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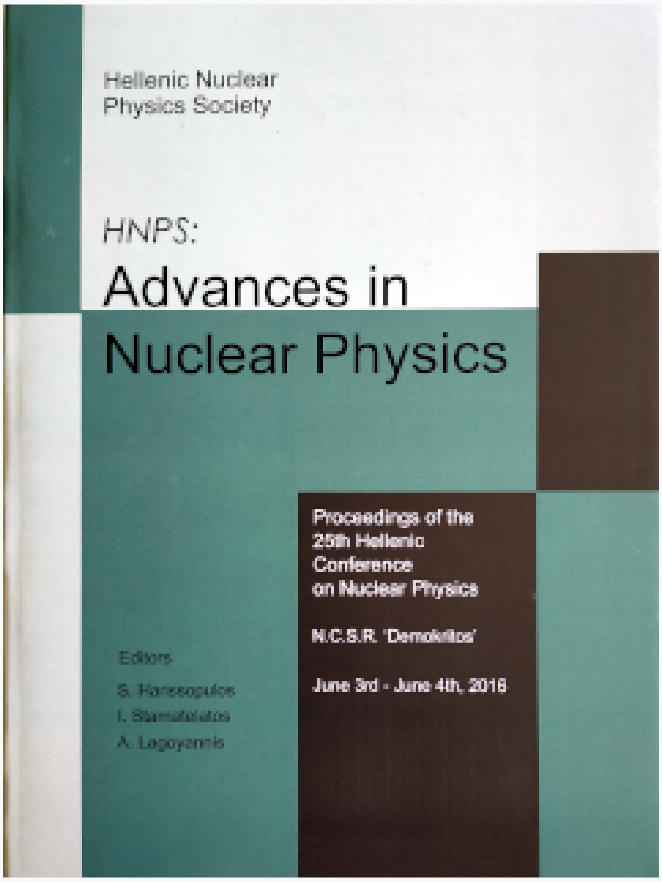

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Assessing chelators' efficiency in removing iron stains from textiles using X-ray fluorescence: Preliminary results

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Abstract The removal of iron corrosion products is considered necessary in textile conservation and it is usually undertaken by the application of chelating agents. The efficacy of the various chelating agents is not always known and therefore, non-destructive analytical methods are often used to assess their performance. In the present work, Energy Dispersive X-ray fluorescence (EDXRF) spectroscopy was used to determine iron in textile samples before and after cleaning. It was shown that EDXRF provides a simple, rapid, non-destructive technique to determine the efficiency of iron removal after conservation treatment in textiles.

Keywords Energy Dispersive X-ray fluorescence (EDXRF), chelating agents, textile conservation

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INTRODUCTION

Ethnographic collections often consist of historical textiles that might have been in contact with iron elements. If such artefacts are stored or exhibited under uncontrolled environmental conditions, corrosion of the iron elements is expected to occur and corrosion products might migrate, stain the artefacts and catalyze various oxidation processes. The removal of iron corrosion products is considered necessary in textile conservation and it is usually undertaken by the use of chelating agents [1]. The efficacy of the various chelating agents is not always known and therefore, non destructive analytical methods are often used to assess their performance.

X-Ray Fluoresence (XRF) spectrometry is used to determine the composition of a sample by measuring the X-rays emitted from a sample when it is excited by a primary X-ray source. Each of the elements present in a sample emits a set of fluorescent X-rays characteristic for the specific element. The intensity of the X-Rays increases with the concentration of the corresponding element. Therefore, XRF spectroscopy enables qualitative and quantitative analysis of the composition of the sample material composition.

In the present work, we examined the feasibility of Energy Dispersive X-ray fluorescence (EDXRF) analysis to measure iron in textile samples before and after being treated with a chelating agent in order to determine the treatment efficiency. The effect of measurement parameters such as the X-ray source filter material and thickness used is discussed.

MATERIALS AND METHOD

The cotton samples analyzed in this study were stained with iron oxides and then cleaned using a chelator applied by gel (Fig. 1). Three samples were analyzed representing a reference, a stained and a cleaned sample. The sample thickness was 0.6±0.02 mm and the sample sizes were 2.5×2.5 cm^2.

