PHYSICAL AND CHEMICAL ASSESSMENT OF A PATRIMONY SAMPLE

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The objective of this paper was to investigate the physical and chemical properties of a sample from a national heritage private collection from the beginning of the 20th century. The fibrous composition was determined by using Scanning Electron Microscopy (SEM) and also Optical Microscopy, these methods also providing an insight on the level of degradation of the sample. Physical and mechanical analysis methods were performed on yarns extracted from the sample with the purpose of gathering informations about the way the textile fabric was produced. The thermal behavior of the extracted yarns was assessed by using Differential Scanning Calorimetry (DSC). This way it could be determined the level of humidity which was present in the yarns, the melting temperature of the crystalline areas in the fibers, the enthalpy of melting and the temperature of thermal destruction of the sample. Although DSC is a destructive analysis, the main advantage of this method is the necessity of a very small amount of sample. According to the results, the sample's composition was 100% wool and it had some level of damage due to environmental factors, considering its old age. DSC analysis indicated a low level of humidity and also confirms the composition of the material as being indeed wool.

Keywords: wool, fiber, DSC

INTRODUCTION

Although the increasing use of synthetic fibers (polyesters, polyamides, polyacrylates) in all domains and industries, natural fibers – animal or plant derived still play a significant role in everyone's lives. The main advantage of using natural fibers over synthetic fibers is the lower rate of pollution with fiber waste, but also the sustainability of raw materials used to produce natural fibers. Textile fabrics that have historical significance must be studied in ways in which their integrity is preserved as much as possible. Non-destructive and micro-destructive analytical methods can be used to provide information about the deterioration of these historical fabrics and also about ways in which they can be restored to almost their initial condition. However, sometimes, the level of degradation can affect the results of the analyzes end can even make some analytical methods impossible to perform.

This particular research highlights the way in which a historical textile sample has been affected by environmental factors, and also provides information about the sample's fibrous composition and its thermal behavior.

MATERIALS AND CHARACTERIZATION METHODS

Materials

The present study was performed using a textile sample. The textile sample is a narrow girdle which was selected from a private collection from the beginning of the 20th century from Radosi – Gorj area.

https://doi.org/10.24264/icams-2018.VIII.14

Figure 1. Sample – narrow girdle

Characterization Methods

The qualitative analyzes for investigating the fibrous composition of the sample were performed by using Scanning Electron Microscopy – SEM (apparatus: Quanta 200, FEI, Netherlands) and Optical Microscopy (apparatus: Olympus BX41, Japan). SEM could be used for identifying the fiber composition of the national heritage samples because the original textile material has not been widely damaged. The only drawback of this method was the necessity of coating the textile sample with an electrically conductive material in order for them to be examined using SEM. Optical Microscopy was also used for the qualitative analysis of the sample with the main purpose of assessing the exfoliation of the fibers and thus the overall level of degradation.

Physical–mechanical properties of the textile yarns extracted from the sample were also assessed within this study in order to provide a better understanding of the material that was analyzed. The following determinations were made: linear density, twisting in yarns and the direction of the twist in yarns.

Differential Scanning Calorimetry - DSC (apparatus: Perkin Elmer, USA) was performed on a yarn sample to investigate the glass transition temperature - Tg, the crystallization temperature, the thermal destruction temperature and enthalpies of transitions (O'Neill, 1964). The sample was placed in a crucible made from aluminium which was then closed. The reference used for the DSC analysis was made from indium. The heating program started at 35°C and this temperature was maintained for 1 minute, then the temperature range was from 35 to 500°C at a heating rate of 10°C/min. When the temperature reached 500°C it was maintained for 1 minute.

RESULTS AND DISSCUSION

Fibrous Composition Determination

The fibrous composition of the sample is shown in Table 1.

Table 1. Fibrous composition identification

Nature of the raw material (SR 13231:1995)	Fibrous composition (EU Regulation 1007/2011)
Wool	100% Wool

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The wool fiber consists of a multitude of cortical cells and flaky cells which are held together by a cell membrane complex, representing the continuous phase in the wool fiber (Wilkie *et al*., 2016). In the SEM micrograph (Figure 2-a) and in the Optical Microscope image (Figure 2-b) presented below it can be observed that there is a slight exfoliation at the surface of some of the wool fibers which indicates that the material most likely has been subjected to various mechanical and environmental factors such as UV radiation, variations in humidity or microorganisms.

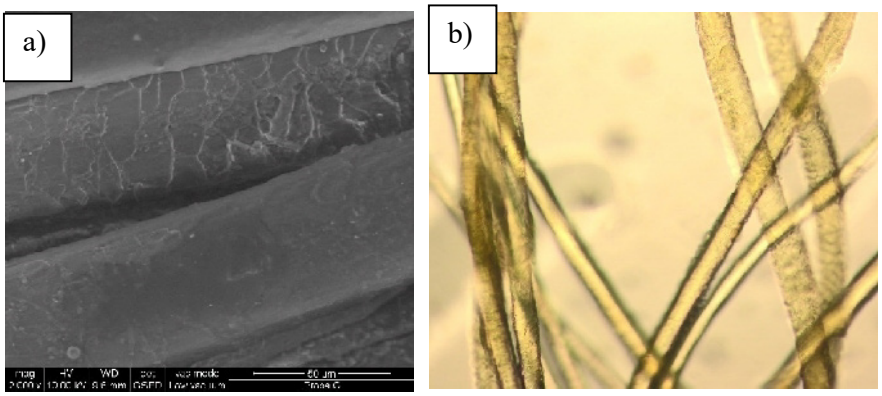


Figure 2. a) SEM micrograph of wool fibers (50 µm); b) Optical Microscope image of wool fibers (20x)

