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# Measurement of Fibre Humidity Using Spectroscopic Method

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## Abstract

A method of measuring fibre humidity, which allows for investigation of very small masses of samples (in the milligram range) using typical FTIR spectrophotometers has been developed. The method is based on measuring the proportion of bands coming from water in a liquid state and those from the fibre substance with near-infra-red spectra. The quantitative proportionality of the peak area in relation to the water content permits to find a simple relationship which describes the humidity of the samples to be established.

**Key words:** humidity, fibre humidity, spectroscopic method, FTIR.

## Introduction

The phenomena occurring during the process of moisture sorption by textile products are the basic parameters on which their behaviour depends both during processing and use. When the water contents in the fibre changes, almost all the mechanical parameters are changed (tensile strength, stiffness, ultimate elongation, etc.) as well as the physical (electric and thermal conductivity, isolation against UV radiation solubility, etc.) and chemical ones (chemical reactivity, resistance to microbes, etc.) [1]. High moisture retention in the product, within a range exceeding ten per cent, makes the product useful for next-to-skin textiles. Textile products with low moisture retention (below one per cent) are used for the external layers of the garment [2].

The humidity of a textile products is usually determined by means of the weighing method by drying it in the temperature above 100°C and by determining the differences in masses [3]. This method requires much time and the use of samples weighing several grams. Determining the moisture contents in textile products by measuring electric conductivity depends on the surface properties of the fibre, the form of product, the weave density and the parameters of the flowing current [4].

## Aim of the Work and the Method of Carrying Out the Measurements

The aim of the work was to work out a method for measuring fibre humidity using a „Magna 860” spectrophotometer from the Nicolet company configured for the near-infrared range. The configuration lay in the choice of the parameters of apparatus operation including range, resolution, the configuration – of the

source - „beamsplitter” - and the detector, the choice of the number of screens, the equipment of the measuring chamber and the preparation of the samples.

Determining the fibre humidity was carried out by measuring the surface of absorption peaks coming from the adsorbed water and finding their quantitative ratio in relation to the peaks of fibre stuff (independent of its humidity) within the NIR band.

In order to verify the results of the measurements carried out according to the proposal above, the same samples were put in parallel to the determination of water contents using the weighing method; samples of 1 gram in weight were tested which were dried in under calibrated conditions at a temperature of 120°C up to dry mass.

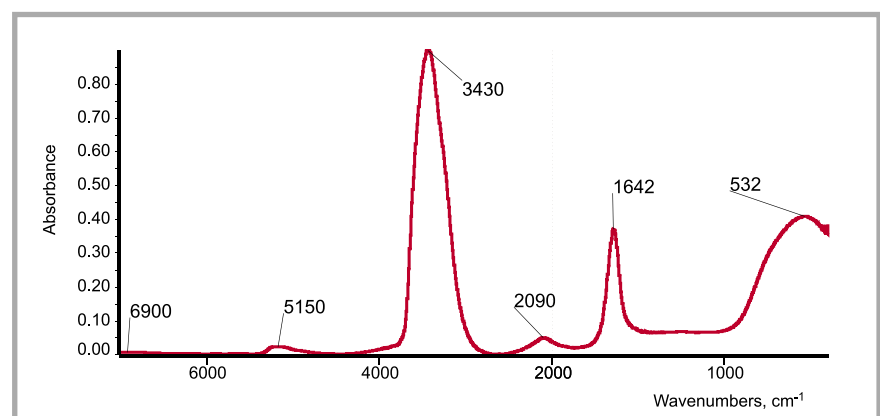
The samples for the humidity measurements using the spectroscopic method were prepared by placing the fibres (weighing approx. 0,0005g) between glass plates, which had been fastened in the grip of colimator in the apparatus’ measuring chamber. Glass plates without samples on them were used as a measure-

ment reference for all the measurements, carried out. It should be emphasised that the separation of samples by the glass plates ensures that any migration of water occur in the measuring chamber.

The measurements were carried out on the conditioned samples in an atmosphere of 60% humidity and 22°C, dried, moisted for 96 h, dampened upon drying and dried upon dampening in the ambient conditions. The measurements of fibre humidity by means of the spectroscopic and weighing methods were carried out in parallel on the same samples.

