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Rediscovering the materials of Arraiolos tapestries: fibre and mordant analysis by SEM-EDS and μ-PIXE

Ana Manhita[*], Cristina Costa[*], Teresa Ferreira[*], José Mirão[**], Helena Vargas[***], Isabel Ribeiro[***], Isabel Seruya[****], Teresa Pacheco[*****], Luís Alves[******] and António Candeias[*]

[*] Universidade de Évora, Chemistry Department and Évora Chemistry Center, Rua Romão Ramalho 59, 7000-671 Évora, candeias@uevora.pt, Portugal.
[**] Universidade de Évora, Geosciences Department and Geophysics Center of Évora, Portugal
[***] Instituto dos Museus e da Conservação, R. Janelas Verdes 37, 1249-018 Lisboa, Portugal.
[****] Universidade Nova de Lisboa, Conservation and Restoration Department, 2819-516 Monte Caparica, Portugal
[*****] Museu Nacional de Arte Antiga (MNAA), Rua das Janelas Verdes, 1249-017 Lisboa, Portugal
[******] Instituto Tecnológico e Nuclear, Estrada Nacional 10, 2686-953 Sacavém, Portugal

Arraiolos tapestries are probably one of the richest artistic Portuguese expressions in terms of textile heritage. It is difficult to date the beginning of the production of rugs in Arraiolos (Southern Portugal), but they were already produced in the late 1600's as they are listed in the inventories of Portuguese aristocratic households in the beginning of the 18th century. Sensitive detection techniques play an increasing role in the chemical investigation of historical objects since the knowledge derived from the chemical composition of materials is of upmost importance for textile conservation and restoration purposes. Textiles deteriorate due to natural causes like heat, radiation, mechanical stress, moisture, microbiological and enzymatic attack. Deterioration of materials causes breakdown of the molecular structure and results in a loss of strength, extensibility and durability, discoloration and fading which affects the appearance of the textiles[1].

In this work we present results of microanalysis by SEM-EDS and μ-PIXE of fibres from Arraiolos rugs (Rug nr. 88, from the 17th century) of the Portuguese Ancient Art National Museum collection (MNAA). The purpose of the study was to identify and evaluate the nature and the conservation state of the fibres and employed mordants by the Arraiolos dyers. This work is integrated in project "REMATAR - Rediscovering the Materials of Arraiolos Tapestries" (PTDC/HAH/64045/2006) funded by Fundacao para a Ciencia Tecnologia which intends to obtain detailed and systematised information on the composition, structure and degradation processes that occur on these tapestries and to use these data to improve their conservation methodologies. Moreover, the collected data on the dyestuffs and mordants will hopefully allow a time scale of materials usage that could assist on the dating of other Arraiolos pieces and the understanding of the sociological and economical factors that influenced the production of these tapestries.

The rich coloration presented in the sampled rug (Fig.1) suggests the use of distinct dyes. Colour hues can be obtained by the use of different mordants with the same dye. Experienced dyers could also use different dye concentration and dyeing conditions to obtain the desired fibre colour. Moreover, the identification of the mordant is an important issue in the analysis of fibre degradation as its nature affects the acidity of mordanting and dyeing solutions.

Fibre samples were taken from the wool fibres used for embroidery (samples A1 to A9) and from supporting canvas (sample A10). SEM micrographs and EDS spectra and 2D maps were carried out using a JEOL JSM-7001F scanning electron microscope coupled with an OXFORD X-ray energy dispersive spectrometer with a Si(Li) detector. The fibre samples were coated with a thin, conductive film of gold. The current used varied from 10 to 25 kV. The μ-PIXE analysis was performed using the proton beam from the 3 MV Van de Graff accelerator at the Instituto de Tecnologia Nuclear (ITN, Lisbon, Portugal).

The EDS and μ-PIXE results (Fig.2 and Fig.3), presented as representative data for all the samples (A1 to A9), showed the presence of sulphur, silicon, calcium and potassium belonging to the wool composition. Besides these, aluminium was found evenly distributed over the fibres, independently of their colour. The presence of aluminium in the blue fibres samples (samples A1 and A8) is likely an indication that a pre-mordanting procedure of the wool was done by the Arraiolos dyers since indigo, the blue dye identified in these samples, is a vat dye and hence does not require a metal mordant.

