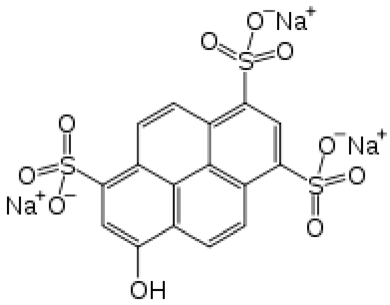
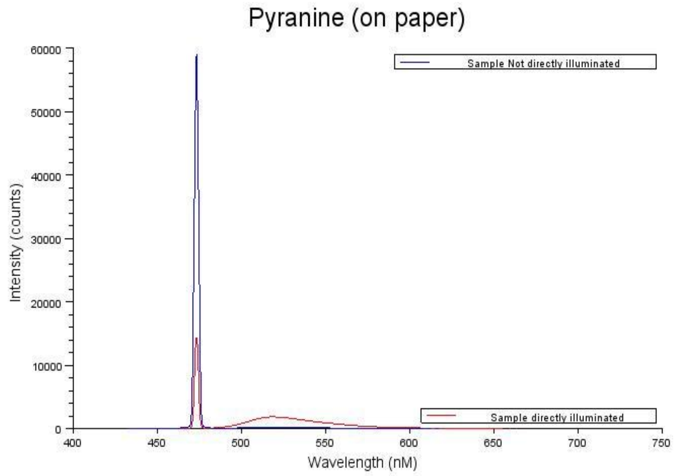
Quantum Yield Measurement of Photo-luminescent Materials.

Preliminary Results



Pyranine: a fluorescent dye



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Abstract:

A brief description of the preliminary project results is presented. Consideration has been given to the experimental method and set up, equipment design and manufacture, suitability of data processing and any further necessary developments. Initial relative results are included for a single sample material (Pyranine) as proof of principal.

Contents

1. Experiment Design.

As described in the literature review the experiment was developed by deMello et al [1] and the schematic layout is shown in fig 1.1.

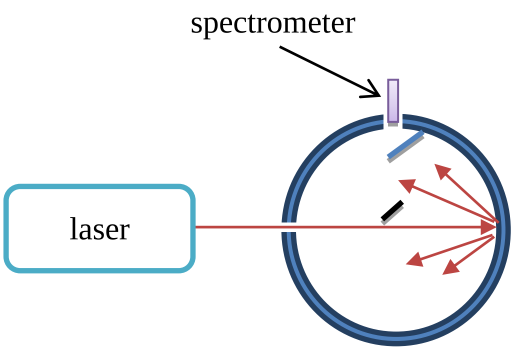
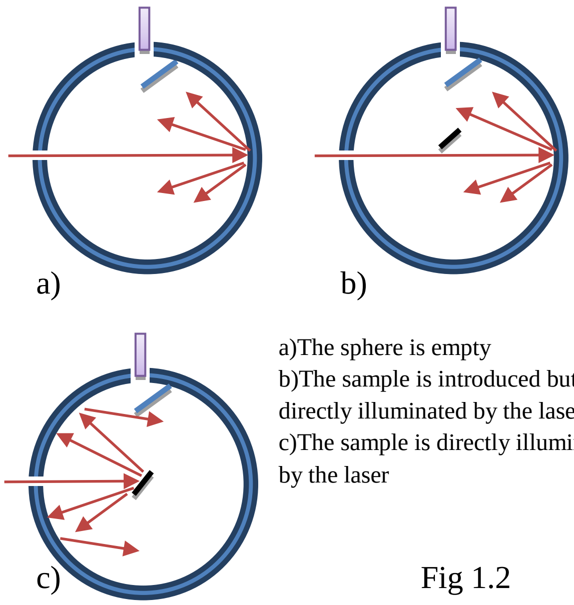


Fig1.1

The three experimental setups that are required are shown in fig1.2



a)The sphere is empty
b)The sample is introduced but not directly illuminated by the laser,
c)The sample is directly illuminated by the laser

Fig 1.2

The chosen excitation light source is a 435nM laser with its output attenuated to around 10mw. The Spectroscope used is a commercially available Ocean Optics USB2000+ with a P400-2-VIS-NIR patch cord fibre optic cable. The Integrating sphere is a custom design which allows close compatibility between the laser illumination, spectroscope and the samples under test.

The physical set up is shown below in fig 1.3.

