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Multianalytical approach to characterize composition and degradation processes of synthetic high-fashion textiles from the Nanni Strada Design Studio archives

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Abstract

Synthetic textiles are constituents of heritage objects and garments often displayed in museum collections or preserved in archives, presenting unprecedented and specific conservation challenges. These issues need to be addressed assessing degradation processes, and developing targeted restoration and preventive conservation practices. The present work aims at characterizing the composition of high fashion garments and design textiles by Nanni Strada conserved in the archives of Nanni Strada Design Studio (Milan). To identify the chemical composition of the fibers used for the manufacturing, and to investigate the degradation processes affecting the objects, the materials were investigated by Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR), Evolved Gas Analysis coupled to Mass Spectrometry (EGA-MS), and multi-shot Pyrolysis coupled to Gas Chromatography and Mass Spectrometry (Py-GC/MS). The results will contribute to a better knowledge of the chemical properties of the textiles produced in the 1970s, and to plan conservation and exhibition strategies.

1. Introduction

Textile fibers have represented an important resource for men throughout all human history. In fact, ancient cultures employed them not just for the realization of wearing clothes, but also for the expression of their habits and traditions. Historically, textile fibers were obtained from natural sources, such as plants and animals [1, 2, 3], but since the 18th century new cost-effective manufacturing methods for the production of textile fibers were developed, and artificial textile fibers were gradually introduced in the global market [4, 5]. During the 20th century, the remarkable development in polymer research led to the synthesis of the first spinnable polymers, which rapidly replaced natural fibers in the dominion of the textile market [6, 7].

Synthetic textiles are constituents of heritage objects and garments that are currently displayed in museum collections or preserved in archives. These art pieces present unprecedented and specific conservation challenges that need to be addressed assessing degradation processes, and developing targeted restoration and preventive conservation practices. To achieve such goal, effective analytical tools are required, capable to characterize modern and contemporary multi component polymeric materials in heritage objects, and to investigate risk factors and causes of degradation. However, the



information about the specific causes of the degradation of synthetic textiles, entailing the loss of both the cohesiveness of the materials and of the color, is still today very limited [8, 9, 10]. Thus, studying the behavior of synthetic textile fibers in time and their interactions with the environment is crucial to set up proper conservation plans for design textiles, stage costumes, and fashion collections in museum exhibitions and in storage locations.

In this context, the present work aims at characterizing the composition of high fashion garments and design textiles by Nanni Strada conserved in the archives of Nanni Strada Design Studio (Milan). Since the 70s Nanni Strada has focused her work on the realization of non-woven fabrics and seamless garments by embracing new technological processes and unconventional materials. In 1979 she was awarded the “Compasso d’Oro” prize by the Associazione per il Disegno Industriale (ADI). Her projects were displayed in several international exhibitions, such as the Museum of Contemporary Art of Los Angeles (2006-2007), the National Art Center of Tokyo (2007), the Museo Nazionale della Scienza e della Tecnologia of Milan (2011), the Triennale Design Museum in Milan (2016), and are included in museum collections [11].

The research is part of a wider study dedicated to contributing to the knowledge, the conservation, and the valorization of design fabrics and heritage garments fabricated in a period of fast evolution in the chemical composition of synthetic fibers. Previous results of the project include the characterization of stage costumes from the set of Fellini's movies, in collaboration with Opificio delle Pietre Dure in Florence [9].

To identify the chemical composition of the fibers used for the manufacturing, and to investigate the degradation processes affecting the heritage textiles, a reference database was created analyzing several textile fibers by Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR), Evolved Gas Analysis coupled to Mass Spectrometry (EGA-MS), and multi-shot Pyrolysis coupled to Gas Chromatography and Mass Spectrometry (Py-GC/MS). EGA-MS and Py-GC/MS are emerging methods for the study of synthetic polymers [12, 13], but their applications to textile fibers are limited so far [8, 9, 14, 15]. The main advantages of these techniques are related to their capability to discriminate and characterize different fractions of a sample, and the small amount of textile fibers required for the analysis. Liquid Chromatography coupled with Diode Array Detector and high-resolution Mass Spectrometry (HPLC-DAD-ESI-Q-ToF) was also employed to complete the information by characterizing the dyes components.

2. Materials and methods

2.1. Chemicals

The solvent used for the extraction step was dimethyl sulfoxide (DMSO; J.T. Baker, Holland), HPLC grade. The eluents for the HPLC-DAD analyses were: water and acetonitrile both HPLC grade (Sigma-Aldrich, USA), while the eluents for HPLC-ESI-Q-ToF analyses were water and acetonitrile, both LC-MS grade (Sigma-Aldrich, USA). All eluents were added with 0.1% v/v formic acid (FA; 98% purity, J.T. Baker, USA). PTFE filters (4 mm thickness and 0.45 μm pore diameter) were used for the purification prior to HPLC injection.

