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CHARACTERIZATION AND IDENTIFICATION OF CHEMICAL AND NATURAL FIBRES BY INFRARED SPECTROSCOPIC METHOD AND COMPUTER

This work is a continuation of a series of articles on the characterization and identification of fibres by the infrared spectroscopic method [1–4], infrared spectrometry and computer [5].

In this paper data are presented about the characteristic properties of infrared spectra of chemical (propylene, polytetrafluoroethylene, polyvinyl alcohol-acetal, viscose, cellulose ester) and natural (cotton, wool and silk) fibres (belonging of absorption bands to one or other atomic group vibration and their intensities). We present here the infrared spectra of their absorption bands as well as intensities of the above fibres.

It should also be noted that we deal mainly with chemical and natural fibres important from the viewpoint of manufacturing textile fabrics.

Polypropylene fibres. K. Ziegler's discovery of organic catalysis of transitional metals for low-temperature and pressure polymerization of alkenes and G. Natta's discovery of stereospecific catalysis permitted, in 1954, the preparation of isotactic polypropylene via polymerization of propylene [9]. At the preparation of polypropylene, besides isotactic polypropylene, also atactic polypropylene is formed, being extracted as an undesirable by-product with boiling n-heptane.

According to K. Ziegler, the polypropylene mole mass is 40,000, boiling point 176 °C, density 0.90 g/cm³ [6]. The isotactic polypropylene alone is fit for the preparation of polypropylene fibre. The 1980 world output of polypropylene amounted to 5 million tons, 28% of which have been used for manufacturing textile fabrics, carpets and sacking.

In the case of strong and very strong absorption bands of infrared spectra of fibres our data correlated (Table 1) well with those given in [7]. At the assignment of vibrations of the atomic groups, data have been taken from the literature. The most characteristic absorption bands of infrared spectra of polypropylene are 841, 842, 974, 975, 998, 1168, 1170, 1376, 1380, 1455, 1457, 1465, 2879, 2920, and 2960 cm⁻¹.

Polytetrafluoroethylene fibres. According to the known method, the monomer tetrafluoroethylene is polymerized in stainless steel autoclaves under pressure in the presence of ammonium peroxydisulphate as a freeradical initiator. The granules formed are collected, washed and dried. The polymer is further extruded [6]. The density of polytetrafluoroethylene is 2.1—2.3, the melting point of the crystalline product is 327°. The polymer is not soluble in conventional solvents. The infrared spectrum of polytetrafluoroethylene contains three strong absorption

Table 1

Absorption bands of infrared spectra of polypropylene fibres, cm⁻¹

Polypro-pylene (USSR)	Meraklon (Italy)	Relative intensity	Assignment [7]
809	810	w	ν_s(CC); r(CH₂)
841	842	m	r(CH)
900	900	w	ν_s(CC); r_b(CH₃)
940	940	vw	r_a(CH₃); r_b(CH₃); ν_b(CC)
974	975	m	ν_a(CH₃); ν_b(CC)
998	998	m	r_b(CH₃)
1045	1045	vw	ν_a(CC)
1100	1100	vw	
1168	1170	m	ν_b(CC) —r_a(CH₃)
1255	1258	w	t(CH₂)
1305	1308	w	ω(CH₂) +δ(CH)
1330		vw	ω(CH) —ν_b(CC)
1358	1360	w	δ(CH) —w(CH₂)
1376	1380	s	δ(CH₃)
1455	1457	s	δ(CH₃)
1465	1465	s	δ(CH₃)
2723	2725	w	
2836	2836	s	ν_s(CH₂)
2879	2879	s	ν_s(CH₃)
2920	2920	s	ν_{as}(CH₂)
2960	2960	s	ν_{as}(CH₃)

Table 2

Absorption bands of infrared spectra of polytetrafluoroethylene fibres, cm⁻¹

Teflon (USA)	Polyphene (USSR)	Assignment [7]
500	500	
555	555	
625	625	w(CF₂); δ(CF₂) specific for crystal structure
640	645	
720	720	
740	740	specific for amorphous structure
770—780	770—780	
1020	1020	
1070	1070	
1155	1155	ν(CF₂)
1200—1250	1200—1250	

bands (Table 2) in the regions 555, 625 and 640 cm⁻¹, which are characteristic of the CF_2 rocking and bending vibration as well as of the crystalline structure. The weak absorption bands 720, 740, 770—780 cm⁻¹ are typical of the amorphous structure.

