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Fibers of Synthetic Origin: An Analytical Approach to Their Composition

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INTRODUCTION

The "Textile Industrial Revolution" ushered in changes in dress and the consumption of fashion. To meet the public's demand for new products and trends, the clothing industry required faster textile processing and an increase in raw materials at lower cost. These requirements led the chemical textile industry to experiment with alternatives to natural fibers, leading to the invention of new materials with trade names such as Tergal®, rayon, Meraklon®, and Lycra®.

The arrival of synthetic fibers caused a revolution in the fashion world, giving more consumers access to clothes that were once affordable only for the privileged classes. In addition, synthetic fibers became part of the creative process of many designers, who experimented and created new trends with these new materials.[1]

Costumes and textiles first entered museums as objects of consumption with ethnographic, historical, or documentary value. Not until the end of 20th century did fashion begin to acquire aesthetic value, achieving the status of "masterpiece" (Fig. 01). Nowadays, some designers create entire collections to be exhibited in galleries and museums instead of on the runway. In this sense, fashion arrived at museums, regardless of materials, dating, or any other type of information commonly associated with museum objects. For example, in Spain some contemporary designers donate a costume from every collection to the Museo del Traje (Costume Museum) in order to increase its modern collection.

Fig. 01. Advertisement. Tights: Medias Janira. AMA Magazine. 1964. Image by María López Rey©.

When museums catalog such acquisitions, the nature of the synthetic fibers is not usually specified. Fibers of natural origin are easily distinguished with an optical microscope, which allows the study of morphology. However, morphological study is not enough in the case of synthetic fibers.

During the first half of the 20th century, a specific extruder nozzle was used for each type of fiber, resulting in a specific cross-section. Cross-sectional shape is thus useful to identify some fibers, but unfortunately this characteristic is not a special feature of every type of fiber.[2]

Synthetic and semisynthetic fibers are polymers with specific chemical characteristics. Different techniques of analysis are therefore required to identify their chemical composition. This information is essential for the complete documentation of garments in a museum collection. Fiber composition can help to date pieces and can even reveal previous restorations.[3] In addition, this information is necessary to implement an appropriate preservation plan and, when necessary, direct conservation treatments based on the chemical composition of the fiber.[4]

MATERIALS & METHODOLOGY

The aim of this project was to develop a methodology for determining the chemical composition of synthetic and semisynthetic fibers frequently used in the textile industry and in fashion design. The analytical techniques used were Fourier transform infrared spectroscopy with attenuated total reflectance (ATR-FTIR) and pyrolysis-gas chromatography/mass spectrometry (Py-GC/MS).

The ATR-FTIR technique is suitable for identifying synthetic polymers. By measuring the radiant energy associated with the vibrations of molecular bonds caused by infrared (IR) radiation and then studying the absorption bands on the resulting spectrum, researchers can identify a material's chemical composition.[5]

FTIR spectra were obtained with a Thermo Nicolet spectrometer (model 380) with DTGS detector (4,000–400 cm^{-1}), using 64 scans and a nominal resolution of 4 cm^{-1}. The spectrometer was equipped with an ATR diamond crystal accessory.

The Py-GC/MS technique has been successfully used in the conservation field to identify a wide range of synthetic polymers.[6] This technique can provide additional information about the chemical composition of polymeric matrices and can identify minority components such as plasticizers.

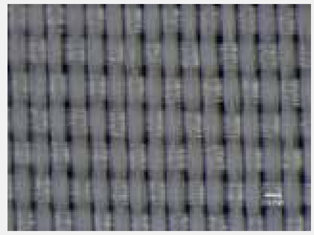
FIBRE (FABRIC OR THREAD)	IMAGE (STEREOSCOPIC MICROSCOP)
Rayon (fabric)	
Polyester (thread)	
Poly (propylene) (fabric)	
Nylon (thread)	
Polyacrylic (thread)	

Table 01. Synthetic and semisynthetic fibers analyzed for this project

The Py-GC/MS analyses were carried out with an integrated system comprising a Pyroprobe CDS 520 pyrolyzer, an Agilent 7890A gas chromatograph equipped with an HP-5MS 5% phenyl methyl siloxane capillary column (30m x 250 μm x 0.25 μm), and an Agilent 5975C mass spectrometer (MS) with a quadrupole mass analyzer. The pyrolyzer transfer line was set at 290ºC and the injector at 280ºC. The GC/

MS experiments were carried out by split injection and detected in the range m/z (29 to 550).

