SYNTHESIS AND ANALYSIS OF THE DYE INDIGO CARMINE FROM INDIGO USING SERRS

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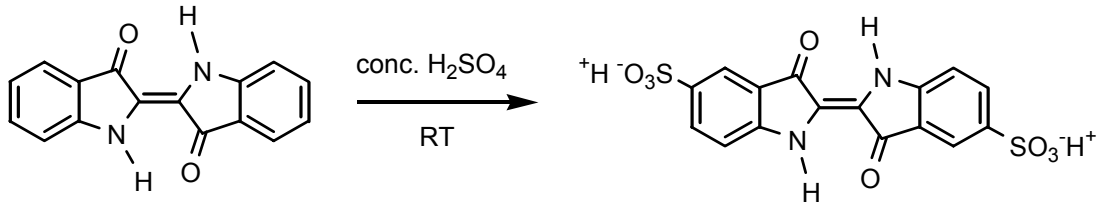
Abstract: In a novel application SERRS has been used to monitor the synthesis of indigo carmine from indigo. The kinetics of the conversion has also been monitored by SERRS.

Surface enhanced resonance Raman spectroscopy (SERRS) has found wide utility as a sensitive analytical tool in a multitude of interdisciplinary scientific investigations (including e.g. chemical analysis, clinical, forensic, environmental and biological sciences, archaeological/historical applications and drug analysis) [1]. There has been a growing interest in the use of SERRS for highly sensitive quantitative chemical analyses, in a wide range of matrices, even down to the single molecule level [2, 3]. In an attempt to extend and develop novel applications of SERRS (using a silver sol) the technique has been used to monitor and analyse the synthesis of the dye indigo carmine from indigo (see Fig.1).



(1) **(2)**

Fig. 1. Schematic of the chemical structures of indigo (**1**) and indigo carmine (**2**) and the reaction showing the conversion of indigo to indigo carmine.

A silver sol, for use as a SERRS substrate, was prepared via a modified Lee-Meisel procedure [4]. It is immediately obvious from the SERRS spectra shown in Fig. 2, that the synthetic and reference samples of indigo carmine are identical. This conclusion was reinforced by MS/NMR data (not shown) of the two compounds.

This study shows that SERRS can be used to monitor synthetic reactions at the micromolar level, as exemplified by the indigo carmine concentration of *ca*., 5 x 10^{-6} mol dm^{-3} in the final silver sol. Indeed a limit of detection of 1 ppm for the SERRS analysis of indigo carmine has recently been reported [5]. Thus the ultrasensitivity of the SERRS technique together with the ease and rapidity of analytical measurements makes this a powerful tool for monitoring the progress of synthetic reactions (involving reactant(s) and /or product(s) which are fluorescent) conducted at the microscale level.

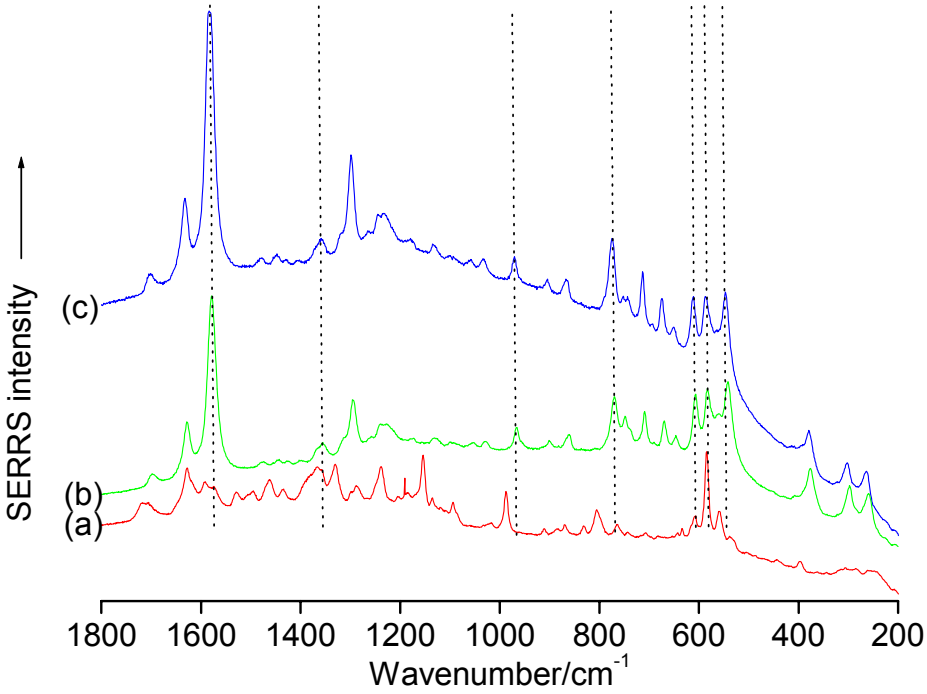


Fig. 2. SERRS spectra of (a) indigo, (b) indigo carmine synthesized from indigo and (c) a reference sample of indigo carmine.

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