The absorption bands 1155 and 1200—1250 cm⁻¹ correspond to the CF_2 stretching vibration. The above absorption bands 555, 625, 640, 720, 740, 770—780, 1155, 1200—1250 cm⁻¹ are most characteristic of polytetrafluoroethylene.

Polyvinyl alcohol (-acetal) fibres. Due to its low stability, polyvinyl alcohol cannot be obtained by polymerization of monomeric vinyl alcohol. Polyvinyl alcohol is prepared indirectly by saponification of polyvinyl acetate, different by-products being formed besides the main product, depending upon the degree of polymerization [7]. The fibres prepared from polyvinyl alcohol are insoluble in water. In order to

Table 3

Absorption bands of infrared spectra of polyvinylalcohol(-acetal) fibres, cm^{-1}

Vinol (USSR)	Kuralon (Japan)	Vinylon (Japan)	Solfon (FRG)	Relative intensity	Assignment [7]
800	800	800	—	w	
850	845	850	850	s	δ(CH₂)
920	920	920	920	w	*
1020	1020	1020	—	s	
1070	1070	1070	—	s	ν(CC)
1100	1100	1100	1100	vs	ν(CO); γ(OH) sec
1145	1145	1145	1145	m	**
1180	1180	1177	—	s	ν(CC)
1240	1240	1240	1237	vs	δ(CH)
1330	1330	1330	1330	m	δ(CH)
1360—1395	1360—1395	1360—1395	1360—1385	w	
1415	1415	1415	1415	m	
1445	1445	1445	1445	m	δ(CH₂)
1480	1480	1475	1475	m	
—	—	1640	1640 vw	w	
1715	1715	1715	1710	m	
1730	1725	1725	1730	m	ν(C=O)
2775	2775	2775	—		2×1387
2865	2860	2860	2860	vs	ν(CH₂)
2910	2910	2910	2910	s	ν(CH)
2940	2940	2940	2940	vs	ν(CH)
3400	3400	3400	3400	s	ν(OH)

* syndiotactic configuration, ** cristallinity band.

Table 4

Absorption bands of infrared spectra of wool fibres, cm^{-1}

Wool (Virginia, USA)	Wool (Italy)	Wool (Ireland)	Assignment [7]
3060	3060	3060	
2970	2960	2960	νa(CH₂); νa(CH₃)
2940	2925	2930	
2875	2870	2870	νs(CH₂); νs(CH₂) sh
	1727	1725	ν(C=O) sh
	1710	1710	
1640—1660	1650—1660	1640—1660	amide band I s
	1550	1550	
	1530	1530	amide band II
1510—1540	1512	1512	I
	1503	1503	
1445	1445	1445	δ(CH₂)
1390	1400	1410	νs(COO⁻)
	1382	1385	
		1340	δ(CH)
1230	1230	1235	amide band III
1170	1170	1170	vw
1075	1080	1082	
1040	1035	1035	—SO—oxidized
	720	720	

obtain water soluble fibres, polyvinyl alcohol fibres are treated with formaldehyde (acetalized), approximately 35—40% of the OH-groups being replaced by acetal (—O—CH₂—O) groups [8]. The interpretation of absorption bands of infrared spectra of polyvinyl alcohol is given in

Table 5

Absorption bands of infrared spectra of silk fibres, cm⁻¹

Silk	Assignment [7]	Relative intensity
550	amide groups	
971		
995—1000	polyclycylalanine	
1060—1070		
1160—1170	amide band III	
1220—1230		
1263		
1410		
1445		
1485—1490		
1495		
1505		
1515	amide band II	s
1530—1540		
1550		
1568		
1630—1645	amide band I	
1660		s
1690		
1710		
2875		
2930		
2900—2980	$\nu(CH)$; $\nu(CH_2)$; $\nu(CH_3)$	
3070—3080		
3300	$\nu(NH)$ *trans*	