This paper presents results corresponding to two fabrics and three threads. According to the information provided by the suppliers, the fabrics were made of polypropylene and rayon, and the threads were polyester, nylon, and acrylic. The synthetic fibers exist under various trademarks; for example, polyacrylic fibres (Orlon®, Acrylan®, Crilenka®, Creslan®, Dolan®, Zefran®, and Crylon®); polyester (Tergal®, Terylene®, Terlenka®, Trevira®, Dracon®, and Terital®); polyamide (Perlon® and Enkalon®); and so on.

Images obtained via a stereoscopic light microscope (Table 01) show that the fabric made of rayon had a plain weave structure and that the polypropylene was a nonwoven fabric. We were unable to determine whether the latter had two sides or just one.

RESULTS & DISCUSSION

Results of the research are summarized in Table 02.

ATR-FTIR of the rayon fabric showed that it is a cellulosic semisynthetic fiber: cellulose acetate. Composition was confirmed by Py-GC/MS, the peak corresponding to acetic acid formed by the fragmentation of cellulose acetate.

The term *rayon* is sometimes associated with regenerated cellulose,[7] but in this case the results confirmed that the fabric composition was cellulose acetate.

The ATR-FTIR bands for the polypropylene sample confirmed the fibers corresponded to this polyolefin.[8] In the Py-GC/MS analysis, a peak linked to 1-propene was observed. This monomer is commonly used in the manufacture of polypropylene.

Analysis confirmed that composition was the same on both sides of the polypropylene fabric (Fig. 02).

The ATR-FTIR spectrum for the nylon threads revealed bands assigned to polyamide. The Py-GC/MS analysis complemented this result and indicated the type of polyamide: ε-caprolactam, a monomer used to produce nylon 6 (Fig. 03).

In the case of the polyester, the bands in the FTIR-ATR spectrum coincided with the aromatic ring, allowing us to conclude that it is a poly(ethylene terephthalate) (PET). The Py-GC/MS analysis identified fragments associated with PET.

The ATR-FTIR spectrum of the polyacrylic thread showed a defined band of C≡**N** group, assigned to acrylonitrile, the monomer used to produce polyacrylic fibers. Bands from a carbonyl group [C=O] and the functional group ester [C-C (O)-O] and [-O-C-] were also observed, showing that the polyacrylic fiber studied is a copolymer of an acrylonitrile and an acrylate. However, the Py-GC/MS analysis for polyacrylic thread failed to detect fragments associated with this copolymer.

