

Analytical technique: **Fluorescence Spectroscopy**

Sampling and type of analysis: this technique can be applied to solid samples, powders, films and solutions of materials and is ideal for the measurement of the emission and excitation spectra of materials which may provide indications of the presence (qualitative) of fluorophores or chromophores. When applied to solid samples, the technique is totally non destructive. The analysis of very dilute solutions (<1 mg in a suitable solvent) can lead to the detection of trace organic materials or impurities. However, the solutions are not destroyed or degraded as part of analysis and hence this remains a non-destructive technique.

Principle of the technique: a sample (either solid, or liquid) is probed using an excitation of a specific wavelength and the fluorescence emission is detected; this can be achieved either with fibre optics or by placing a sample within the spectrometer. Both excitation wavelength and emission wavelength can be varied or scanned by use of excitation and emission monochromators. Thus, the spectral characteristics of materials can be determined and it may be possible to map the presence of various different chromophores in a system due to differing excitation or emission profiles.

Analytical procedure for the analysis of samples in CIBA: various types of samples either for direct analysis (without preparation) by placing samples on silica quartz discs and using a fibre-optic sampler, or by dissolving sample (1 mg) in distilled water, dichloromethane (analytical grade) and placing dissolved samples in a 1 cm x 1 cm quartz cuvette within the spectrometer.

Instrumental parameters: a Jobin Yvon Fluorolog spectrofluorimeter was employed for analysis, equipped with a double monochromator in excitation and a single monochromator in emission and a Xenon lamp as an excitation source. Samples were analysed at with slits of 5 nm for each monochromator with either 90 ° between the two detectors or 23 ° between detectors for highly scattering samples (front face).

Synchronous scans were recorded with various offsets between excitation and emission wavelength, with ranges typically from 15 – 250 nm for analysis using fibreoptics/10-250 nm for the analysis of solutions and with excitation wavelengths ranging from 250-500 nm. A scanning speed of 10 nm per second was selected. Samples were probed with a series of synchronous fluorescence spectra which were recorded in a cube of matrices using Origin. These were then analysed using MATLAB.

Information yielded: Spectrofluorescence can yield information regarding the presence of fluorophores which are characterised by their excitation/absorption and emission spectra; such spectra can be presented in a variety of different ways including the use of excitation emission spectra. Fluorescence spectroscopy is particularly suited for the screening of organic materials and has been widely employed in the analysis of oils, foods, proteins and colourants. While the properties of fluorophores in complex mixtures rarely allow the separation of different molecular components, fluorescence can nonetheless be useful for the examination and characterisation of the condition of different materials as a function of ageing.

Example of analysis: the fluorescence excitation emission spectrum of reference material known as “Decotto di rose Verde” is shown below:

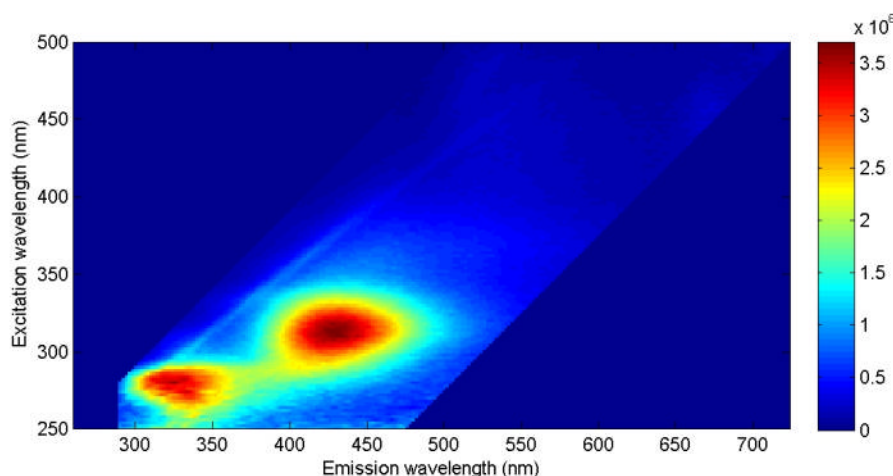


Figure 1 Excitation Emission spectrum recorded from an aqueous solution of “docotto di rose verde”

Clear emission maxima are observed in Figure 1 which is characterised by two major peaks, the first centred at excitation: emission of 270:325 and the second at 330:440. This type of spectrum can be considered characteristic for the extracted reference material and thus allows a rapid non-destructive assessment not of specific molecular compounds, but rather differences between the sample which occur during ageing.