NATURAL DYES IN MADDER (*RUBIA* SPP.) AND THEIR EXTRACTION AND ANALYSIS IN HISTORICAL TEXTILES

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Type of communication: Oral ☑ Poster ☐

Abstract *(max 500 words including references)*

Textiles coloration using extracts from the roots of various madder species (Rubia spp.) has been performed for centuries. To date, 68 anthraquinone colorants have been detected in Rubia spp. used to dye textiles. Many of these dyes are sensitive to hydrolysis and degradation from enzymes, extraction chemicals and processing temperatures (Figure 1), and are often overlooked as colorants in historical textiles. Conclusions in literature of the past 30 years concerning colorants present *in planta* and, particularly, in madder-dyed artefacts are being challenged as new analysis methods are developed.

The recent advent of 'soft' extraction techniques has demonstrated that anthraquinone glycosides and other sensitive molecules, such as carboxylated compounds, need to be preserved; this valuable chemical information embedded in the dye structure may be lost if extraction and analysis is too harsh. Some compounds thought to be present in madder and madder-dyed artefacts are in fact degradation products resultant from the extraction process, and degradation pathways have been developed to better understand the reactivity and stability of these compounds. Detailed analysis of dyes in textile artefacts can reveal important cultural and heritage information concerning historical textiles relative to the specific dye species, the area of the world where this may have grown, how and where it was dyed, and, perhaps, where it was traded. 'Soft' extraction techniques are needed in the analysis of madder-dyed textiles due to the sensitive nature of these molecules, and their preservation up to the point of detection is key to unlocking full and valuable chemical information embedded in the dye structure, which otherwise is lost.

Another key aspect is one of preservation, and the aim is to fully understand the nature of these dyestuffs in order to determine degradation patterns and structure-efficient conservation techniques. Understanding the precise molecular structure of these natural dyes and their chemical reactivity is important to provide knowledge of their interactions with physical substrates, such as textile fibres, which could be used to develop superior techniques for analysis of artefacts.

The flowchart in Figure 2 is proposed as a decision tool to enable *Rubia* species to be distinguished from one another based on compounds detected in extraction and analysis of dyed fibre samples; this assumes that the extraction methods used are soft and preserve chemical structure integrity to enable a valid assessment, but does take into account very sensitive components such as galiosin and pseudopurpurin.

The future of the detection of colorants in the study of artefacts could involve sequential application of different analysis methods where each technique could be used for maximum advantage. For example, an initial analysis of *Rubia*-dyed fibres with SERS to ascertain concentrations of pseudopurpurin (notably very unstable to extraction), followed by soft extraction and analysis by HPLC-DAD/LC-MS, could provide an all-encompassing method that provides maximum detection.

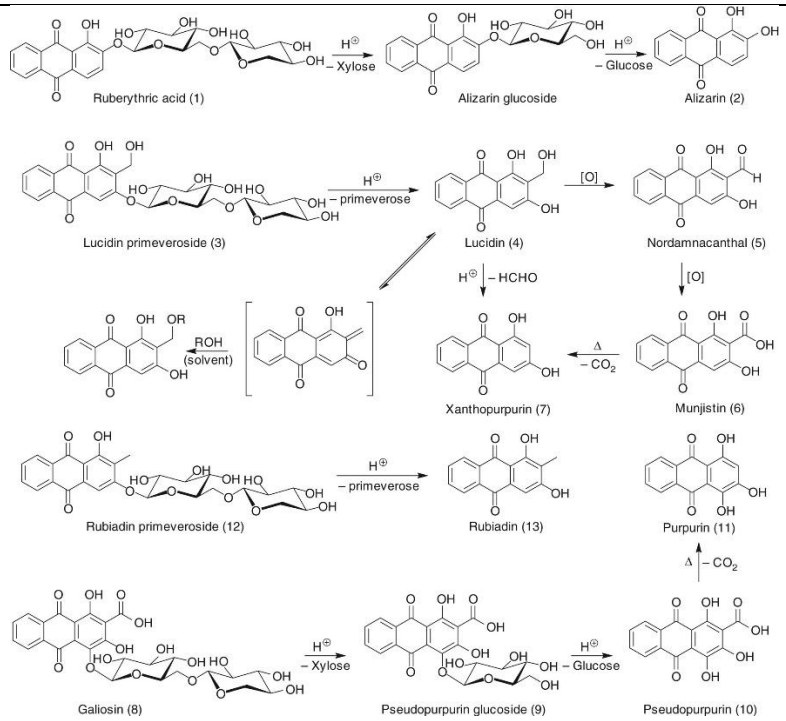

Figure 1. Possible inter-relationships between anthraquinone compounds found in *Rubia* species based on chemical or biochemical interconversion.

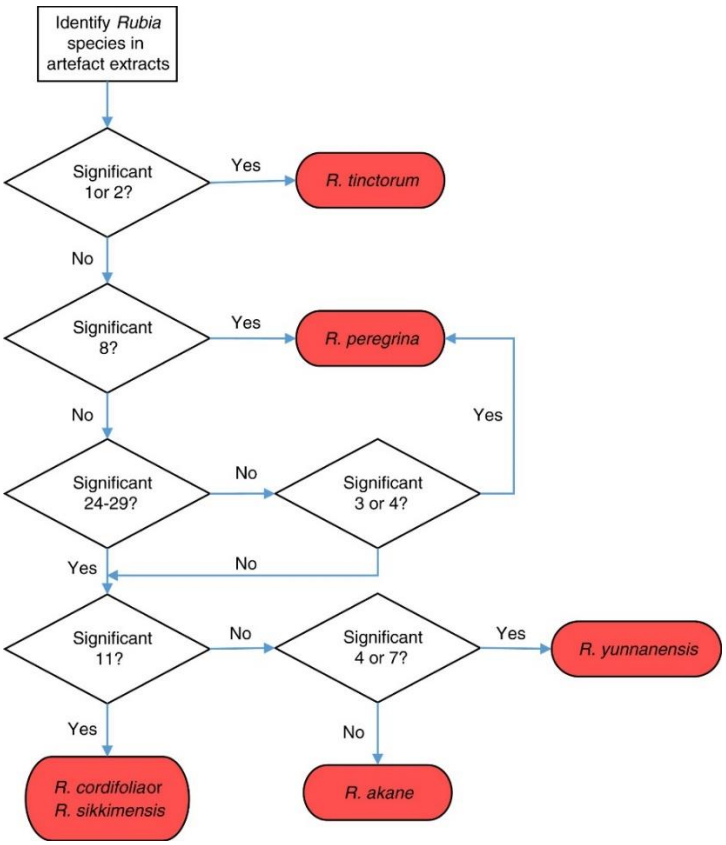

Figure 2. Decision flowchart to enable *Rubia* species to be distinguished based on compounds extracted and analysed in dyed fibre samples.