Table 3. A weak absorption band 920 cm⁻¹ gives evidence of the syndio-
tactic structure. A very strong absorption band 1100 cm⁻¹ corresponds
to the $C=O$ and secondary (OH) group vibration. A strong absorption
band 1145 cm⁻¹ belongs to the crystalline phase of polyvinyl alcohol.
The strong absorption bands 1730 and 1725 cm⁻¹ and weak ones 1360—
—95 cm⁻¹ belong to the remaining vinyl acetate structure. The interpre-
tation of other vibrations is given in the same table. The most character-
istic absorption bands are 850, 1100, 1145, 1240, 1330 and 1445 cm⁻¹.

Wool fibres. The amino acid composition of wool has been found to
contain 18 amino acids in different concentrations [7], the amount of
nine amino acids exceeding 4%. If the infrared spectra of individual
amino acids are easy to take and measure by a medium-resolution
spectrometer, then those of wool, which is a combination of a number
of amino acids, are much more difficult to take.

Three wool samples of different origin were subjected to investi-
gation. The results are presented in Table 4. They have been compared
with those of Rau [7]. An absorption band 1727 cm⁻¹ corresponds to
the carboxylic group $(C=O)$ vibration. According to Rau the absorption
bands 1040 and 1035 cm⁻¹ belong to the SO-group of oxidized wool.

Silk fibres. The infrared spectra of silk and wool fibres are differentiated
mainly by the presence of absorption bands 971, 995—1000 cm⁻¹, which
are characteristic of polycyclylalanine in the case of silk. The spectrum
of wool contains no such bands. An absorption band 550 cm⁻¹ in the
infrared region is also typical of silk, indicating the presence of amido
groups. The absorption bands of silk somewhat coincide with those
reported by Rau (Table 5) [7].

Cellulose fibres (cotton, viscose, polynose, cellulose esters). As the

Table 6

Absorption bands of infrared spectra of cellulose and cellulose ester fibres, cm⁻¹

Cotton (USSR)	Viscose (USSR)	Polynosic (FRG)	Acetate (FRG)	Tricel (England)	Three-acetate (FRG)	Assignment [7]
557			557	557	555—560	
610			605	603	605	
				643		
665					730—753	
			760—790			$\gamma(CH_2)$
			840	840	830—840	
900	898	895	900	875—900	875—900	
990	990	980				
1030			1020	1030	1030	
1060						
			1090	1090	1090	$\nu(C-O)$
1114	1130	1140				
1165	1165	1165	1160	1165	1163	$\nu(C-O-C)$; $\nu_{as}(C-O-C)$
		1210	1210	1210	1210	$\delta(OH)$ in plane
1280		1280	1280	1280	1280	(CH)
1320—1370	1330; 80	1330; 80	1380	1380	1380	$\delta(CH)$
	1410—1450	1410—1450				$\delta(CH_2)$
1430			1440	1440	1440	
1465	1470	1470	1465	1465	1465	δ (OH) in plane
1485	1480	1480	1482	1485	1482	
1632—1644	1640	1640				absorb. H_2O
			1730—1760	1730—1780	1730—1780	$\nu(C=O)$
			2880	2880	2880	$\nu(CH_2)$
2900	2900	2900	2940	2940—2960	2930—2960	$\delta(CH)$
3400	3400	3400				$\nu(OH)$
			3470	3470	3470	abertoon (C=O)

cellulose structure has been thoroughly investigated earlier by the X-ray and infrared spectroscopic methods, we shall not deal with this problem any more. We shall only compare the essential absorption bands of infrared spectra of cellulose for characterizing and identifying them by a computer.

