PROTOCOL FOR IDENTIFICATION AND ASSESSMENT OF NATURAL AND SYNTHETIC TEXTILE FIBERS

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Proper identification of textile materials is essential, as people use textiles for clothing and shelter, dental and medical devices, protective firefighting, or even military clothing. There have been several developments regarding fiber identification using instruments such as Fourier transform infrared spectroscopy, Raman spectroscopy, or electron microscopy. However, the traditional methods are prevalent as they are the cheapest alternative. In the present paperwork, an accelerated weathering test was conducted on two different textile materials – cotton (natural fiber) and polypropylene (synthetic fiber). Alternating cycles of UV exposure, along with humidity and relatively high temperatures were employed for the weathering test. In order to evaluate the degradation degree of the two fibers, the results were compared and investigated using non-destructive and micro-destructive analysis techniques such as Scanning Electron Microscopy (SEM), to evaluate the surface modifications of the fibers, and colorimetry, to quantify the color changes. In addition, Fourier-Transform Infrared Spectroscopy (FT-IR) indicated the modifications of functional groups that occurred after the weathering test. A non-destructive technique – X-Ray Diffraction (XRD) was also performed to obtain information about the crystalline structure. The obtained information will be used for cultural heritage studies.

Keywords: fibers, accelerated weathering, cultural heritage.

INTRODUCTION

The history of textile is almost as old as the history of human civilization and as time moves on the history of textile has further enriched itself (Kvavadze *et al.*, 2009). Traditionally, natural fibers have been used in all cultures for making functional products like clothes, bandages, uniform, etc. Clothing and textiles have been important in human history and reflect the materials available to a civilization as well as the technologies that were acquired. The social significance of the finished product reflects their culture (Byko, 2005). It is therefore important to develop new and efficient protocols to properly identify the fibers as this can provide answers that can be further used for cultural heritage studies. Identifying fibers involves observing the physical and chemical properties of the fiber for which there are a wide diversity of instruments available (Robertson, 2010). As fibers behave differently depending on the fibers' composition, the light, temperature and humidity they are exposed to (Yang and Ding, 2006) in the present paper two different fibers, cotton (natural fiber) and polypropylene (synthetic fiber), were analyzed after conducting an accelerated weathering test, using non-destructive and micro-destructive techniques such as Scanning Electron Microscopy (SEM), Fourier-Transform Infrared Spectroscopy (FT-IR) and X-Ray Diffraction (XRD).

MATERIALS AND METHODS

A QUV instrument from Q-Lab, equipped with fluorescent UVB-313 lamps, was used to simulate an accelerated weathering process on five samples of the two types of fiber. The weathering cycle used consisted of an 8 h UV exposure cycle at 70°C, followed by a 4 h condensation cycle (60% humidity) at 50°C. A sample from each fabric was collected every three days, resulting five samples at different times of exposure.

RESULTS AND DISCUSSION

SEM

A FEI Quanta 200 Scanning Electron Microscope was employed for assessing the morphological changes of the samples (Table 1). The magnification used was 500X.

Table 1. SEM micrographs of the control and aged cotton and polypropylene samples

Sampling time	Cotton	Polypropylene
t ₀ (control)
t ₁
t ₂
t ₃
t ₄
t ₅

The accelerated weathering process did not cause a significant modification of the cotton fibers' morphology. However, some fiber breakage was noticed, mostly at the latter periods of exposure. The dry heat caused the desiccation of the cotton fibers, hence their chemical and physical degradation (Timar-Balazsy and Eastop, 1998). Due to the high cellulose content of cotton, the additional UV radiation was absorbed by the fibers, causing more damage.

For the polypropylene samples, the effect of the weathering test was visible via SEM from time t_2 , in terms of fiber breakage. In the last three sampling periods, the fibers were not only broken, but they were also brittle, with cracks appearing transversally on them, confirming the damaging effects of the weathering experiment.

FTIR

IR spectra were recorded using an FT-IR-ATR instrument from ThermoFisher, over a spectral range of 4000-400 cm^{-1} (Figures 1 and 2).

Figure 1. IR absorption spectra of the control and aged cotton samples

Figure 2. IR absorption spectra of the control and aged polypropylene samples

For cotton fabrics there are no significant changes in the IR absorption spectra of the exposed samples compared to the reference fabric. Only slightly changes in the intensity of the bands can be observed. This indicates that the changes at the structural level are minimal and the cotton fabric is not significantly altered after the exposure to the

mentioned conditions. Table 2 indicates the IR spectra correspond with the literature data (Chung *et al.*, 2004).