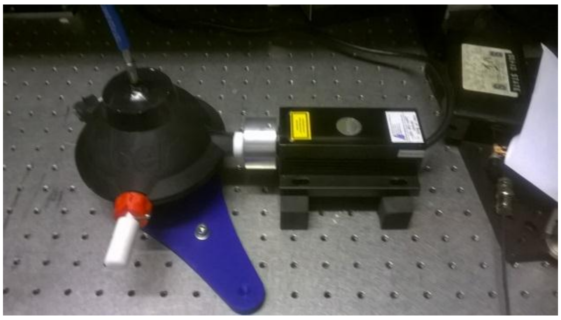
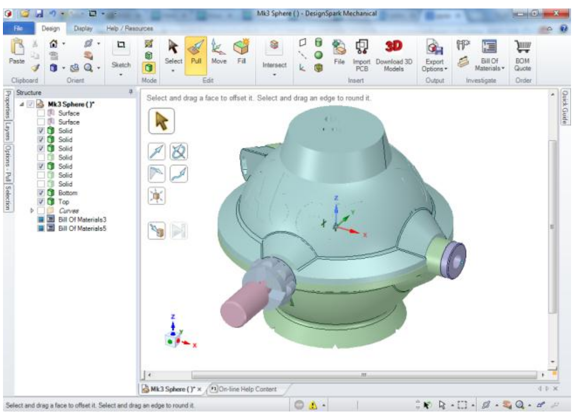


Fig1.3

2. Equipment Design

2.1 Integrating Sphere.

The initial Sphere design was solid modelled using Design Spark Mechanical. This was 3D printed ½ scale to prove the construction method. Following some modifications a fully functional Mk2 design was successfully built full scale and has been used to develop the experimental method and provide the initial results included here (fig 1.3). A Mk3 version which is currently under construction contains detail changes such as increased clearance in the light trap labyrinth to accommodate the high build thickness of the internal coatings and also includes some functional improvements and fool proofing.



Mk3 Model Image Fig2.1

2.2 Sample Holder

The sample holder has been designed and built. This utilises a simple external mechanism to easily position the sample in or out of the laser beam. It also allows for rapid changes between samples without disturbing the alignment of the experimental set up.

Fig 2.3

A clamp has been made which holds the relatively light weight sphere firmly to the optics bench, ensuring alignment is maintained during operation.

3. Initial Results.

3.1 Internal Coating Characterization

The spectral profile of the internal coating is of considerable importance. A range of 'homemade' and a single commercial product were compared to a known calibration reflectance standard (Ocean optics WS-1-SL). The homemade coatings proved disappointing due to the low purity (98%) of the barium sulphate used. A source of 99.98% purity has been found should further testing and development be considered beneficial. The result of the commercial product Avian-B is shown in fig 3.1 and this was selected to coat the Mk2 sphere used for the preliminary results. Importantly no unwanted fluorescence from the primer or undercoating materials was seen.

8°/Hemispherical Reflectance Data

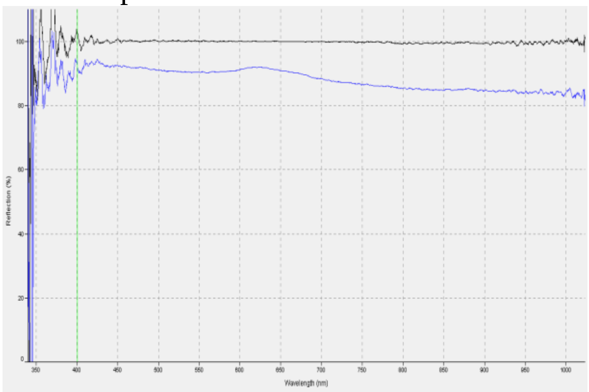


Fig 3.1

The Avian-B result was marginally lower than that expected from the coating supplier data sheets [2] and significantly shows a slight fall off in the range 650-800 nm. This will be an area of investigation prior to coating the Mk3 Sphere. If persistent it may require a correction factor to be applied at the data analysis stage.

3.2 Stray Light Testing

When fully assembled the integrating sphere was subject externally to a high intensity white light source. This was moved all over the external surface and in particular concentrated on the split line and entrance ports, all the while observing the internal spectroscopy looking for any increase above dark light noise levels. Some stray light was seen to be entering at the base of the spectroscope SMA coupling. This was blocked using black PVC tape and a change was made to the mounting area on the Mk3 model to resolve the issue in future.

3.3 First Samples

As proof of principal and to check the functionality of the design a Pyranine dye was applied to a small piece of paper and loaded into the sample holder. The experimental method was tested and found to be both quick and simple, producing results in line with those anticipated. (Fig 3.3)

3.4 Scilab Code

To utilise the data from the spectrometer a Scilab code has been written which reads the data output from Ocean optics SpectraSuite as a CSV file, plots the data graphically and integrates the areas under the separate curves between user defined limits. From the integrated area values the code calculates the quantum yield. The result for Pyranine is shown in fig 3.3.