Table 6 shows and compares the absorption bands of natural and chemical cellulose fibres determined by O. Kirret et al. with those given in [7]. An absorption band 557 cm⁻¹ is highly typical of α-cellulose or cellulose I, disappearing in the spectrum of hydrate celluloses (viscose and polynosic fibres). The absorption bands 1165 and 1465 cm⁻¹ are present in the spectra of all cellulose fibres, thus belonging to the basic structure vibration. The absorption bands 1730—1760, 1730—1780 and 2880 cm⁻¹ are typical of cellulose ester fibres, corresponding to the C=O vibration. The latter band is absent in the spectrum of cotton and viscose fibres. The cellulose fibres (cotton) are also characterized by the absorption bands 557, 665, 900, 1165, 1320, 1465, 1480 and 1640 cm⁻¹. The cellulose ester fibres are characterized by the absorption bands 557, 605, 840, 900, 1090, 1165, 1380, 1465, 1745 and 2880 cm⁻¹.

Table 7

Some examples for identification of chemical and natural fibres with computer

Number of fibre	Absorption band, cm⁻¹	Non-essential wave-number KTUN	Essential wave-number KKE	Coincidence KNR	Identified fibre
1	612, 640, 690, 758, 832, 967, 1194, 1255, 1334, 1384, 1430, 1430, 2855, 2912, 2970	3	14	41	polyvinylchloride
2	560, 630, 640, 725, 740, 775, 1080, 1160, 1240	3	8	44	polytetrafluoroethylene
3	845, 980, 1000, 1100, 1172, 1260, 1310, 1330, 1380, 1460, 1470, 2725, 2840, 2880, 2920, 2960	5	11	47	polypropene
4	900, 1065, 1130, 1140, 1165, 1250, 1320, 1435, 1532, 1550, 2900	1	5	50	cellulose (viscose)
5	1100, 1140, 1180, 1240, 1330, 1370, 1415, 1440, 1715, 2780, 2865, 2910, 2942	4	8	62	polyvinylalcohol (-acetal)
6	850, 980, 1000, 1068, 1160, 1230, 1260, 1400, 1450, 1656, 1698, 2941, 3096, 3290	2	9	57	natural silk
7	1091, 1120, 1237, 1385, 1410, 1445, 1452, 1530, 1550, 1663, 1725, 2870, 2900, 2925	3	10	59	wool

Table 7 presents examples of using the computer for identification of fibres.

Summary

Infrared spectrometric method was used for characterizing chemical and natural fibres. The wave numbers of absorption bands of infrared spectra were used to compile a computer programme, by means of which different individual and natural fibres can be identified.

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MÕNINGATE KEEMILISTE JA LOODUSLIKE KIUDAINETE ISELOOMUSTAMINE JA IDENTIFITSEERIMINE INFRAPUNASE SPEKTROMEETRIA JA ELEKTRONARVUTI ABIL

Artiklis on vaadeldud polüpropüleeni, polütetrafluoroetüleeni, polüvinüülalkoholatsetaali, viskoosi, tselluloosestri ning puuvilla, villa ja siidi iseloomustamise ja identifitseerimise võimalusi nende infrapunaste spektrite absorptsiooniribade intensiivsuse alusel.

Samade autorite varem koostatud arvutiprogrammi alusel on võimalik identifitseerida erinevatesse klassidesse kuuluvaid individuaalseid kiudaineid.

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ХАРАКТЕРИСТИКА И ИДЕНТИФИКАЦИЯ НЕКОТОРЫХ ХИМИЧЕСКИХ И ПРИРОДНЫХ ВОЛОКОН МЕТОДОМ ИК-СПЕКТРОМЕТРИИ С ИСПОЛЬЗОВАНИЕМ ЭВМ

Рассматриваются возможности получения характеристик конкретных химических и природных волокон методом ИК-спектрометрии на основании очень сильных, сильных и средних полос поглощения в их спектрах. По заранее составленным программам для ЭВМ идентифицированы различные индивидуальные волокна, в частности полипропилен, политетрафторэтилен, поливинилалкоголь-ацеталь, вискоза, эфирцеллюлоза, а также хлопок, шерсть и шелк.