

Article

# Chromatographic and Spectroscopic Identification and Recognition of Natural Dyes, Uncommon Dyestuff Components, and Mordants: Case Study of a 16th Century Carpet with Chintamani Motifs

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**Abstract:** A multi-tool analytical practice was used for the characterisation of a 16th century carpet manufactured in Cairo. A mild extraction method with hydrofluoric acid has been evaluated in order to isolate intact flavonoids and their glycosides, anthraquinones, tannins, and indigoids from fibre samples. High-performance liquid chromatography coupled to spectroscopic and mass spectrometric detectors was used for the identification of possible marker compounds with special attention paid to natural dyes present in the historical samples. Weld, young fustic, and soluble redwood dye were identified as the dye sources in yellow thread samples. Based on the developed method, it was possible to establish that red fibres were coloured with lac dye, whereas green fibre shades were obtained with indigo and weld. Tannin-containing plant material in combination with indigo and weld were used to obtain the brown hue of the thread. Hyphenation of high-performance liquid chromatography (HPLC) with quadrupole time-of-flight mass spectrometry (QTOF MS) and triple-quadrupole mass spectrometry (QqQ MS) enabled us to recognise four uncommon and thus far unknown dye components that were also found in the historical samples. These compounds probably represent a unique fingerprint of dyed threads manufactured in a Turkish workshop. Scanning electron microscopy with energy-dispersive X-ray detector (SEM-EDS) and Fourier transform infrared spectroscopy (FT-IR) were used for the identification and characterisation of substrates and mordants present in the historical carpet. Carbon and oxygen were detected in large quantities as a part of the wool protein. The presence of aluminium, iron, and calcium indicated their usage as mordants. Trace amounts of copper, silica, and magnesium might originate from the contaminants. FT-IR analysis showed bands characteristic for woollen fibres and SEM micrographs defined the structure of the wool.

**Keywords:** natural dyes; flavonoids; flavone glycosides; anthraquinones; extraction procedure; liquid chromatography mass spectrometry

## 1. Introduction

Scientific analysis of objects of artistic and historic significance is the key to reconstructing their story and elucidating the circumstances under which they have been created. Investigation of the origin, nature, and chemical behaviour of the coloured materials used in the production of historical artefacts may shed new light on their original colour and appearance [1]. Knowledge of historical