The basis for the spectroscopic method is the measurements of absorptive peaks for water occurring at 5150 cm<sup>-1</sup>, which do not coincide with the absorptive spectra of most fibre polymers. The linearity of measuring results obtained by the Magma 860 is assured as all results are below the absorbance of 1A and the Magma 860 guarantees linearity up to 2.5 A. The infrared spectra for water in the range of 7000 – to 400 cm<sup>-1</sup> has been shown in Figure1.

The measurement results were processed and worked out using Omnic software.



**Figure 1.** FTIR absorption spectra for demineralization water in the measuring region between 7000 and 400cm<sup>-1</sup>.

## Results of the Investigation

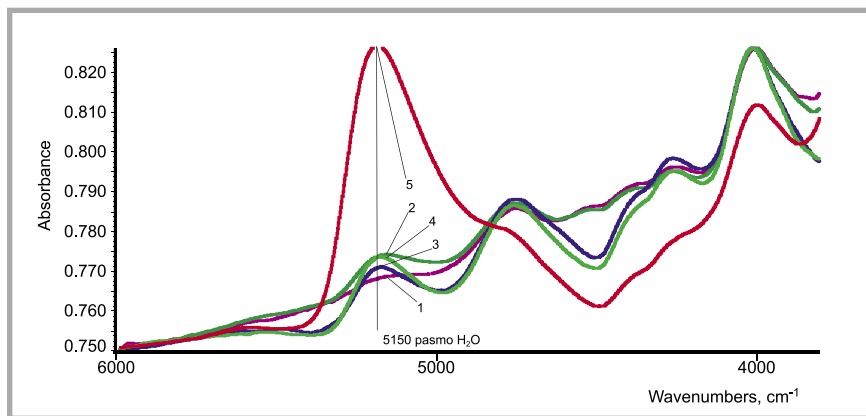
The dependence between the areas under the absorption peaks is proportional to the quantity (concentration) of appropriate oscillators in the sample investigated (the according to the Lambert-Beer law) [4]. The choice of peaks to be measured for the particular fibre depends on its position, intensity and placement of the absorption peaks, i.e. in the bands from about 5000 to 4000  $\text{cm}^{-1}$ . The comparison of surfaces under the appropriate peaks (selected individually for the fibre stuff) should be proportional to the quantity of water absorbed. The humidity measurement can be carried out directly for the samples, for which it was possible to precisely determine the mass of the sample through which the measuring beam passes (e.g. for a liquid). In the case of fibres this is practically impossible and that is why the measurement of humidity must take place in an indirect way on the basis of the preset quantitative ratio between the mass of water and the mass of fibre stuff.

The results of spectroscopic measurements for some selected fibres with different water contents, the comparison of surfaces under the appropriate peaks, the comparative measurements of the humidity of textile products by means of the weighing method, as well as the dependences between these measurements, have all been shown in Figures 2-4, in Table 1 and in the diagrams shown in Figures 5-7.

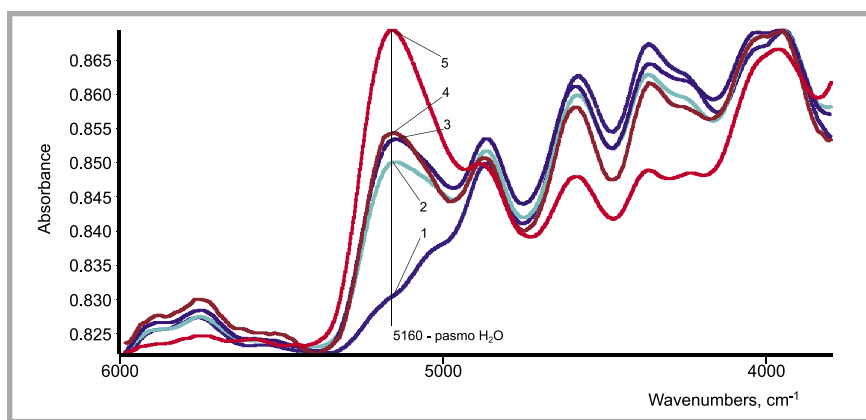
The water-absorptive band comprises several peaks (Figure 1). Water-absorptive bands usually coincide with those of the substances with which they are investigated within medium infra-red range MIR. This makes the precise determination of parameters of these bands difficult or even impossible.