2.2. Materials

A set of reference textile materials was investigated by ATR-FTIR, EGA-MS, and Py-GC/MS to build a library of spectra and chromatograms that could simplify the assignment of unknown specimens. The set included fabrics made of the most common natural (both animal and vegetal), artificial, and synthetic textile fibers. Some of the samples were provided by the Tapestries and textiles workshops of the Opificio delle Pietre Dure di Firenze (OPD, Florence, Italy) while others were bought as swatches on an online shop (www.tessuti.com).

Several samples collected from high fashion garments and design textiles designed by fashion designer Nanni Strada in the 70s were analyzed to identify the chemical composition of the fibers used for the manufacturing, and to investigate the degradation processes affecting some of these samples. The samples, which are part of the Nanni Strada Studio Archive collection (Milan), were selected by

researchers of the Department of Chemistry of University of Pisa together with Nanni Strada and the restorer Barbara Ferriani.

Part of the samples was collected from the design project “Il Manto” (Figure 1a), a coat designed in 1973 that showed a certain degree of yellowing following exposition to light. The degradation was more evident on the recto of the fabric than on the verso.

A second sample derives from the design project “La Pelle” (Figure 1b), a long-sleeve dress manufactured with poly-tubular technology, the first dress in history entirely seamless woven [16]. The gathering on the sleeves of the dress experienced irreversible elongation during a particular hot summer while the dress was part of an exhibition in Milan.

Finally, a third sample was collected from the project “Amazonica” (Figure 1c), a piece of disposable garment realized using an ink-jet printed non-woven fabric.



Figure 1. Nanni Strada, a) “Il Manto” (1973), b) “La Pelle” (1973), c) “Amazonica” (1976). Courtesy of Nanni Strada.

2.3. Methods

2.3.1. EGA-MS. The instrumentation consisted of a Multi-Shot Pyrolyzer EGA/Py-3030D microfurnace (Frontier Laboratories Ltd. Fukushima, JP) coupled to a 8890N Gas Chromatograph (Agilent Technologies, Palo Alto, USA). A deactivated and uncoated stainless-steel transfer tube (UADTM-2.5 N, 0.15 mm i.d. x 2.5 m length, Frontier Lab) was used to connect the injection port and the mass spectrometer. The products evolved from the samples over the temperature range were transferred to the mass spectrometer, ionized, and analyzed as a function of time. The detector was an Agilent 5977 Mass Selective Detector (Palo Alto, USA) single quadrupole mass spectrometer operating in electron ionization mode (EI) at 70 eV, in positive mode scanning in the range from 35 to 600 m/z. The MS source was kept at 230 °C and the MS quadrupole at 150 °C.

About 300 µg of each sample were placed into a clean stainless-steel cup and inserted into the microfurnace. The temperature program chosen for the microfurnace was: initial temperature 50 °C then 10 °C/min up to 800 °C. The interface temperature was 100 °C higher than the furnace temperature, up to 300 °C. The injection port operated at 280 °C, with a 1:20 split ratio. The chromatographic oven was kept at 300 °C during the whole EGA analysis. The analyses were performed in constant flow mode at 1.0 mL/min (He, purity 99.995%). The collected data were processed by MassHunter Workstation Software (version 10.0).

2.3.2. Py-GC/MS. The instrumentation used for Py-GC/MS was the same used for EGA-MS. However, the transfer tube was replaced by a deactivated silica pre-column (2 m x 0.32 mm i.d., Agilent J&W,

USA) and an HP-5MS fused silica capillary column (stationary phase 5% diphenyl-95% dimethylpolysiloxane, 30 m x 0.25 mm i.d., 0.25 μm Hewlett Packard, USA).

About 80 μg of each sample were placed into a clean stainless-steel cup, inserted into the micro-furnace and pyrolyzed for 0.20 min. The temperatures of pyrolysis were chosen based on the thermal regions showed by the EGA profiles of each sample. The injection port operated at 280 $^{\circ}\text{C}$, with 1:10 split ratio, and the interface temperature was kept at 280 $^{\circ}\text{C}$. The pyrolysis products were eluted in constant flow mode at 1.2 mL/min (carrier gas He, purity 99.995%); the chromatographic program was: initial temperature 40 $^{\circ}\text{C}$ for 5 min, 10 $^{\circ}\text{C}/\text{min}$ to 310 $^{\circ}\text{C}$ for 20 min. The collected data were processed by MassHunter Workstation Software (version 10.0) and the NIST Mass Spectral Search Program (version 2.4).

