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CHARACTERIZATION AND IDENTIFICATION OF POLYESTER FIBRES AND THEIR MODIFICATIONS BY INFRARED SPECTROMETRIC METHOD

The production of polyester fibres has attained the first place in the world output of synthetic chemical fibres, leaving polyamide fibres in the second and polyacryl nitrile fibres in the third place. Commercial polyester fibres are known under many hundred trade names, hence it is of interest to find corresponding suitable methods for identifying them.

According to the International Standardization Organization's (ISO) recommendation R 2076 [1], polyester fibres are synthetic fibres from linear high-molecular compounds formed by esterification of dicarboxylic acids with glycols or other alcohols, in the chain of which there is 85 wt. % polyester formed from diol and terephtalic acid. Polyesterether fibres are such high-molecular compounds that have been formed by straight-chain polycondensation of hydroxy benzoic acid and some diol [2]. Besides the polyester fibres obtained from terephtalic acid and ethylene glycol, they have been lately produced this way that at polycondensation diol 1,4-dimethylolcyclohexane [3] has been used instead of ethyleneglycol. Polyester fibres Kodel and Vestan thus obtained have somewhat different physical constants (m. p. 293 °C, density 1.23 g/cm³, etc.).
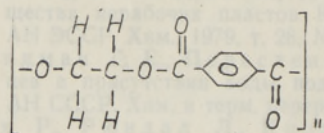

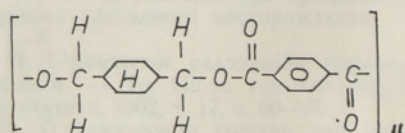Fig. 1. Polyethylene glycol terephtalate (abbr. polyethylene terephtalate).

Fig. 2. Poly-(-1,4-dimethylol)-cyclohexane terephtalate (abbr. polycyclohexane terephtalate).

The aim of this work was to find the possibilities of characterizing and identifying polyester fibres of different chemical structure and origin with the help of the infrared spectrometric method. Infrared spectra were taken on a UR-10 using the KBr pellet method.

The following fibres were subjected to analysis (analyst R. Soosalu):

1. Polyethylene glycol terephtalic fibres (abbr. polyethylene terephtalate): (a) Lavsan, USSR, (b) Trevira, FRG.

2. Polyethylene glycol terephtalate fibre by esterification of ethylene glycol and acids were used: 90% terephtalic acid and 10% isophtalic acid, Stuttgart, Institute of Chemical Fibres, FRG.
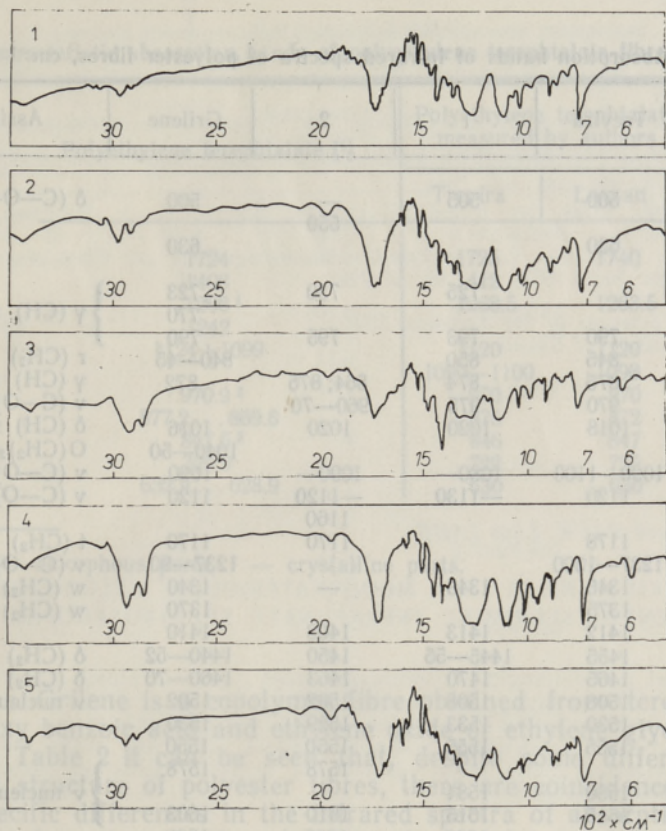
Fig. 3. The infrared spectra of some polyester fibres: 1, 2 — polyethylene glycol terephtalate, Lavsan (1), Trevira (2); 3 — polyethylene glycol terephtalate (90%) + isophtalate (10%); 4 — polycyclohexane terephtalate; 5 — polyesterether (Grilene).

