STUDY OF THE BEHAVIOUR OF SILK PONGEE USED FOR THE CONSOLIDATION OF HISTORICAL TEXTILES

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ABSTRACT: *Consolidation is one of the most delicate and decisive process to preserve any fabric. The addition of new sewed fabrics such as 'silk pongee' to the original textiles is a common practice nowadays to consolidate, reinforce and compensate losses in historic textiles. Pongee is a plane weave silk used as reinforcement fabric due to its low density of the yarns in both the weft and warp directions that provides a considerable transparency as well. Results obtained from this research focus on the physic-chemical characterization of pongee. Data from specimens subjected to artificial accelerated ageing (dry and wet heat and UV radiation) are also shown. A multi-method approach is proposed combining microscopy (LM and SEM/EDX) and spectroscopy (reflectance, FTIR) techniques and traction tensile tests in order to characterize the overall behavior of the 'silk pongee' before/after ageing processes. Tensile tests were run in weft and fill directions. Stress strain curves helped to determine the stiffness and flexibility of 'silk pongee' as well as their elongation and strength to failure within specific environmental conditions. Comparison of Vis and IR spectra obtained in ATR mode from different areas of the pongee textile has evidenced that the yellowness and oxidation grades increase as follows: firstly UV radiation, secondly wet heat and finally dry heat. LM and SEM examination enabled the identification of empty fiber and microfissures in different areas on the surface. The measured mechanical properties are mainly focused on the maximum deformation and strength of samples prior failure.*

KEYWORDS: pongee, consolidation, historic textiles, ageing, microscopy, spectroscopy, tensile test

1. INTRODUCTION

Silk is a proteinaceus fibre produced by silkworms or spiders. The common one is that that is produced by the Bombyx mori domestic silk worm which makes a cocoon during pupation. Cocoon is made by two filaments with triangular cross section and joined by an amorphous protein called sericin, which is usually removed for commercial purposes, that process is known as degumming. Silk fibres are comprised of poly(alanine) (Ala) and poly(glycine-alanine) repeats. The molecular structure of silk is composed of amorphous matrix with some crystallites dispersed in. The extensive crystallinity of silk dictates its physical and chemical properties (Garside and Wyeth 2007: 871). Crystals are formed by polypeptide chains in a pleated β-sheet conformation, what provides rigidity to the fibre because of their anchoring points (Garside and Wyeth, 2007: 871; Lefèvre et al, 2009: 136; Qiu, 2009: 602; Gosline, 1986: 37; Termonia, 1994: 7378; Edwards HGM, Farwell, 1995: 901). Despite its extraordinary mechanical strength, the resistence of silk, which characterizes its capacity for shape and energy recovery under mechanical loading, is usually very poor (Qiu, 2009: 6023; Gosline, 2002: 121).

Silk has been utilized as a textile fibre for over 5000 years (8). Silk properties made it useful in different fields such as for building blocks of extra- and intracellular matrices, were it plays an important role in structure support, scaffolding, stabilization, and the protection of cells, tissues and organisms (Qiu, 2009: 6023). Silk fibres are found in many important historic textiles and artefacts (Garside and Wyeth 2007: 871). It is becoming increasingly difficult to ignore the fact that historic textiles suffer some damages although they have been storaged in the best conditions.

An appropriate characterization of the physical and chemical conditions in which the textile is, makes easy the fact of treat, handle, move or even expose it. The baste majority of methods used to determine fibre conditions are destructive ones. Nevertheless, several attempts have been made to demonstrate correlations between the physical deterioration of silk samples and certain measurable spectroscopic, chromatographic and chemical signatures, what is essential when considering appropriate conservation, display and storage strategies (Garside and Wyeth 2007: 871; Ojah and Dolui, 2006: 1529; Fuster-López et al., 2008: 159; Yusá-Marco et al., 2008: 153).

Consolidation is one of the most delicate and decisive process to preserve any fabric (Vicente-Palomino et al., 2006: 139). The addition of new sewed fabrics such as 'silk pongee' to the original textiles is a common practice nowadays to consolidate, reinforce and compensate losses in historic textiles. Pongee is made of 100% silk and is used as reinforcement fabric due to its low density of the yarns in both the weft and warp directions that provides a considerable transparency as well. The aim of this work is focused to determine the effect that accelerated ageing can induce in pongee fabric. Thus we used dry and wet heat and, also Ultra violet (UV) radiation. Results we provide us an idea about the effect that silk pongee used to consolidate an historic fabric can induce on it.