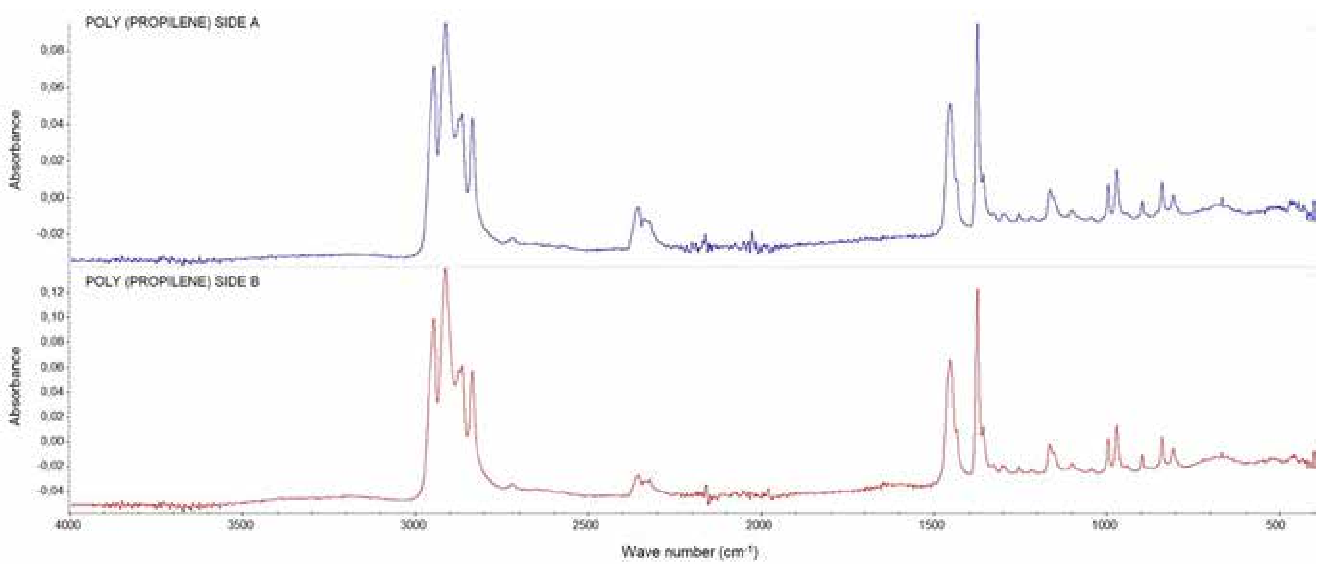


Fig. 02. ATR-FTIR spectra of both sides of polypropylene fabric. Image by LabMat©.

FIBRE	ATR- FTIR	PyCG/MS		IDENTIFICATION
Commercial name	ATR-FTIR absorption bands (cm^{-1})	m/z	Compound	Polymer type
Rayon (Fabric)	Celullose: –OH: 3475 $–CH_3$: 2920 –CH: 1366 –C–O–C: 1160 Acetate: C=O: 1735 C–O: 1213 and 1029 O–C–O: 600 $–C(O)–O^-$: 1431	29, 43,60 32,41,69,100 43,73	Acetic Acid. Metil ester insaturate acid. Propyl ester acetic acid.	Celullose acetate (CA)
Polyester (Thread)	$–CH_2–$: 2962 and 2917 C=O (ester): 1708 [C–C(O)–O] (ester): 1256 [–O–C–](ester) :1174 and 1092 Aromatic ring: C–H aromatic ring : 1016, 970, 871 and 720	32,40,44 29,44	Acetaldehyde Dimethyl ether	Poly (ethylene terephtalate) (PET)
Poly (propylene) (Fabric)	CH_3: 2949 and 2867 CH_2: 2915 and 2836 C-H ($–CH_2–$): 1455 and 1358 C-H ($–CH_3–$):1375 $–CH_3$: 1166 and 972 $–CH_2–$: 997, 898 and 840	41 29,43,57 29,41,56,69,84 29,43,55,70,83,126	1–propene Pentane 2, methyl, 1pentene 2,4–dimethyl heptene	poly (propilene) (PP)
Nylon (Thread)	–NH: 3295 cm^{-1} Amide II overtones: 3067 CH_2– 2931 and 2860 Amide I: [C=O]: 1630 Amide II and Amide III (–NH and C–N): 1541 and 1262 C–H : 1460, 1415 and 1374 Amide IV [C(O)–N–H]: 681	30, 42,55,85,113	ε–caprolactam	Polyamide. (PA)
Polyacrylic (Thread)	$–CH_2–$: 2919 and 2850 C–H: 1452 and 1367 Acrylonitrile: C≡N: 2241 Acrylates: C=O: 1735 [C–C(O)–O]: 1231 [–O–C–]: 1069	No results	No results	Copolymer acrylonitrile–acrylate

Table 02. FTIR-ATR absorption bands and m/z characteristic of Py-CG/MS present in the analyzed materials; materials identified