3. Poly-(1,4-dimethylol-)-cyclohexane terephtalate fibre (abbr. polycyclohexane terephtalate), Stuttgart, Institute of Chemical Fibres, FRG.

4. Polyesterether, Grilene, Switzerland.

Elemental analysis of some polyester fibres are presented in Table 1.

Table 1

Elemental analysis of some investigated polyester fibres

Polyester fibres	C	H	O from difference
Polyethylene terephtalate Terylene type:			
Lavsan	62.80	4.18	
Trevira	62.31	4.12	
Polycyclohexane terephtalate Kodel type:			
Vestan X 16	70.44	6.77	
Kodel II	71.12	6.82	
Polyesterether			
Grilene	62.74	4.16	

Table 2

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

Lavsan	Trevira	1	2	Grilene	Assignment
503	500	505	—	500	δ (C—O—C)
			550		
630	630			630	
705					
725		725	730	723	⎫
				770	⎬ γ (CH) nucleus
792	790	793	795	790	⎭
847	845	850		840—45	r (CH$_2$) (*trans*)
872	875	874	864; 875	872	γ (CH) nucleus
970	970	975	960—70		v (C—O) (*trans*)
1018	1018	1020	1020	1016	δ (CH) nucleus
1040				1040—50	O(CH$_2$)$_2$O (gauche)
1098	1090—1100	1080—	1090—	1090	v (C—O) (gauche)
1120	1120	—1130	—1120	1120	v (C—O)
			1160		
1178	1178		1170	1170	t (CH$_2$) (gauche)
1237—90	1237—1300	1348		1237—90	v (C—O)
1343	1345		—	1340	w (CH$_2$) (*trans*)
1370	1375			1370	w (CH$_2$) (gauche)
1410	1412	1413	1408	1410	
1440—55	1455	1445—55	1450	1440—52	δ (CH$_2$)
1468	1465	1470	1463	1460—70	δ (CH$_2$) (*trans*)
1505	1508	1508	1502	1502	v nucleus
1530	1530	1533	1529	1530	
1550—54	1555	1555	1550	1550	⎫
			1578	1578	⎬
1582	1580	1584			⎬ v nucleus
1610	1615	1615	1610	1603	⎭
1630	—	1633	1630	1630	
1645	—	1649	1640	1640	
1678	—	1682	1673	1673	
1690	—	1697	1690	1690	
1710; 25	1712	1715	1707; 1720	1705; 1720	v (C=O)
1740	1725; 40	1730	—	1735; 1750	
		1780—90			
1960	1955			1960	
	2905			2900	v$_s$ (CH$_2$)
		2920	2920		
2965	2965	2965		2960	v$_a$ (CH$_2$)
3050		3055		3040—50	v (CH) nucleus
3425	3425	3430	3410—20	3420	2xv (C=O)

1 — Polyethylene glycolphtalate (terephtalate 90% + isophtalate 10% fibre).
2 — Poly-(1,4-dimethylol-)-cyclohexane terephtalate fibre.

A fa Hewlett-Packard analyzer Model 185 was used for elemental analysis, the weighed portion was 0.7 mg. Columns 1 and 2 in Table 2 present the absorption bands of infrared spectra of polyethylene glycol terephtalate fibres (Lavsan, Trevira), column 3 — of polyethylene glycol terephtalate 90% and isophtalate 10%, column 4 — those of poly-(1,4-dimethylol)-cyclohexane terephtalate and column 5 — of polyesterether fibres. The presence of clear absorption bands corresponding to the ester structure and benzene ring is characteristic of the infrared spectra of polyethylene glycol terephtalate. The —O(CH$_2$)$_2$O group present in a polymer chain unit may be either in a gauche- or trans-form as two-rotation