2. EXPERIMENTAL SECTION

2.1. Materials

Degummed, white silk known as pongee is used, it has been supplied by Soditex S.L. company, and it was used as purchased. It was 45 g/ m^2 weight with 60 yarns/cm in weft direction and the same density for weft one. Yarns characteristics are 4 Tex yarns in warp direction and 7 Tex yarns in weft direction.

2.2. General methods and instruments

2.2.1. Ageing treatments

A first series of specimens were subjected to thermal ageing for 30 days. They were introduced in a Climatic chamber Dycometal DL-100, with constant temperature and relative humidity of 80º C and 65% as standard UNE 57092-4 says.

A second series of specimens were treated in the same way but without humidity and with 105º C in temperature as standard UNE 57092-1 says.

A third series of specimens was subjected to UV light exposure by irradiating them in a Dycometal chamber model QUV-Basic provided of an UV lamp UVB 313EL (Q-panel Lab Products), which produces mostly short-wave UV with maximum intensity at ca. 310 nm and its equivalent to a 40 watt fluorescent lamp. Temperature was maintained at constant value of 45º C. Samples were exposed to UV light for 72 hours.

2.2.2. Change in colour

One of the colour systems on which most colorimetry studies are based is the CIE L*a*b*system (Aspland, 1993: 34; Billmeyer and Saltzman, 1982).

To check if samples suffer some modification on the white colour, some measurements in a reflexion spectrophotometer were made, and we determine the white degree, yellowness and ΔEab.

Tests were developed in agreement with the standard guidelines for textile fabrics (Standard ISO105-J01, 1997: Part J01; Standard ISO105-J02:1997 Part J02; Standard ISO105-J03:1997: Part J03).

2.2.3. FTIR spectroscopy

IR absorption spectra were performed in attenuated total reflectance mode (ATR) with a Vertex 70 Fourier Transform infrared spectrophotometer (Bruker optik GmbH) with a FR-DTGS (fast recovery deulerated triglycine sulphate) temperature stabilized coated detector. Number of co-added scans was 32 with a resolution of 4 cm^{-1}. Data was processed with Opus software, version 5.0.

2.2.4. Tensile test

It was conducted with a Zwick dinamometre Zwick/Röel Z005 five samples were tested for every series and pongee samples were of 200 mm length and 50 mm width.

Environmental temperature was 20ºC and relative humidity of 65%. That conditions and all the parameter of the test were fixed as standard UNE EN ISO 1924-2:1994 says.

2.2.5. SEM

Surface morphology of silk fibre and its fracture were characterized using a JEOL JSM-6300 scanning electron microscope (SEM). Each sample was fixed on a standard sample holder and sputter coated with carbon. It was then examined by the SEM with suitable acceleration voltage (3 kV) and 15 mm working distance.

3. RESULTS AND DISCUSSION

First of all we studied chromatic values (Table 1), we could find noticeable variations in lightness/darkness (L*) because of the treatment. Samples with UV treatment present more darkness than the others in which lightness is reduced but not so markedly. On the other hand chromatic values changed. On the values for redness/greenness (a*) some changes can be appreciated when moisture is present changes moves towards more red colours but if it is not colour turns to more green colour what is increased by UV exposure. Results obtained from the preliminary analysis of b*, show colour modification on yellowness/blueness. When we study b* values it indicates yellowness is induced by ageing, and mainly because UV treatment, being less affected when moisture was present in ageing treatment.(Table 1)

To check ageing effect FTIR spectra were studied for every sample. Figure 1 shows the spectra for every sample. Figure 1 shows the full spectrum and Figure 2 shows details in the region from 1800 to 1000 cm^{-1}. For both Figures black line fits with pongee without treatment, and grey line belongs to pongee with UV treatment. These spectra summarize the main changes induced in the specimens after the application of the accelerated ageing treatment. (Figure 1)(Figure 2)

The band observed at 3278 cm^{-1} corresponds to the vibrational stretching of hydroxyl groups (Monllor et al., 2009: 365 ; Monllor et al., 2007: 2481; Yao et al., 2007 : 635; Bonet et al., 2004: 4). The bands are usually sensitive to the silk conformation in the IR spectra