EDS analyses also revealed the existence of small traces of lead and tin. However, they were assumed to be a contamination from the vats employed in the wool treatment, as they were barely detected. Salts of copper and iron, along with alum (double sulphate of aluminium and other metal), are reported worldwide as mordants for the yellow and red hues. Historical data on Arraiolos tapestries dyeing techniques only refer the usage of alum [2]. Data on the Rug nr. 88 samples confirm the absence of copper and iron, while aluminium was found in all the analysed samples. The EDX and μ-PIXE results seem to confirm that alum was, in fact, the only mordant used in the dyeing process of wool fibres for the manufacture of Rug nr. 88.

SEM micrographs of fibre surfaces display, in general, the typical scale structure of wool (Fig.4). However, in some cases it was observed roughened surfaces with damage of this scale structure due to wear and natural ageing. The presence of transverse cracks and longitudinal splitting are indicative of embrittlement due to loss of flexibility and elasticity, causing cracks when subjected to bending or stretching. Several types of fractures were observed in the analysed fibres, being the most commonly found a combination of granular and radial fractures. Some fibrillar multiple split ends were also found, but it was likely that they are naturally occurring ends, with fibrillation due to wear [3, 4]. In some cases, biological colonization was found (mould and bacteria) which may also contribute to loss of tensile strength.

In relation to the canvas (rug support matrix) the morphological analysis of the fibres show that the warp threads of Rug nr. 88 are linen, which is in agreement with historical data [2]. Furthermore, in general, the canvas fibres are in good state of conservation presenting only small cracks (Fig.5).

In conclusion, SEM-EDS in combination with μ-PIXE results allowed an evaluation of the fibre surface, fracture morphology and mordant analysis. Alum was used as mordant which is in accordance with the available historical treaties and the rug fibres exhibit the expected degradation due to wear and ageing. These results together with data from the dye identification and their degradation products as well as tensile strength analysis are now being pursued in order to evaluate the overall state of conservation of the Arraiolos museum pieces.

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Figure1. Sampling points in 17th century Rug nr. 88 from the MNAA collection.