2.3.3. ATR-FTIR. ATR-FTIR spectra were collected directly on fabric sub-samples using a Thermo Scientific Nicolet iS50 spectrometer coupled with a Smart ITX-ATR accessory equipped with a Ge crystal. Thirty-two scans were collected from 4000 to 650 cm^{-1} with a resolution of 4 cm^{-1} . The analyses were performed at room temperature (22 $^{\circ}\text{C}$). Jasco Spectra Manager software (version 1.53.04) was used for data analysis.

2.3.4. HPLC-DAD and LC-MS/MS. The HPLC system consists of a PU-2089 quaternary pump equipped with a degasser, an AS-950 autosampler, an MD-2010 spectrophotometric diode array detector (DAD) and an FP-2020 fluorescence detector (FD) equipped with a Xenon lamp (150 W). All modules were Jasco International Co. (Japan). ChromNav software was used to carry out data acquisition and data analysis. The DAD detector operated with spectra acquisition in the range of 200–650 nm every 0.8 s with 4 nm resolution. The $\lambda_{\text{ex}}/\lambda_{\text{em}}$ selected for the fluorescence program used were: 350/550 nm for 10.0 min, 552/588 nm from 10.1 to 24.3 min, 490/550 nm from 24.4 to 25.9 min and 450/510 from 26.0 to 39.0 min. The gain selected was 100 \times .

For HPLC-ESI-Q-ToF analyses, an HPLC 1200 Infinity was used, coupled to a Jet Stream ESI-Q-ToF 6530 Infinity detector, and equipped with an Agilent Infinity autosampler (Agilent Technologies, Palo Alto, CA, USA). MassHunter Workstation Software (version 10.0) was used to carry out mass spectrometer control, data acquisition, and data analysis. The mass spectrometer operated in ESI ionization in both positive and negative mode and the 34 working conditions were: drying gas N₂ (purity >98%) temperature 350 $^{\circ}\text{C}$ and 10 L/min flow; capillary voltage 4.5 KV; nebulizer gas pressure 35 psi; sheath gas temperature 375 $^{\circ}\text{C}$ and 11 L/min flow; fragmentor voltage 175 V. High resolution MS and MS/MS spectra were acquired in positive mode in the range 100–1000 m/z at a scan rate of 1.04 spectra/s (CID voltage 30 V, collision gas N₂, purity 99.999%). The FWHM (Full Width Half Maximum) of quadrupole mass bandpass used during MS/MS precursor isolation was 4 m/z. Autocalibration was performed daily using Agilent tuning mix HP0321 (Agilent Technologies) prepared in acetonitrile.

The chromatographic separation was performed in both systems on an analytical reversed phase column Poroshell 120 EC-C18 (3.0 \times 75 mm, particle size 2.7 μm ,) with a pre-column Zorbax (4.6 \times 12.5 mm, particle size 5 μm) both Agilent Technologies (Palo Alto, CA, USA). The eluents were A: formic acid (FA 0.1% v/v) in LC-MS grade water and B: formic acid (FA 0.1% v/v) in LC-MS grade acetonitrile. The flow rate was 0.4 mL/min and the program was 15% B for 2.6 min, then to 50% B in 13.0 min, to 70% B in 5.2 min, to 100% B in 0.5 min and then hold for 6.7 min; re-equilibration took 11 min. During the separation, in both systems, the column was thermostated at 30 $^{\circ}\text{C}$. The injection volume was 10 μL and 5 μL for the HPLC-DAD-FD and LC-MS/MS systems, respectively.

The organic dyes were extracted from the textiles by adding 300 μL of dimethyl sulfoxide to a few mg of sample. The textiles were then sonicated (ultrasonic bath Sonorex Supra 10P, Bandelin Electronics, Germany) for 10 min at 60 $^{\circ}\text{C}$. The supernatant was filtered with PTFE syringe filter and directly injected in the chromatographic system.

3. Preliminary results

The ATR-FTIR, EGA-MS, and Py-GC/MS results allowed us to identify polyurethane and polyester as components of the samples collected from “Il Manto”. The yellowing observed in the fabric is probably correlated to the hydrolysis of the polyurethane fraction [17]. As suggested by the infrared spectra, polyurethane was probably deposited as thin layer over the main polyester fabric, explaining why the recto is more degraded than the verso. This treatment is quite common for waterproofing clothes, and particularly for the realization of outdoor garments like coats. The Py-GC/MS chromatograms obtained at 380 °C and 600 °C for “Il Manto” are shown in Figure 2.