Physical–Mechanical Properties

Linear density represents the mass of a yarn per unit length. The twisting in yarn represents the number of rotations around its own axis per nominal length between the clamps before the de-twisting of the yarn. Most fibers have either an S twist or a Z twist. S-twist yarns are spun counter-clockwise, in other words, if the yarn is held in a vertical position, the spirals created by the fibers around their own axis are inclined in the same direction as the diagonal segment of the letter S. Z-twist yarns are spun clockwise and the fibers mentioned previously are inclined in the same direction as the diagonal segment of the letter Z.

Table 2. Physical–mechanical properties of the yarns extracted from the sample

Analysis name				Results	Standard Regulation
Linear density	Tex (Nm)	Warp	Beige yarn	127,3x1 (7,86/1)	SR EN 7271:2008
			Brown yarn	140,7x1 (7,11/1)	
			Red yarn	182,9x1 (5,47/1)	
		Weft		124,1x2 (8,06/2)	
Direction of the twist		Warp		S	ISO 2/ 1973
		Weft		-	
Twist	t/m	Warp	Beige yarn	111,7	SR EN ISO 2061:2015

https://doi.org/10.24264/icams-2018.VIII.14

Analysis name	Results	Standard Regulation
Brown yarn	110,0	
Red yarn	123,3	
Weft	-	

In the warp of the sample there have been identified three types of yarns, each one having S-twist direction, as shown in Table 2. The direction of the twist of the yarns extracted from the weft could not be determined due to the degradation of the sample and the impossibility of extracting yarns with a sufficient length.

Thermal Properties

DSC was used to confirm the fibrous composition of the sample and also to provide information regarding the crystallinity of the fibers (keratin in particular), the glass transition temperature of the macromolecular components found in the sample, the thermal destruction temperature and the enthalpy of transition.

Table 3. DSC thermograms for the sample

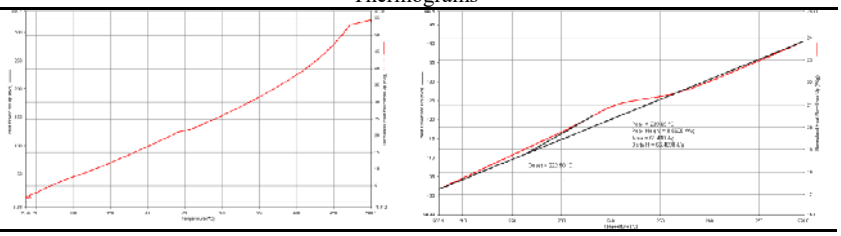
Thread	Thermograms
Dark brown	

Table 4. Thermodynamic parameters for the sample

Thread	Sample weight [mg]	Onset temperature [°C]	Peak temperature [°C]	Delta H [J/g]
Dark brown	5.9	222.50	239.65	66.4098

In wool threads, the glass transition temperature depends on the humidity content of the sample (Phillips, 1985). By increasing the water content, the width of the endotherm peak and the glass transition temperature decrease. These effects occur due to the breakdown of the polar networks in the amorphous regions of the keratin (Phillips, 1985). In other words, water can act as a plasticizer in wool fibers (Wortman *et al*., 1984).

The first thermogram presented above shows a slight peak at 90-100°C, which corresponds to the vaporization of the water present in the sample. The broad and barely noticeable peak can suggest that water was present in the sample, but in very low quantity. The glass transition temperature could not be identified within this study because the peak most likely corresponds with the one of the water vaporization. According to a study of Phillips, D.G., the glass transition temperature of wool is approximately 85°C. This confirms the difficulty of identifying the glass transition

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temperature in a wool sample which contains moisture. The endotherm peak which begins at 222,5°C, with a maximum at 239,65°C represents the melting of the crystalline areas in the fibers. This peak can be correlated with the degree of crystallinity in the material. A higher melting temperature is associated with a higher crystallinity degree. At 350-430°C there is a peak which is slightly noticeable, corresponding to the decomposition of the macromolecular chains by carbonization and thermolysis (Wortmann and Deutz, 1998), with the release of carbon dioxide, sulfur dioxide, nitrogen dioxide, nitrogen monoxide, methane, butane (Xia *et al*., 2016).

CONCLUSIONS

In this paper, a textile sample from a national heritage collection was analyzed using SEM and Optical Microscopy, standardized physical-mechanical methods and DSC. These analysis methods provided information regarding the composition of the material and its level of degradation. The assessment of these properties could be performed by correlating the results with those existing in the literature. The results show some level of degradation of the fabric, but overall the condition of the material is satisfying.

Acknowledgement

This work was supported by a grant of the Romanian Ministry of Research and Innovation, CCCDI-UEFISCDI, project number PN-III-P1-1.2-PCCDI-2017-0878/55PCCDI, within PNCDI III.

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