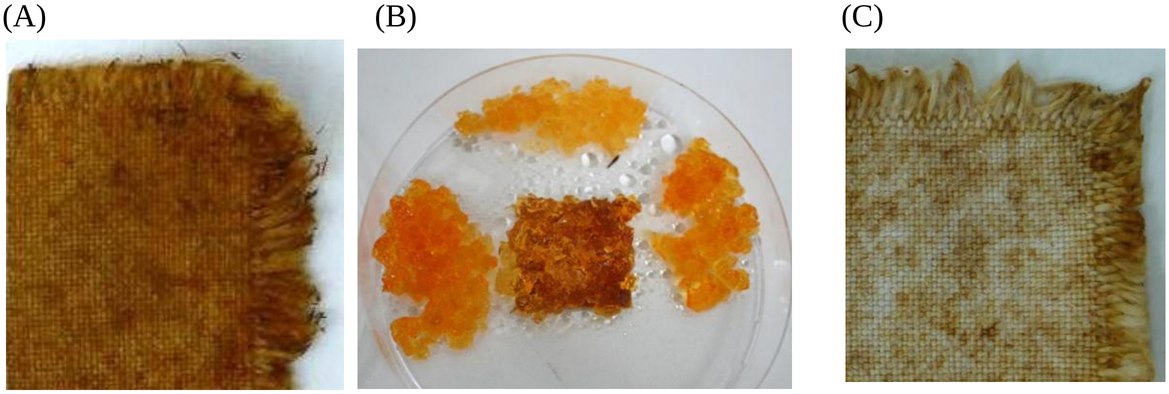

Fig. 1 Cotton samples (A) before cleaning (B) during and (C) after cleaning procedure

The EDXRF system used was developed by Amptek Inc. It consists of a Mini-X X-ray tube with $_{47}$Ag anode and the X-123 detection system including a silicon drift detector, a digital pulse processor and a multi-channel analyser. The system specifications are shown in Table 1. The samples were mounted on a polyethylene sample holder. DppMCA software was used for data acquisition and XRS-FP software was employed for quantitative analysis of the XRF spectra.

X-ray tube	Detector
Voltage/Current: 10-50 kV / 5-200 μA	Type: Silicon drift detector
Collimators' diameter: 1 and 2 mm	Resolution: 125-140 eV FWHM at 5.9keV
Filters available: Al, Cu, Mo, Ag, W	Maximum count rate > $5.6 \cdot 10^5$ cps

Table 1. Specifications of the EDXRF system

The EDXRF spectrometer directly measures the energies of the characteristic fluorescent X-rays emitted from a sample when it is excited by primary X-rays. The peak positions of the spectrum define which elements are present (qualitative analysis) while the peak intensities designate their concentrations (quantitative analysis). Fundamental Parameters (FP) method is used for quantitative analysis, where the concentration calculations are based on the Sherman equation [2]:

$$C_i = R_i\left(\frac{1+\sum_j \alpha_{ij} C_j}{1+\sum_j \varepsilon_{ij} C_j}\right) \qquad (1)$$

where C_i is the concentration of the analyte i, R_i is the relative peak intensity of the analyte i, C_j is the concentration of the matrix element j and α_{ij}, ε_{ij} are the influence coefficients correcting the secondary absorption effect and the enhancement effect of the matrix element j on the analyte i, respectively.

RESULTS AND DISCUSSION

XRF spectra were obtained from background, the reference textile, the stained textile, and a cleaned textile, in order to determine iron and correct the iron peak from background interferences due to the presence of iron in components of the experimental device. As it can be seen in Fig. 2(a) several elements were identified. Nickel, copper and zinc are elements originating from parts of the apparatus, i.e. the metallic base and the beam collimator. Argon is constituent of the air while chlorine comes from the plastic holder used to mount the filter at the end of the X-ray tube.

