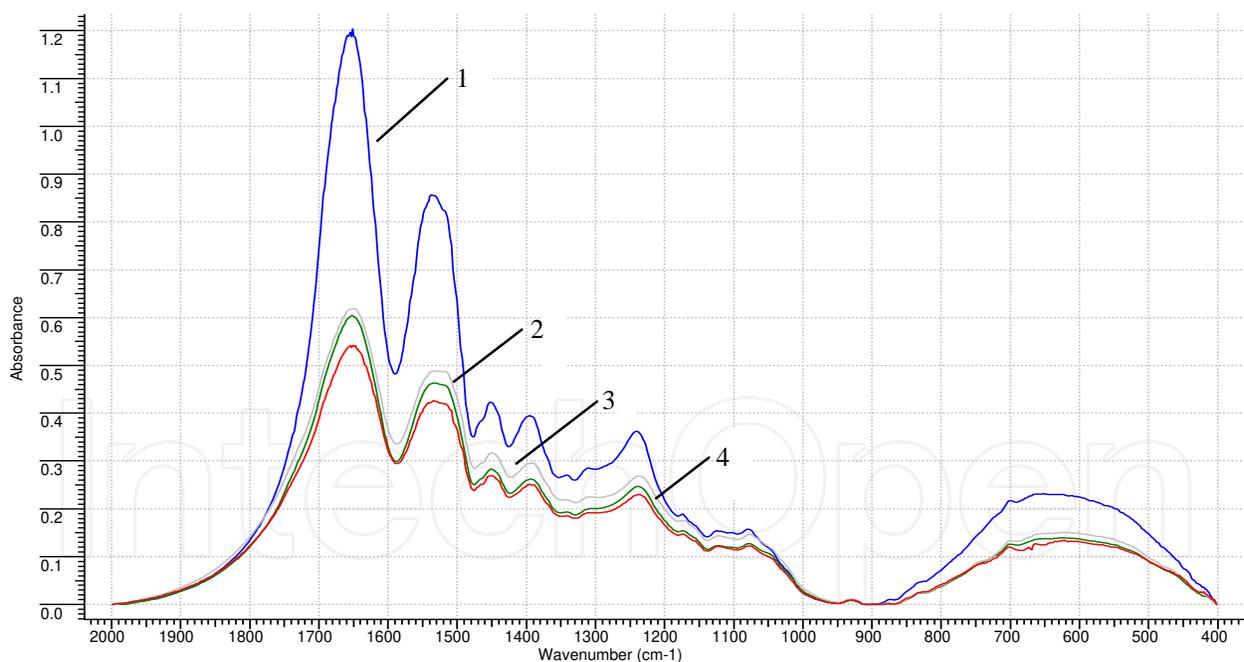


Fig. 8. FT-IR spectra of wool fibre- 25,5µm: 1- initial fibre, 2- chitosane ( $M_2$ ) treated fibre, 3- fibre after enzyme (3%) and chitosane ( $M_2$ ) modification, 4- fibre after enzyme (1%) and chitosane ( $M_2$ ) modification, where:

( $M_1$ ) chitosane preparation - molecular mass  $\bar{M} = 360$  thousand, deacetylation degree (DD) = 76.3 %

( $M_2$ ) chitosane preparation -molecular mass  $\bar{M} = 229$  thousand, deacetylation degree (DD) = 97.0 %