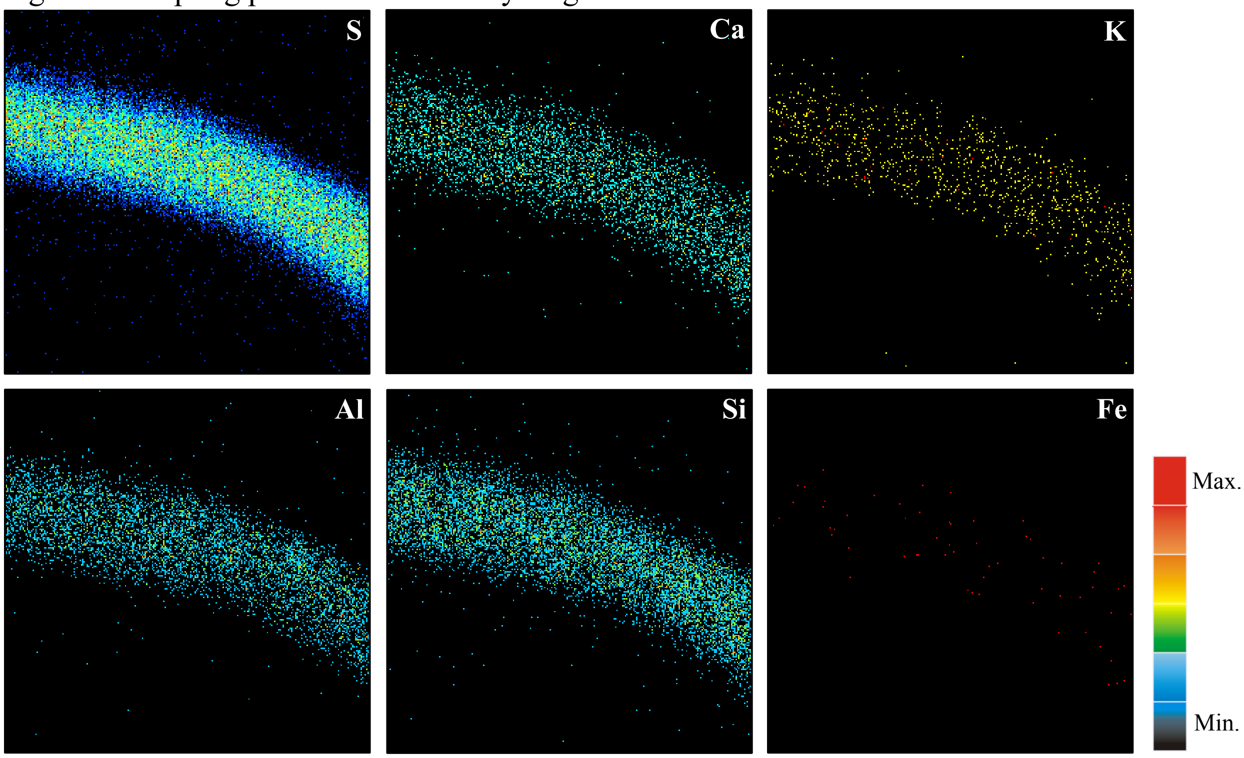

Figure 2. 2D elemental mapping by μ-PIXE of historical samples.

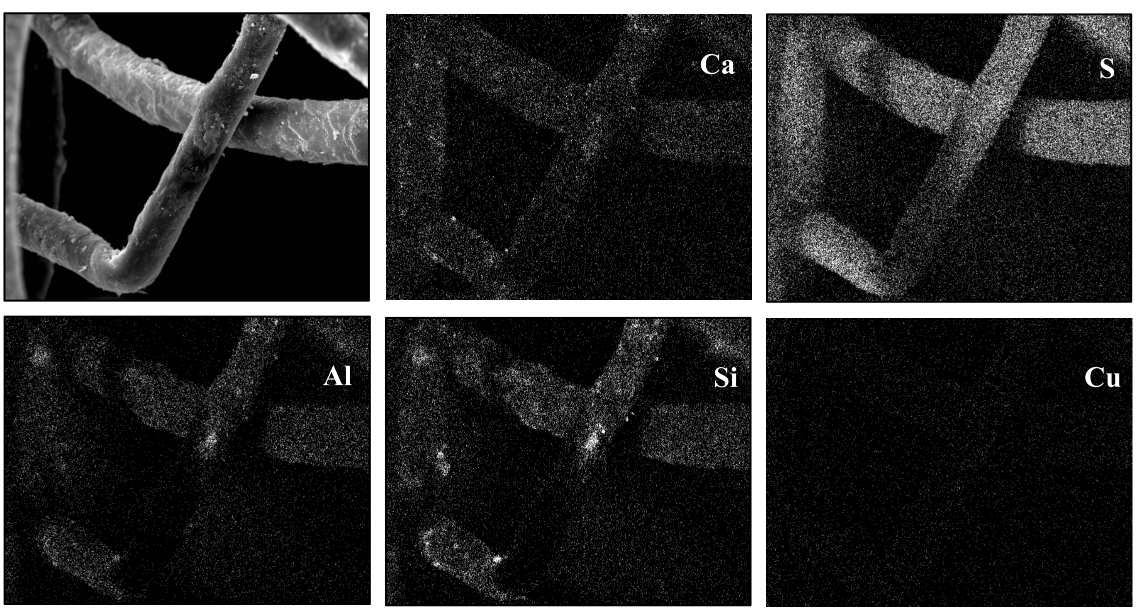

Figure 3. 2D elemental mapping by EDS for sample A1.