SAMPLE	L*	a*	b*	ΔL*	Δa*	Δb*
Non treated	93,0161	0,8828	5,5329			
UV	86,5075	0,6188	21,9628	-6,5085	-0,2639	16,4298
T	91,2121	0,0441	10,9752	-1,8039	-0,8387	5,4422
T + RH	92,6426	0,9657	6,2247	-0,3734	0,0829	0,6918

Table. 1. Chromatic values for every sample and differences with treatment. L*= lightness/darkness, a*= redness/greenness, b* = yellowness/blueness. UV = Ultraviolet, T= aged by temperature, T + RH = aged by temperature and moisture

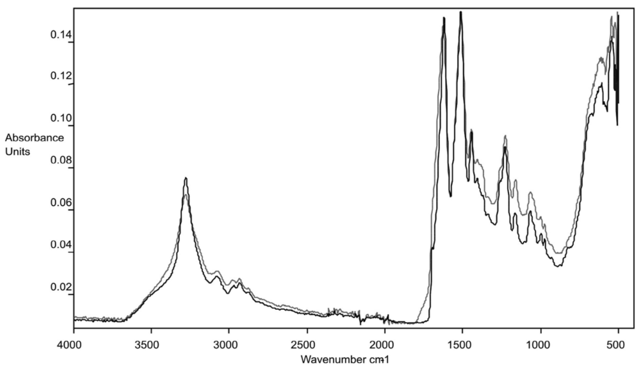

Figure. 1. FTIR spectra of silk pongee in the region for 4000-400 cm-1.(Black line) Unaged sample. (Grey line) UV aged sample

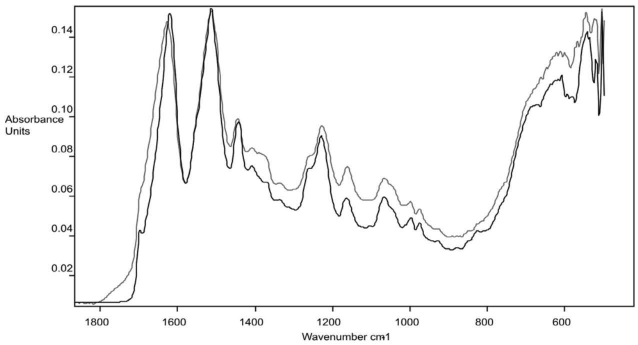

Figure. 2. FTIR spectra of silk pongee in the region for 1800-600 cm-1.(Black line) Unaged sample. (Grey line) UV aged sample

at amide I, II and III modes. These spectra showed the characteristic absorption bands of silk in a β-sheet conformation, 1621 cm^{-1} with the presence of a shoulder at 1697 cm^{-1} (amide I), 1508 cm^{-1} (amide II) and 1261 and 1228 cm^{-1} (amide III) (Monllor et al., 2007: 2481). On UV treated spectra we can observe that begins to appear a shoulder at 1735 cm^{-1} which corresponds to the vibrational stretching of the carbonyl groups from carboxylic acid, indicative of the increment in the oxidative grade from silk fibre because of ageing treatment.

Spectra of the samples aged with temperature and with temperature in presence of moisture are not shown because they fit with pongee without treatment what means that there are no significant changes in structure.

SAMPLE	Resistance (N)	Elongation (%)
Non treated	425,16	12,11
UV	65,58	2,61
T		
T + RH		

Table. 2. Traction resistance of samples with ageing. UV = Ultraviolet, T= aged by temperature, T + RH = aged by temperature and moisture

When tensile test is studied, we can observe some results that confirm what we shown above. Table 2 shows values obtained for every sample and we can observe that when ageing is caused by temperature or by temperature with moisture, non significant differences can be noticed. On the other hand we can observe noticeable variations when UV treated samples are studied, either breaking force or elongation are considerably reduced because of the ageing treatment. (Table 2)

Figure 3 shows the fibres surface morphology after tensile test of the unaged and aged samples. It can be noticed that there are not much differences in cross section of the fibre in ageing treatments with temperature either for the one without moisture or the one with moisture. On the other hand, silk broken after UV treatment shows significant differences because it seems fibre is drier. It makes that broken surface is more roughness than others. (Figure 3)