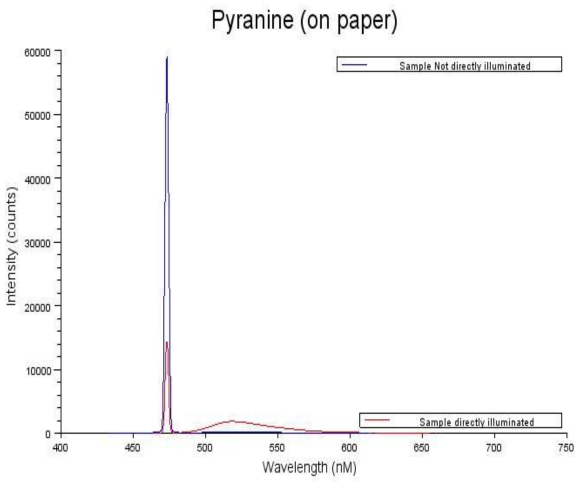


Fig 3.3

A quantum yield of 61.7% was calculated.

A second unknown fluorescent orange dye was also tested to ensure that the results were consistent. A screen shot of the SpectraSuite graph is shown in fig 3.4 which clearly shows the shift in output toward the red end of the spectrum and a reduced level of absorbed laser light.

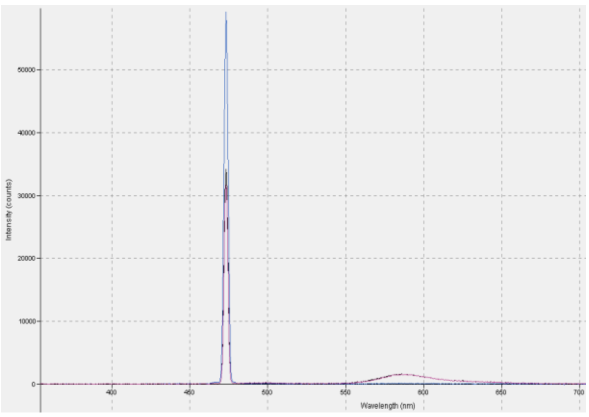


Fig 3.4

4. Next Steps

4.1 Calibration.

Obtaining relative PLQY values for an individual material has, at this stage, been proven to an initial degree of accuracy.
Further work will be done to improve the accuracy of data and an attempt to calibrate the equipment for absolute values will be made. To this end a NIST calibrated light source with its data sheet has been obtained.
Such a lamp can be placed at a precisely measured distance as described by Johnson et al [3] and its light is allowed to enter the sphere through a precision aperture of known area as shown in fig 4.1.

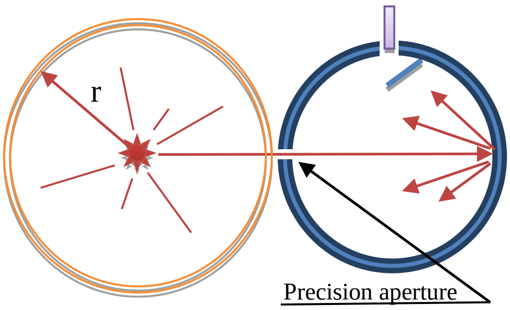


Fig4.1

This will allow a known quantity of flux into the sphere. For any known photon flux f (λ) entering the sphere a suitable calibration file can be made which will produce a correction to the data and result in a y axis scale in units of µwatts/nm giving in a profile which is correct in both shape and magnitude.
A precision aperture bushing is drawn and requires manufacture. It is proposed that a graded hardened steel ball which has an ISO standard diameter of 8 ± 0.0012mm is used to size the aperture by passing it through a soft ductile material such as aluminium.

Once further samples are obtained and calibration is complete it is thought progress to final results should be relatively straightforward.

Bibliography

[1] J.C. de Mello et al ***"An Improved Experimental Determination of External Photoluminescence Quantum Efficiency"*** Advanced Matter 9, 230 (1997)
[2] Avian Technologies Data Sheet "***Avian B White Reflectance Coating***" unpublished
[3] A.Johnson, S.Lee, J.Klein, J.Kanicki,. ***"Absolute Photoluminescence Quantum Efficiency Measurement of light-emitting Thin Films"*** Review of Scientific Instruments 78,096101 (2007)
[4]OceanOptics Technical Note:***"Absolute Irradiance Measurement"*** unpublished