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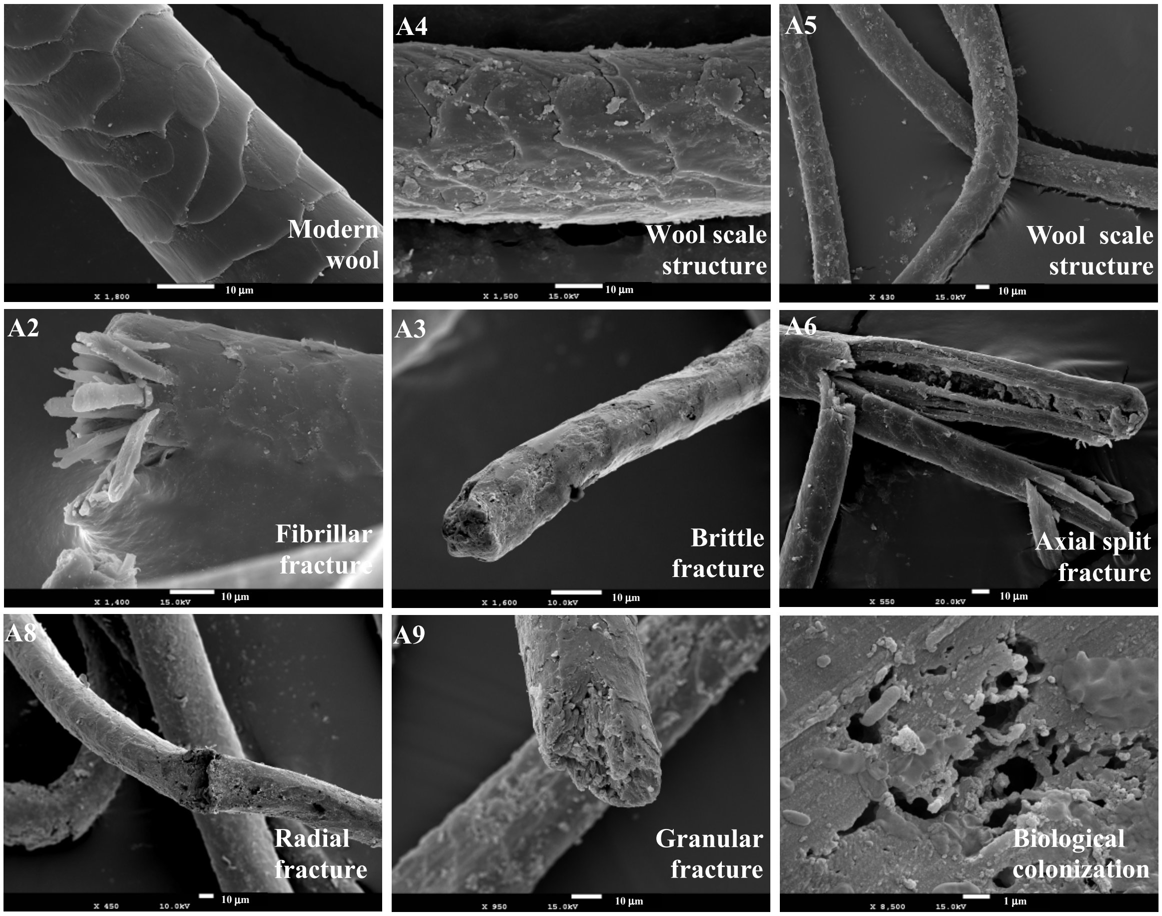

Figure 4. SEM micrographs of the historical fibres exhibiting distinct types of fractures and biological colonization.

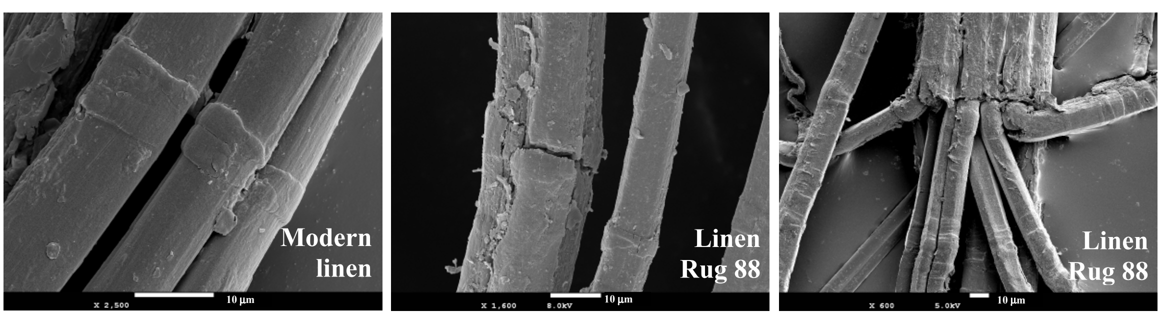

Figure 5. SEM micrographs of modern linen and warp threads from Rug 88 showing that linen was used as the support fibre.

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