Table 3

Characteristic absorption bands of polyethylene terephtalate fibres, cm⁻¹

Polyethylene terephtalate [4]	Polyethylene terephtalate measured by authors	
	Trevira	Lavsan
1724	1725	1740
1408	1412	
1266 [1]	1268.5	1263.5
1242		
1124 + 1099	1120	1120
	1090—1100	1098
970.9 [2]	970	970
877.2 ... 869.6	875	872
847.5 [2]	846	847
729.9	722	725
632.9 ... 628.9	630	630

N o t e: [1] — amorphous parts, [2] — crystalline parts.

structures. Grilene is a copolymer fibre obtained from terephtalic acid, paraethoxy benzoic acid and ethylene oxide or ethylene glycol [4].

From Table 2 it can be seen that, despite some differences in the chemical structure of polyester fibres, there are coincidences as well as some specific differences in the infrared spectra of absorption bands. So the infrared spectra of polycyclohexane terephtalate contain no absorption bands in the regions 630, 850, 1040, 1080, 1345, and 2965 cm⁻¹. Table 3 shows the most characteristic absorption bands of polyethylene terephtalate.

This fact may be used for characterizing and identifying polyethylene terephtalate and polycyclohexane terephtalate type synthetic fibres. Data about the infrared spectra of polyesterether fibre Grilene presented in column 5 of Table 2 generally agree well with infrared spectroscopic data on polyethylene terephtalate (except for some insignificant differences), owing to which it may be said that there is a similarity between the chemical structure of the two polymers. The absence of the absorption band corresponding to the —O(CH₂)₂O group in a polymer chain unit in infrared spectrum is in common for both the polymers. It may be said that a chain unit of the experimentally produced polymer contains aromatic groups in considerable amounts; at polycondensation, aromatic starting monomers were used.

In our previous work [5] it was shown that by pyrolysis gas chromatographic method it is possible to qualitatively distinguish polyester fibre modifications by the peaks in different positions on a chromatogram. Maximum values (or characteristic absorption band values) may easily be computed.

In conclusion it may be said that polyester fibres and their different chemical modifications can be characterized by infrared spectra and corresponding vibrations.

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POLÜESTERKIUDAINE JA TEMA MODIFIKATSIOONIDE ISELOOMUSTAMINE NING IDENTIFITSEERIMINE INFRAPUNASE SPEKTROSKOOPIA ABIL

Artiklis käsitletud polüetüleenglükoolterftalaadi tüüpi (terüleeni tüüpi) kiudaine ja tema modifikatsioonide infrapunaste spektrite absorptsiooniribad on paljudel juhtudel sarnased, kuid mõnel modifikatsioonil on ka erinevusi. Kiudude identifitseerimisel tuleb arvesse võtta mõningaid elemendianalüüsi andmeid; näiteks käib see polü(1,4-dimetülool)-tsükloheksaanterftaalkiu kohta, mille süsiniku- ja vesinikusisaldus on teiste polüesterkiudude omast märksa kõrgem.

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ХАРАКТЕРИСТИКА И ИДЕНТИФИКАЦИЯ ПОЛИЭФИРНЫХ ВОЛОКОН И ИХ МОДИФИКАЦИЙ МЕТОДОМ ИК-СПЕКТРОСКОПИИ

Сняты и частично интерпретированы инфракрасные спектры полиэтиленгликольтерефта-левых (лавсан, тревира) (90%) и изофталевых (10%) поли(1,4-диметилол)циклогексан-терефталевых (типа кодел) и полиэстерэфирных волокон (грилен).

ИК-спектры полиэфирных волокон и их модификаций имеют во многих случаях аналогичные полосы поглощения, но для отдельных модификаций свойственная поли-эфирному волокну полоса поглощения отсутствует.

В случае идентификации поли(1,4-диметилол)-циклогексантерефталевых волокон учитываются данные элементного анализа, поскольку содержание углерода и водорода в них резко отлично от содержания углерода и водорода в других полиэфирных во-локнах.