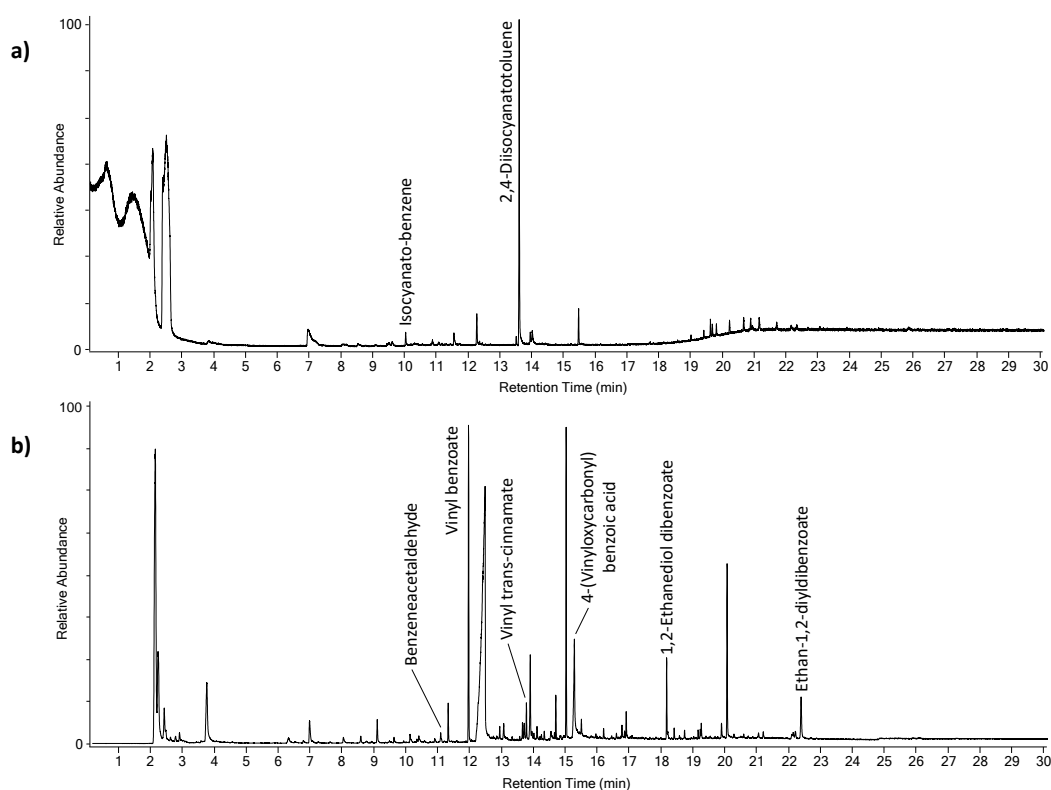


Figure 2: Py-GC/MS chromatograms obtained after multi-shot pyrolysis at 380 °C (a) and 600 °C (b) of "Il Manto", shown in Figure 1a.

Regarding “La Pelle”, ATR-FTIR suggested the presence of polyamide in the sample, as confirmed by EGA-MS and Py-GC/MS analyses. In particular, the evaluation of the relative abundances of the chromatographic peaks related to cyclopentanone and ϵ -caprolactam allowed us to identify the polyamide as nylon 6 [12]. HPLC-DAD-ESI-Q-ToF analysis completed the characterization of the fiber with the identification of the dye component as Disperse Yellow 3. This monoazo pigment dye has low aqueous solubility and it is employed in the production of wool, furs, nylon, acrylic, and other synthetic fibers [18]. Thus, the loss of elasticity can be attributed to the photo-oxidation of the polyamide chain induced by radical reactions promoted by light exposure, and/or the influence of the dye molecules on the glass transition temperature (T_g) of the fiber [19, 20]. Further thermal analyses by Differential Scanning Calorimetry (DSC) are required to fully clarify the cause of degradation.

The pyrogram of the sample collected from “Amazonica” features the typical clusters of peaks composed by three signals related to α,ω -diene, α -alkene, and alkane of a given polyethylene oligomer, pointing to the synthetic origin of the fiber, as polyethylene (PE) [12]. The results were in accordance with those obtained through ATR-FTIR and EGA-MS analysis. Some oxidation products of PE components were also detected, whose specific nature is currently under investigation.

The results will contribute to a better knowledge of the chemical properties of the textiles produced in the period of interest, and to plan conservation and exhibition strategies. Moreover, improving the understanding of the chemico-physical properties and degradation processes affecting synthetic fibers is a highly transferable result, beneficial also for the fields of industrial textile technology and for environmental studies related to pollution from synthetic microfibers.

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