Within the NIR range of the absorption of electromagnetic radiation, the absorption band of water occurs at 5150  $\text{cm}^{-1}$ , which, practically, does not coincide with the NIR bands for fibre polymers. It is clear and well-resolved which makes the measurement of this band's parameters easier. The band intensity for water at 5150  $\text{cm}^{-1}$  is significantly lower than for the remaining bands in the MIR range (the measurement is possible even with significant water contents).

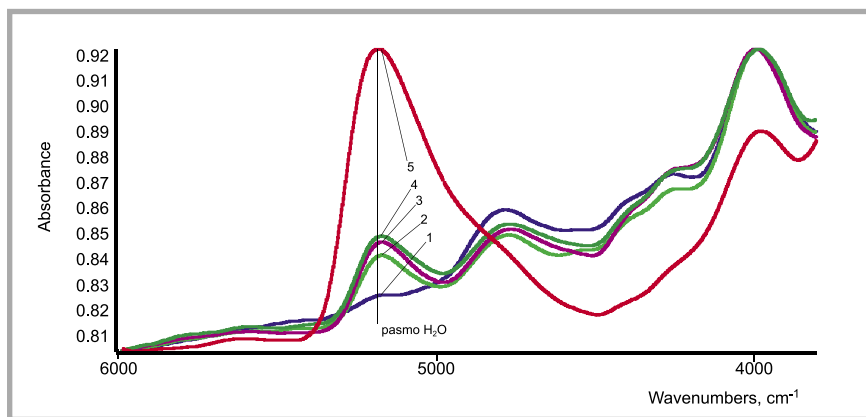
The values of quantitative coefficients related to water contents in relation to fibre stuff as shown in Figures 5-7 remain in



**Figure 2.** Comparison of FTJR absorption spectra in the range of 6000 – 4000  $\text{cm}^{-1}$  for cotton fibres which have a different humidity; cotton: 1- dried at 120 °C, 2- air conditioned after drying, 3- without treatment, 4- dried after soaking, 5- after soaking in water.



**Figure 3.** Comparison of FTJR absorption spectra in the range of 6000 – 4000  $\text{cm}^{-1}$  region for wool fibres which have a different humidity; wool: 1- dried at 120 °C, 2- air conditioned after drying, 3- without treatment, 4- dried after soaking, 5- after soaking in water.



**Figure 4.** Comparison of FTJR absorption spectra in the range of 6000 – 4000  $\text{cm}^{-1}$  region for viscose fibres which have a different humidity; viscose: 1- dried at 120 °C, 2- air conditioned after drying, 3- without treatment, 4- dried after soaking, 5- after soaking in water.

good correlation with the humidity values for the fibres determined by means of the weighing method [5]

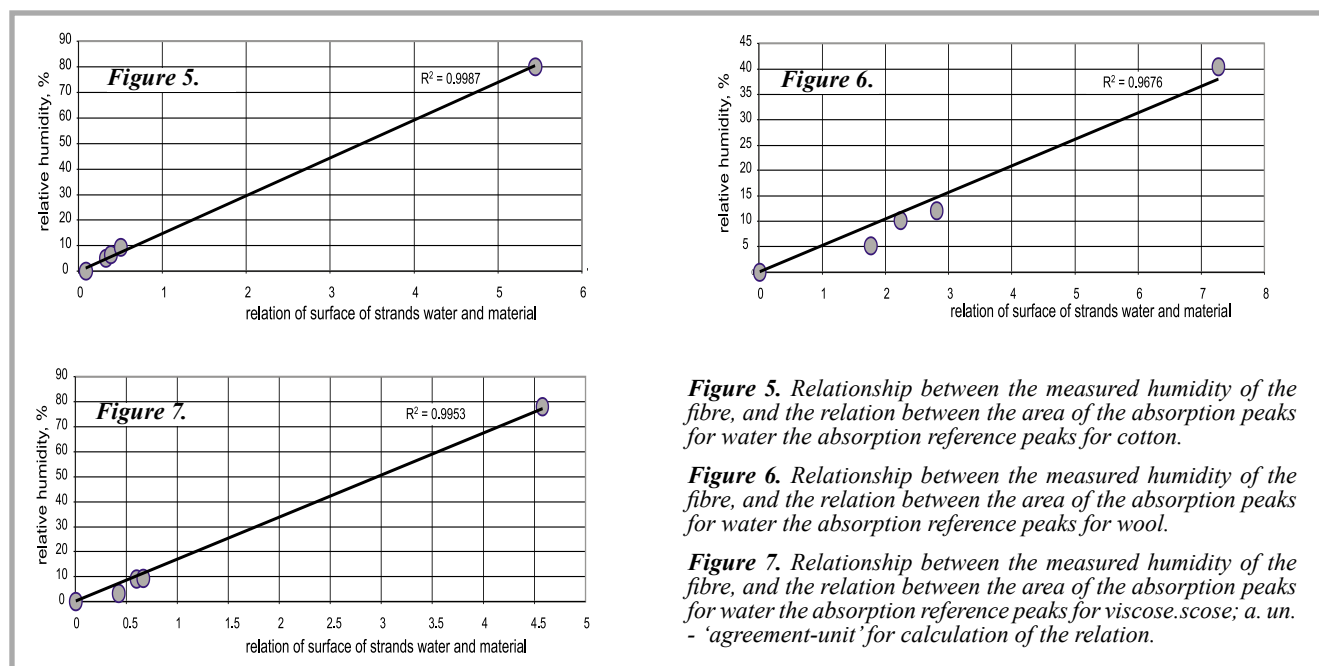
An analysis of the sets of bands NIR for fibres dried at the temperature of 120 °C indicates some residual water in the samples. This is because the drying of fibres in this temperature does not remove

