

## Combined use of microscopic examination and X-ray spectroscopic analysis for assessing gilding and silvering techniques in Portuguese illuminated manuscripts

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Among the most valuable Portuguese illuminated manuscripts, the *Manuelin foral* charters produced in the early 16<sup>th</sup> century during the reign of D. Manuel I of Portugal well reflect the king's policy aiming at restructuring the fiscal system in the whole country and asserting himself as the regulatory entity of a modern State. These documents on parchment correspond to juridical texts from the 12<sup>th</sup> century onwards copied into renewed codices [1]. All are supplemented by the addition of a full page illuminated frontispiece comprising either heraldic motifs with profusely decorated marginalia (types 1 and 3) or the precious initial "D" (from D. Manuel) and floral borders (type 2) [2]. The present work aims at studying these manuscripts illuminated with gold and silver, in order to broaden our understanding of the gilding and silvering techniques applied on parchment between 1500 and 1520, in Portugal.

Ten *Manuelin foral* charters, well representative of the existing typologies, were selected and systematically compared. In order to provide the characterization of the parchment and precious coatings used, a thorough microscopic examination of the manuscripts was performed by means of : Stereoscopic microscopy (60-120×) equipped with normal and raking light and a digital camera, Digital Microscopy (Dino-Lite) equipped with visible and ultraviolet radiation for colorful images (215×) and Scanning Electron Microscopy (SEM) imaging for high magnification (up to 10,000×) to observe the metallic coatings morphology and thickness. Non-destructive analysis was carried out *in situ* by Energy Dispersive X-ray Fluorescence (EDXRF) with portable instrument, for obtaining a previous elemental composition (Fig. 1). Further analysis providing qualitative and semi-quantitative data, namely for evaluating the gold and silver fineness, was performed on a limited number of micro-samples by SEM coupled with Energy Dispersive X-ray Spectroscopy (EDS) equipped with mapping facilities.

These combined techniques allowed to put in evidence the vestigial materials from the processes undertaken for the parchment making [3] with salt (NaCl), lime (CaO) and pumice stone (SiO<sub>2</sub>+Al<sub>2</sub>O<sub>3</sub>) [2], and chalk (CaCO<sub>3</sub>) too, as white inert employed to give parchment its whiteness and opacity (Fig. 2). Shell-gold and shell-silver as powdered metals were directly implemented on parchment by means of animal glue or gum binding media in charters of type 1 and 3, while very thin gold leaf (< 1 μm) was applied over lead-based tempera preparatory layers (120-180 μm thick) in charters of type 2 (Fig. 3). Silver was always employed in its finest form but without further protective layer (thus its recursive state of corrosion) while gold was used in different grades (24, 23.5, 22 and 9 ct) according to the ornamental needs.

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- [1] Garcia J.M., *Os forais novos do reinado de D. Manuel*, Lisboa: Banco de Portugal, 2009.  
 [2] Le Gac A. *et al.*, *Applied Radiation and Isotopes*, **82**:242-257, 2013.  
 [3] Le Gac A., in *Parchment and leather heritage. Conservation-Restoration*, Torun: Wydawnictwo Naukowe Uniwersytetu Mikolaja Kopernika Publ., p. 31-60, 2012.

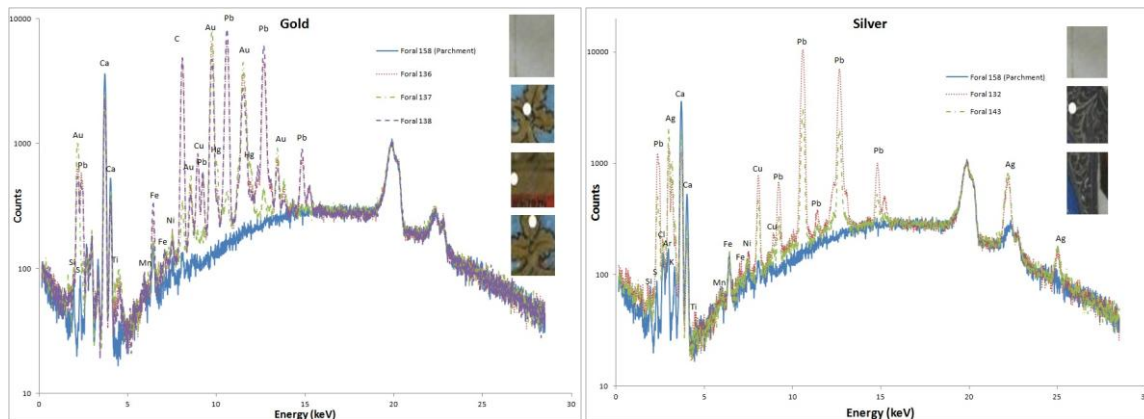


Figure 1. EDXRF spectra of gold and silver coatings (with parchment) from *Foral of Teixedo*