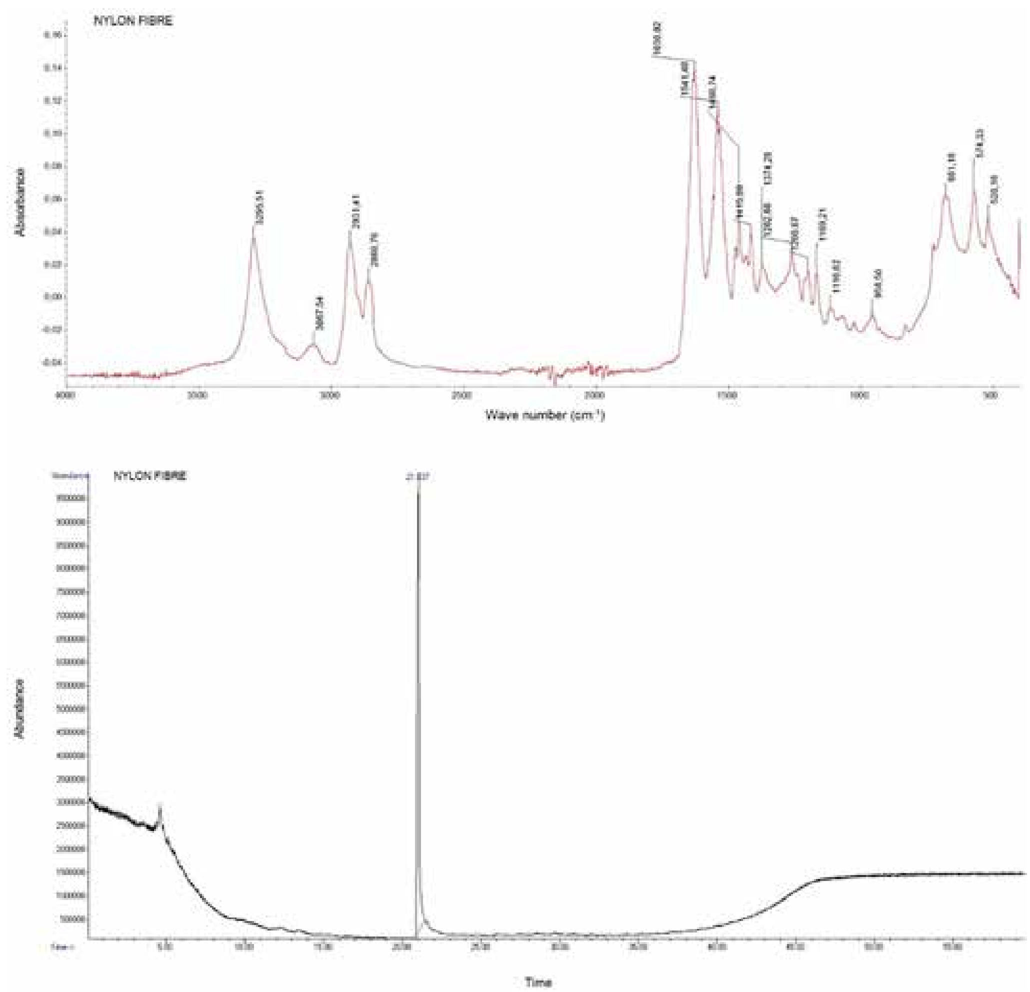


Fig. 03. ATR-FTIR spectra and pyrogram of nylon fiber. Image by LabMat©.

CONCLUSIONS

ATR-FTIR is a useful technique for analyzing fibers, threads, and fabrics that are part of cultural heritage. The main advantages of this method are that the samples do not need to be prepared and results are obtained simply by pressing the fiber or thread against the ATR diamond crystal.

The ATR-FTIR and Py-GC/MS techniques are complementary. In some cases, Py-GC/MS can provide the exact composition of a polymer, such as identifying ε-caprolactam, the monomer used in the production of nylon 6.

To differentiate among various trademarked synthetic fibers, conservators should perform analyses that will determine the fiber's specific chemical composition.

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Curriculum Vitae

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(Endnotes)

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2 Perry, D., et al., *Identification of Textile Materials*, Manchester, UK, Textile Institute, 1985, p. 31.

3 Houck, M., ed., *Identification of Textile Fibres*, Cambridge, UK, Woodhead Publishing, 2009, p. 335.

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