all the water which had been bounded in physico-chemical way.

Spectroscopic investigation by means of the proposed method allows the grade of dryness of the fibres (textile product) to be monitored and the temperature at which the effect of total water removal is obtained to be precisely determined.

**Table 1.** Comparison of measurement parameters for cotton, wool and viscose; a. u. - 'agreement-unit' for calculation of the relation between areas of the absorption peaks of H<sub>2</sub>O and the peaks for cotton, wool, and viscose.

Material	Description of sample	Areas under absorption peaks of H <sub>2</sub> O at 5150 cm <sup>-1</sup>	Areas under absorption peaks at 4015 cm <sup>-1</sup> (cotton) 4595 cm <sup>-1</sup> (wool) 3990 cm <sup>-1</sup> (viscose)	Relation between areas of the absorption peaks of H <sub>2</sub> O and the peaks for cotton, wool, and viscose	Humidity to obtained by the weighing method
		a. u.	a.u.	-	%
COTTON	dried in 120 °C	0.1474	1.6895	0.0872	0.0
	air conditioned after drying	0.5799	1.7728	0.3271	4.87
	without treatment	2.6547	6.9080	0.3843	6.49
	dried after soaking	4.0650	8.0580	0.5045	9.28
	after soaking in water	15.043	2.7620	5.4464	80.17
WOOL	dried in 120 °C	0.0336	2.2651	0.0148	0.00
	air conditioned after drying	6.0887	3.4399	1.7700	5.15
	without treatment	8.2939	3.7012	2.2409	10.09
	dried after soaking	3.6128	1.2848	2.8119	12.14
	after soaking in water	7.5694	1.0411	7.2706	40.48
VISCOSE	dried in 120 °C	0.0217	4.1845	0.0052	0.0
	air conditioned after drying	2.724	6.3751	0.4273	3.42
	without treatment	4.127	6.8588	0.6017	9.0
	dried after soaking	2.922	4.3668	0.6691	9.37
	after soaking in water	22.206	4.8488	4.5797	78.0



Placing the sample in a temperature chamber and carrying out spectroscopic investigations by means of the proposed method allows the observation of the process of water desorption as a function of temperature, even in the case of simultaneous desorption of other substances with a low boiling point, which is impossible to achieve by means of the weighing method.

### Conclusions:

- Fibre humidity determined by means of the weighing method is in good correlation with the results obtained by means of the spectroscopic method.

- For the fibres in low humidity ranges the spectroscopic method enables the determination of residual water contents.
- The spectroscopic method enables the estimation of water contents in the fibres apart from other chemical substances.
- The measurement of fibre humidity can be carried out on the samples with a mass of several thousandths gram.
- The spectroscopic method can be used for the fibres or fibre blends for which the water-absorption band can be precisely determined at 5150 cm<sup>-1</sup>.

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