

**STUDIES IN CHARACTERISATION AND  
DYEING BEHAVIOUR OF SOME COMPONENTS  
OF INDIAN MADDER (*Rubia cordifolia*) ON  
NYLON AND POLYESTER**

by

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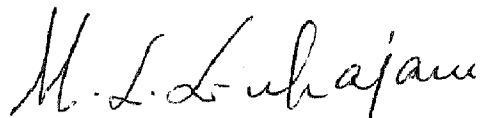
## CERTIFICATE

This is to certify that the thesis entitled "**Studies in Characterisation and Dyeing Behaviour of Some Components of Indian Madder (*Rubia cordifolia*) on Nylon and Polyester**", being submitted by **Ms. Shipra Kumari**, to the **Indian Institute of Technology, Delhi**, for the award of the degree of **Doctor of Philosophy** in the **Department of Textile Technology**, is a record of bonafide research work carried out by her. Ms. Shipra Kumari has worked under our guidance and supervision, and fulfilled the requirements for submission of the thesis.

The results contained in this thesis have not been submitted, in part or in full, to any other University or Institute for the award of any degree or diploma.



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*MAA & PAPA*

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## ABSTRACT

Madder is a historical dye and has always remained a topic of research since madder dyed fabric was first found long ago in Egyptian mummies. There are two main dye yielding species of madder i.e., *Rubia tinctorum* and *Rubia cordifolia*, out of them later one is of Indian origin. It is reported to contain several anthraquinone derivatives differing only in their substitution pattern. Chemists have undertaken the work of isolation, purification and characterisation of these anthraquinone colourants. However the characterising data presented are most often scattered and incomplete. Conflicting results have also been reported in some cases.

The present work has been undertaken with the objective of developing optimised techniques for separation of major components using chromatographic methods and to generate the characterising spectroscopic data for the purified components. Efforts have also been made to study the dyeing, light and wash fastness characteristics of pure components.

Methods to characterise the coloured components of the madder extract by Thin Layer Chromatography and High Performance Liquid Chromatography have been optimised. Preparative column chromatography has been used to collect the components in large amounts that can further be scaled up for commercial production.

Characterisation was done using the spectroscopic techniques, such as UV-Vis, FTIR, <sup>1</sup>HNMR, MS and melting point. These spectroscopic data can subsequently be used for identifying the components in museum textiles and for certification and authentication of natural dyes.

Three major components have been purified and characterised. They are nordamncanthal (1, 3-dihydroxy-2-formyl anthraquinone), purpurin (1, 2, 4-

trihydroxy anthraquinone), and munjistin (1, 3-dihydroxy-2-carboxy anthraquinone). They account for approximately 80-90% of the coloring matter of the madder extract.

The sensitivity of the components to temperature and pH change have been studied and reported. Purpurin was found to decompose above 90°C.

The results of kinetic studies conducted showed that diffusion of dyes on nylon is good. However, the dye uptake of munjistin on polyester was poor. The rate of dyeing increased with increase in dyeing temperature. The values of diffusion coefficient and time of half dyeing of three colourants are reported.

In equilibrium studies it was observed that the sorption pattern of the colourants were quite different. It was evident that the basic structure of colourant being same, the substitution group played an important role in deciding the adsorption mechanism. Munjistin having ionic group showed Langmuir isotherm on nylon showing that adsorption of dye is through site mechanism. Purpurin and nordamncanthal are sorbed by the partition mechanism because of their hydrophobic nature.

Thermodynamic parameters such as affinity, heat of dyeing and entropy have been calculated and reported.

Photo-fading and light fastness of these colourants on nylon were also investigated. Effect of mordant and mordanting method on colour obtained and fading rate has been studied. The light fading behavior of these anthraquinone derivatives were greatly dependent on the type and position of substituents present. Nordamncanthal and munjistin having formyl and carboxyl group respectively showed poor light resistance. Concentration of mordant affected the colour but not the fading behaviour. Post mordanting with ferrous and copper sulphate improved light resistance and slowed down the fading rate.

Light and wash fastness grade of dyeings with these colourants have also been evaluated and included.

The various investigations clearly show that the components of madder have the same basic chemical structure i.e. hydroxy anthraquinones, but behave differently solely because of a difference in the substitution pattern.

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