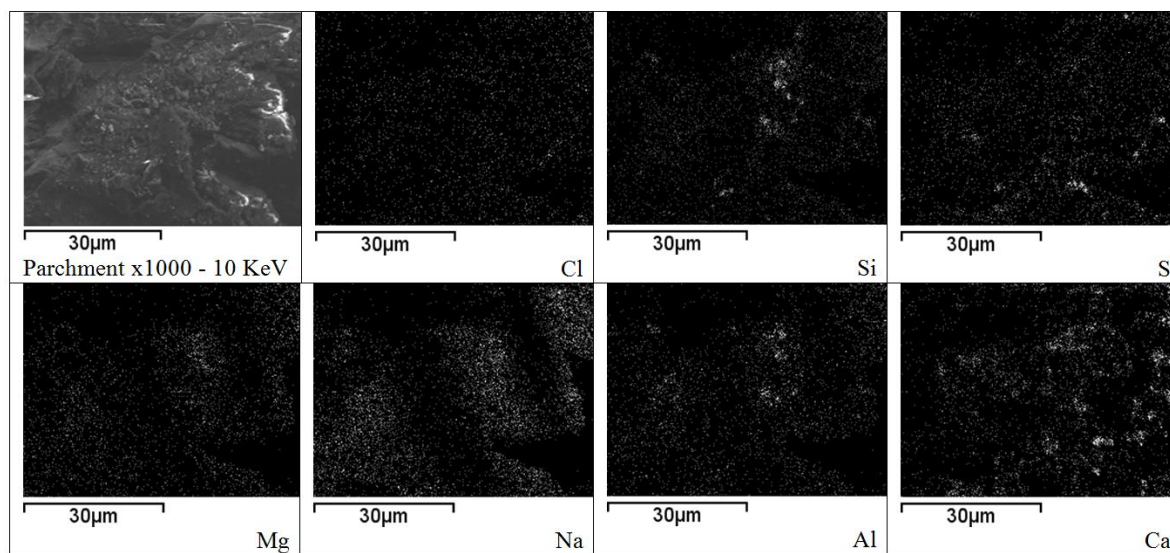


Figure 2. SEM-EDS Mapping of parchment from *Foral of Castanheira & Povos*

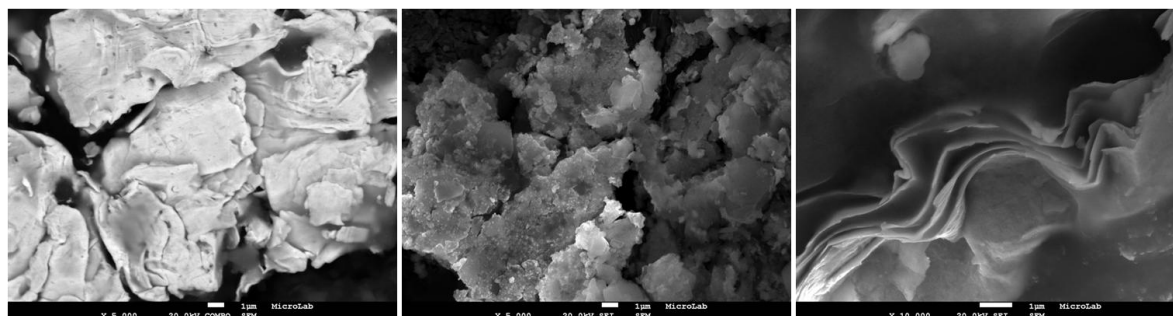


Figure 3. SEM Imaging: Shell-gold in *Foral of Juromenha* (BSE-5,000×); Shell-silver in *Foral of Abiul* (SE-5,000×); Gold hand-beaten leaf in *Foral of Mões* (SE-10,000×).