4. CONCLUSIONS

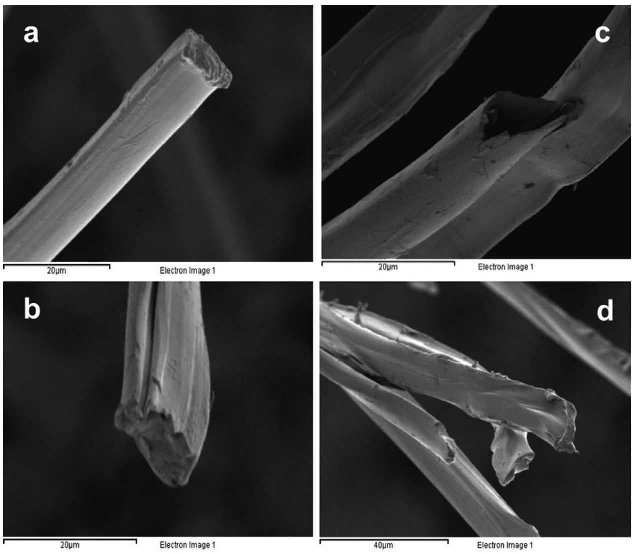

Figure. 3. Surface morphology of aged silk pongee after tensile test. a) Unaged sample. b) UV aged sample. c) Temperature aged sample. d) Temperature and moisture aged sample

This study produced results which allow knowing that UV exposure can modify pongee appearance what implies the modification of the historical fabric treated with it. Storage and conservation should imply to avoid UV exposure and moisture should be maintained. The main structural changes were shown by FTIR analysis in which we could observe there was an increment in the oxidative grade of silk when treated with UV but not in the rest of samples studied. Thus implies a considerable reduction in mechanical behaviour for UV treated sample that is not appreciated in the rest of samples studied. To sum up we can conclude that Comparison of Vis and IR spectra obtained in ATR mode from different areas of the pongee textile has evidenced that the yellowness and oxidation grades increase as follows: firstly UV radiation, secondly wet heat and finally dry heat. SEM shows that UV samples look more uneven and deteriorated evidencing a stiffer fracture, whereas temperature aged (with or without moisture) or unaged samples show a smooth and flat surface. It means that UV treatment dries the fibre and makes it to break in a different way.

The main changes were observed in the pongee silk samples with UV light ageing. Changes can be summarised in more variation of colour, structural changes, losses more mechanical properties and breaks with roughness surfaces.

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Versión española

TITLE: ***Estudio del comportamiento del pongee de seda como tejido consolidante de tejidos históricos***

ABSTRACT: *La Consolidación es uno de los procesos de conservación textil más delicados y decisivos a la hora de preservar cualquier tejido. La adición de un tejido mediante costura como el pongis a un tejido original es una práctica común hoy en día con el fin de consolidar, reforzar y compensar pérdidas en tejidos históricos. El denominado pongis es un tejido de seda de ligamento tafetán, utilizado como tejido de soporte debido a su baja densidad, tanto en trama como en urdimbre, que proporciona una considerable transparencia. Los resultados obtenidos de esta investigación se centran en la caracterización físico-química de este, con el propósito de caracterizar el comportamiento del pongis, antes y después de los procesos de envejecimiento. Se propone la optimización de un método múltiple que combine diferentes técnicas analíticas: microscopia (LM y SEM/EDX), técnicas de espectroscopia (FTIR-ATR) así como ensayos de tracción que se llevaron a cabo en dirección trama. Las curvas de esfuerzo-deformación obtenidas ayudaron a determinar tanto la rigidez como la flexibilidad del ponguis así como su capacidad de elongación y resistencia previa a la rotura en unas condiciones ambientales específicas. La comparación entre los espectros Vis e IR llevados a cabo en diferentes áreas del tejido evidenciaron que el grado de amarilleamiento y oxidación se acrecienta en el siguiente orden, dependiendo del proceso de envejecimiento: en primer lugar radiación UV, seguidamente calor húmedo y finalmente calor seco. Los exámenes llevados a cabo mediante LM y SEM permitieron la identificación de la totalidad de la fibra y de micro fisuras en diferentes áreas de la superficie. La determinación de las propiedades mecánicas se centra principalmente en la deformación y resistencia máximas de las muestras previa a la rotura.*

KEYWORDS: *pongee, consolidación textil, tejidos históricos, envejecimiento, espectroscopia, ensayos de tracción*