CHARISMA

Cultural Heritage Advanced Research Infrastructures:
Synergy for a Multidisciplinary Approach to Conservation/Restoration

Rare compounds studies in *Reseda luteola* L. and *Rubia tinctorum* L.

Selection of Rare Components

Art Néss Proaño Gaibor[1], Susanna Bracci[2], **Maarten van Bommel**[1*], **Jo Kirby Atkinson**[3], **David Peggie**[3]

The most encountered colorant compounds in dyed textiles and lake pigments in cultural heritage objects from Europe derived from weld (*Reseda luteola* L.) and madder (*Rubia tinctorum* L.) plants. Some of the components present in these dye plants have been elucidated and are broadly available as chemical standards; however many components in these plants are not, despite the fact, that they are often found in these objects and play a role in the colouring mechanism.

Within a joint research of the *Cultural Heritage Advanced Research Infrastructures: Synergy for a Multidisciplinary Approach to Conservation/Restoration* (CHARISMA) project, a method was developed to isolate these rare compounds from the plants in order to study their behavior and elucidate their structure, if possible. This method can be used to isolated pure standard components and share them with laboratories working in the field of colorant identification. A preparative liquid chromatography system along with a fraction collector (fig.2) was set up to isolate flavonoid and anthraquinonic components from concentrated weld and madder extractions. In figure 1 the selected fractions are shown.

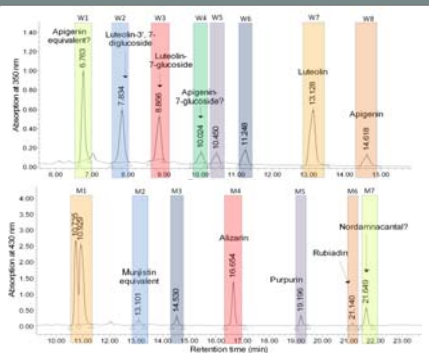


Fig.1 Chromatogram weld (above) and *madder* (beneath) showing in coloured bars the selected fractions

Fraction Collection of Rare Standards

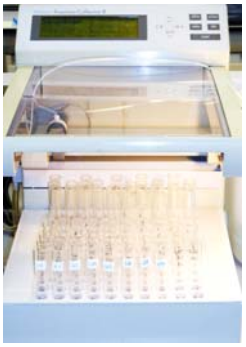


Fig.2 fraction collector

These samples (called fractions, fig.3) are examined for purity with a high performance chromatography system with photodiode array detector. The fractions are hydrolyzed to identify its aglycone (fig.4). The pure fractions are then analyzed with an electro spray ionization (ESI) Quadrapole time-of-flight (Q-tof) mass spectrometer (fig.5) and also with nuclear magnetic resonance (^{1}H and ^{13}C-NMR).

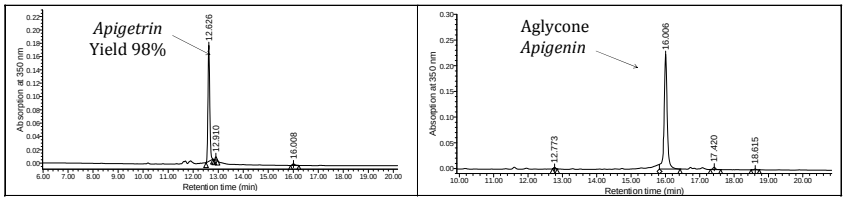


Fig.4 Chromatograms of one weld fraction (L) and same fraction after hydrolysis (R)

Fig.3 Collected fractions

Fig.5 ESI Q-Tof with CapLC

Elucidations and future distribution

Several rare standard of *Reseda luteola L.* and *Rubia tinctorum L.* were obtained with high purity. Most of these standards were identified with mass spectrometry fragmentation using collisional-induced dissociation (MS/MS) in both positive and negative mode. An example is reported in the spectrum on the right (fig.6). The obtained standards are shown below with its respective molecular structure, those with a question mark still need to be confirmed by NMR. After complete identification we will proceed with the distribution of these standards to the interested partners within the CHARISMA project. This method can be also used to analyse and isolate unknown components in other similar flavonoid and anthraquinonic dye plants.

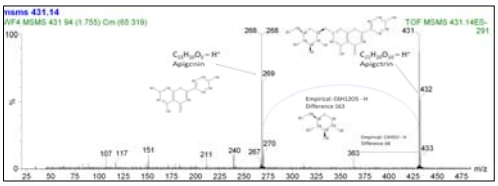
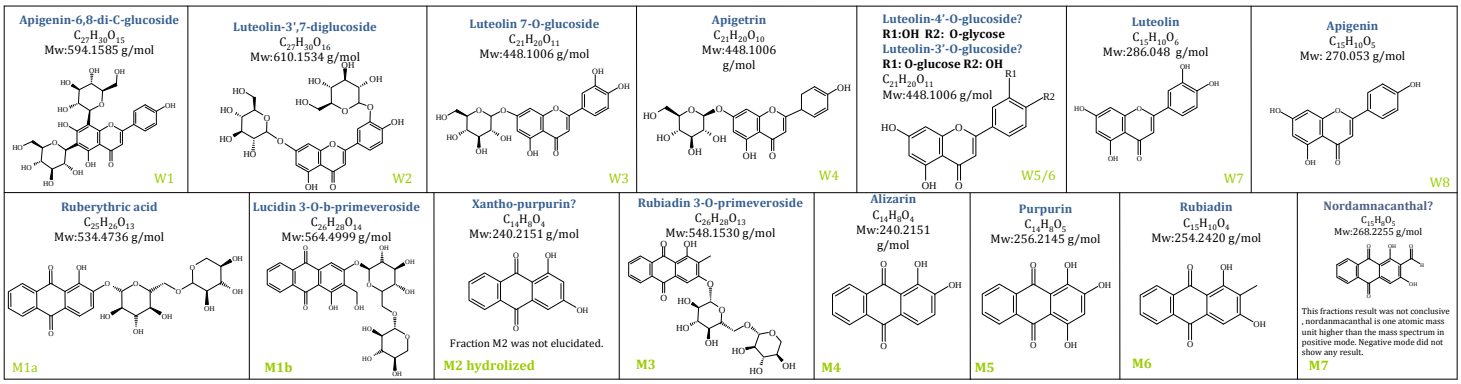


Fig.6 Weld Fraction; MS/MS negative mode Spectrum



Collaborating partners:
1 - Cultural Heritage Agency of the Netherlands (OCW-RCE)
2 – Institute for the Conservation and Promotion of Cultural Heritage (ICVBC-CNR), Italy
3 - The National Gallery London (NGL), United Kingdom

***Corresponding author: m.van.bommel@cultureelerfgoed.nl Cultural Heritage Agency of the Netherlands (OCW-RCE)**

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