Table 2. IR absorption band positions for cotton compared to literature data

Experimental band position [cm ⁻¹]	Band position reported in literature [cm ⁻¹]	Band attribution
3600-3000	3550-3100	H-bonded OH stretch
2897	2980-2800	C-H stretching
1636	1644	Adsorbed H ₂ O
1427	1429	CH wagging (in-plane bending)
1361	1368	CH bending (deformation stretch)
1334	1337	OH in-plane bending
1314	1316	CH wagging
1280	1281	CH deformation stretch
1247	1247	OH in-plane bending
1203	1203	OH in-plane bending
s1159	1160	Asym. Bridge C-O-C
1105	1108	Asym. Bridge C-O-C
1052	1057	Asym. In-plane ring stretch
1028	1030	C-O stretch
897	900	Asym. out-of-phase ring stretch: C1-O-C4; b glucosidic bond

On the other hand, the overlapped spectra of the polypropylene samples show notable changes, especially in terms of band intensity. The bands at 3296 and 3070 cm⁻¹ in the unexposed fabric spectrum are missing in all the other spectra. This indicates that any trace of supramolecular interaction is destroyed even after short exposure time. The bands at 2950 cm⁻¹, 2917 cm⁻¹, 2868 cm⁻¹, 2837 cm⁻¹ are assigned to the CH₂ asymmetrical stretching vibration, CH₃ asymmetrical stretching vibration, CH₃ symmetrical stretching vibration and symmetrical CH₂ stretching vibration, respectively. The bands at 1456 cm⁻¹ and 1375 cm⁻¹ are attributed to the deformation vibration asymmetrical and deformation vibration symmetrical of the CH₃ (Souza *et al.*, 2017). These bands suffer an increase of the intensity during the exposure. The significant difference between *t*₀ and *t*₁ spectra might be due to the presence impurities which seem to be removed in the exposure process. The overlapped spectra of the *t*₁₋₅ samples do not present considerable changes of bands position or intensity.

XRD

The XRD diffraction measurements were performed on a Proto AXRD diffractometer, using as X-ray the Cu K α radiation ($\lambda = 1,54\text{\AA}$), between 2 θ angles 5-35° (Figures 3 and 4).

Figure 3. PXRD of the cotton sample, at different exposure times

Figure 4. PXRD of the polypropylene sample, at different exposure times

The diffraction lines recorded at different exposure times do not follow a certain trend. In the case of cotton, the crystallinity increases up to 6 days of exposure then decreases. This is observed following the diffraction line at $2\theta = 22,8^\circ$, representing the crystallographic plane (002) of the cellulose (Park *et al.*, 2004). A possible explanation is that the degradation destroys first the non-crystalline areas, only later the crystalline ones.

The XRD peaks and corresponding crystallographic plane for polypropylene are listed in table 3.

Table 3. XRD peaks and corresponding crystallographic plane for polypropylene (Yang and Ding, 2006)

2θ (°)	Crystallographic plane	Crystalline form
14,0	(110)	α
16,0	(300)	β
17,0	(040)	α
18,5	(130)	α
20,5	(301)	β
21,5	(111)	α

Polypropylene is present in nature as different polymorphic forms: α , the most crystalline, β , less crystalline and γ , almost completely amorphous. Table 1 contains the specific diffraction lines for each form (Türkçü, 2014). In this case, the sample contains mostly α polymorphic form. When exposed to the degradation conditions, the amorphous part of the fiber is destroyed, along with the β component present in the sample, so the overall crystallinity of the sample is increased. This can be observed by following the increase in intensity of the (110) and (111) lines and the disappearance of the (301) line. It also can be observed a small line forming at $2\theta=31^\circ$, having higher intensities for the most exposed samples.

CONCLUSIONS

All three performed analyzes show a higher resistance of cotton to polypropylene after the accelerated weathering process was conducted. The changes at the structural level were minimal and the cotton fabric is not notably altered after the exposure to the mentioned conditions. Therefore, we can conclude that even if synthetic fibres are known to be strong and durable, given the exposure conditions of these two fibres, the cotton maintained better. As natural fibres usually have a smaller environmental impact than synthetic fibres due to less chemicals involved in the production process the results are encouraging. Research in this area will continue, in order to improve and help our environment, but also to better maintain our patrimonial objects.

Acknowledgements

This work was elaborated through Nucleu Program, conducted with MCI support, project no. 4N/2019/PN 19 17 05 01. The publication fee of the paper is funded by the Ministry of Research and Innovation within Program 1 - Development of the national RD system, Subprogram 1.2 - Institutional Performance - RDI excellence funding projects, Contract no. 6PFE/2018.

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<https://doi.org/10.24264/icams-2020.V.12>