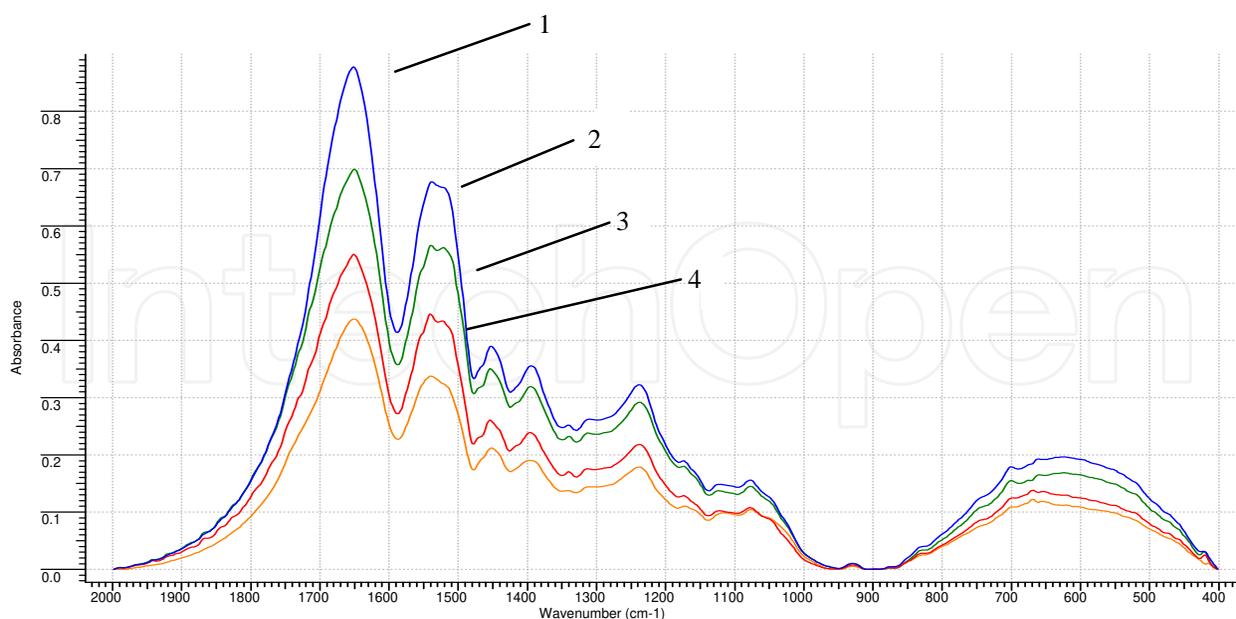


Fig. 9. FT-IR spectra of wool fibre-19,5µm: 1- initial fibre, 2- chitosane ( $M_2$ ) treated fibre, 3- fibre after enzyme- and chitosane ( $M_1$ ) modification, 4- fibre after enzyme - and chitosane ( $M_2$ ) modification, where:

( $M_1$ ) chitosane preparation - molecular mass  $\bar{M} = 360$  thousand, deacetylation degree (DD) = 76.3 %

( $M_2$ ) chitosane preparation - molecular mass  $\bar{M} = 229$  thousand, deacetylation degree (DD) = 97.0 %

The absorption spectra obtained with IR - spectroscopy demonstrate post-processing changes in fibre structure of wool, and polyacrylonitrile fibres. On the basis of the test results, the effect of enzymatic processing on fibre surface, confirmed by fibre mass decrease, can be observed and, in case of chitosan-treated fibres, the presence of bands specific for chitosan, which indicate binding of the polymer to the fibres.

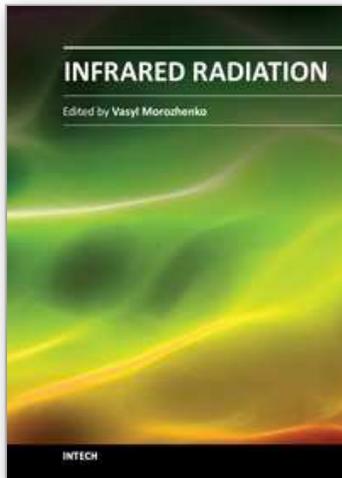
## 5. Conclusion

The possibility of applying IR- spectroscopy analysis in the fibres structure and properties investigations is very broad. On the basis of examples shown it can be concluded, that the IR spectroscopic methods can be used:

- for identification of fibres polymer matter (kind of fibre) [14, 15],
- for investigation of fibres structure parameters as: crystallinity degree [7,16] , inner orientation (molecular orientation in noncrystalline part of fibres matter, crystallites orientation and total orientation- separately),
- for assessment of fibres ageing effects[7],
- for assessment of fibres surface changes under the different action-physical, chemical and biochemical treatments,
- for analysis the kind of substances deposited on the fibres surface during their modification processes.

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This book represents a collection of scientific articles covering the field of infrared radiation. It offers extensive information about current scientific research and engineering developments in this area. Each chapter has been thoroughly revised and each represents significant contribution to the scientific community interested in this matter. Developers of infrared technique, technicians using infrared equipment and scientist that have interest in infrared radiation and its interaction with medium will comprise the main readership as they search for current studies on the use of infrared radiation. Moreover this book can be useful to students and postgraduates with appropriate specialty and also for multifunctional workers.

### **How to reference**

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