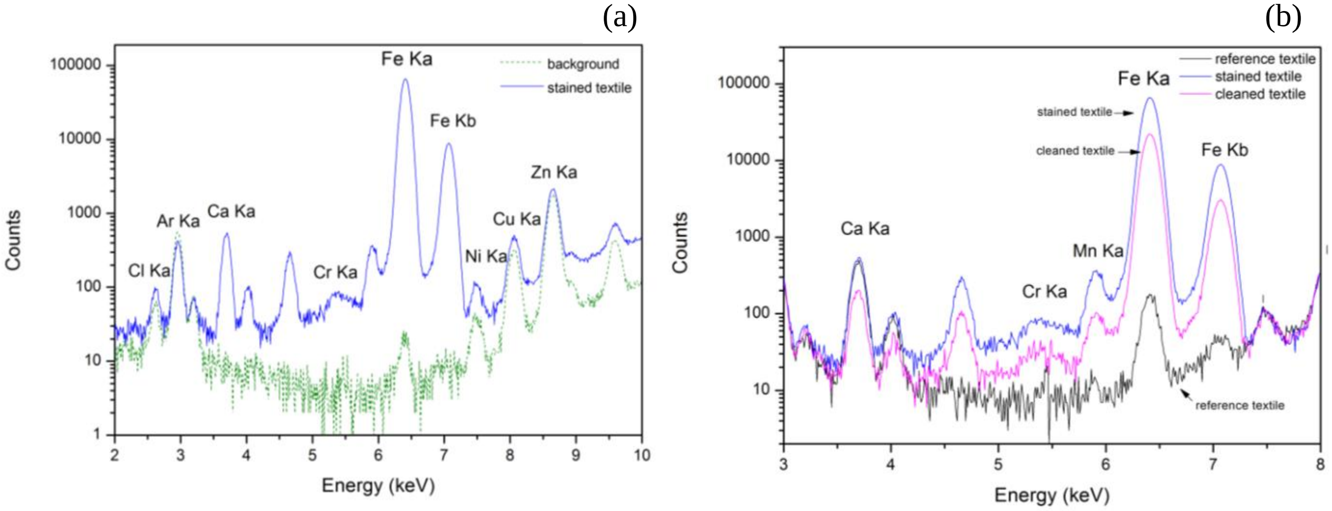

Fig. 2 XRF spectra of (a) the background and the stained textile and (b) of the iron stained sample before and after cleaning, as well as the reference sample.

The settings used for the measurements were 2×250 μm Al filters, 2mm collimator, voltage 19.9±0.1 kV, current 30.3±0.1 μA and acquisition time of 1800 s. The filter material and its thickness are important parameters for the optimization of the measurement. The filter built into the primary X-ray beam allows modification of the X-ray energy to preferentially enhance the analysis of the desired elements in the specific matrix. The selection of the aluminum filter with a total thickness of 500 μm was based on a comparison study of the available filters (Al 1000, 500 and 250 μm, Cu 75 μm, Mo 50 μm, Ag 25 μm and W 25 μm). The selected filter provided the best relative error of measurement for the Fe Kα peak. Moreover, Fig 2(b) shows the XRF spectra obtained from a stained cotton sample before and after cleaning treatment in comparison to the reference textile. The results of this study showed that the application of the gel technique reduced the iron content in the textile stained textile sample by a factor of 0.65. In this figure the XRF spectrum from the reference sample is also shown for comparison. The reference spectrum indicates the desired final result of a successful cleaning procedure for conservation.

CONCLUSIONS

Spectrometric analysis methods such as X-ray fluorescence (XRF) spectroscopy or scanning electron microscopy coupled with energy dispersive X-ray micro-analysis (SEM/EDXA) are methods that are now commonly applied to the analysis of metal threads in textiles. These methods offer excellent sensitivity, perform multi-element analysis and enable identification of the metal threads in textiles. Although EDXRF spectroscopy cannot provide information on local inhomogeneities that can be obtained by other more elaborate X-ray techniques and methods, it was shown capable to provide a rapid, non-destructive measurement enabling to determine specific thread metals in textiles and therefore may offer significant assistance to conservationists enabling them to determine the efficiency of different cleaning procedures and techniques on the examined samples. Apart from the iron, which was the element of main interest, calcium, chromium and manganese were also evident in the spectra. The concentration of the above elements has also decreased after the cleaning treatment.

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