Application News

No. UV-018

Spectrophotometric Analysis

Micro Sample Measurement with the Shimadzu UV-2600 and Standard Optical Microscope

C.M. Talbott; Sudhir Dahal; Shani Samuel

■ Introduction

In the past two decades, there have been significant advances in the miniaturization of electronic components utilized in industrial and consumer products. Now, for example it is possible to obtain a complete computer system packaged to be the size of a small cell phone. This drive for miniaturization has not only been focused on the electronic components but also on the other items that make up these advanced products. A prime example of this symbiotic miniaturization is the wide assortment of cameras now found on most cell phones and laptop computers. The small cameras not only have miniature electronics, but also contain very small lens systems to focus the incoming light onto the array. Lenses used in such systems can be very small in size measuring to just a few millimeters in diameter. The ability to accurately characterize the transmission and reflection of small optical components is critical to optimal function of these systems. The ability to characterize small optical components (5 mm minimum diameter in size) has been discussed in an earlier publication (Shimadzu Application News, No. UV-015, 2015).

There is a need, however, to optically characterize even smaller components and materials. The ability to reproducibly mount items that are less than 3 mm in diameter in the standard UV-Vis sample compartment can challenging. This paper describes the use of a Shimadzu UV-2600 UV-Vis Spectrophotometer coupled to a standard optical microscope via an optical fiber relay system to evaluate small filter samples approximately 1 – 2 mm in width.

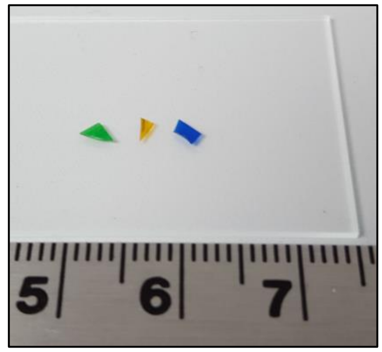
■ Discussion

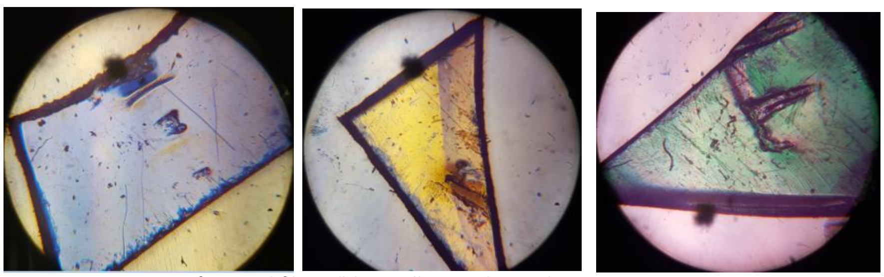
The Harrick FiberMate II along with two 100 μm Vis/NIR fibers (0.37 NA PCO4) were used to connect the Shimadzu UV-2600 Spectrophotometer to a standard optical microscope.

The biggest challenge in connecting a microscope to a spectrophotometer is the reduction in light throughput that occurs at each lens/mirror element. A Zeiss Universal microscope equipped with Epi Illumination was selected because of its ability to minimize lens elements in the light path. In addition, the epi illuminator can be used with Zeiss EpiPlan HD 4X 0.65 objectives, which have a reduced number of lens elements.

Fiber optic couplers were fabricated to connect the fibers to the microscope; one each for the source lamp input and one for the trinocular camera port. In addition, the lower tilt-in substage condenser element of the microscope was removed from the light path, as was the polarizer/analyzer, the blue tint filter, and the epi reflection assembly to eliminate reflection losses.

Small sections of optical filter material (red, yellow, and blue) were excised from larger filters to be approximately 1 – 2 mm at their greatest width. These small filter pieces were placed on a glass microscope slide for analysis. The Shimadzu UV-2600 was set up for medium scan speed between 375 nm and 875 nm, a 0.5 nm sampling pitch, and a 5 nm bandwidth. In addition, a wire mesh attenuator (Shimadzu PN 206-82299-92) was placed in the reference light path to compensate for the reduction in sample beam intensity by the fibers and microscope. Visible light at 575 nm was used to position and focus the samples. A background was acquired on the focused empty slide.

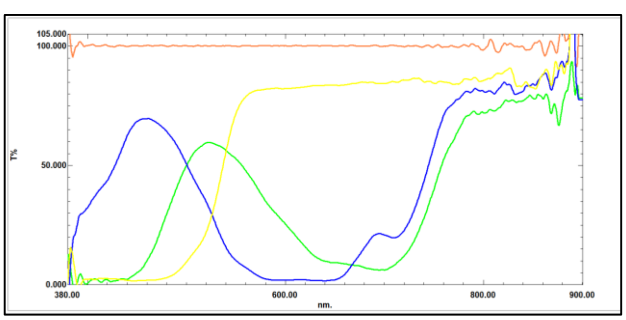




Microscope images of excised filters (blue, yellow and green)

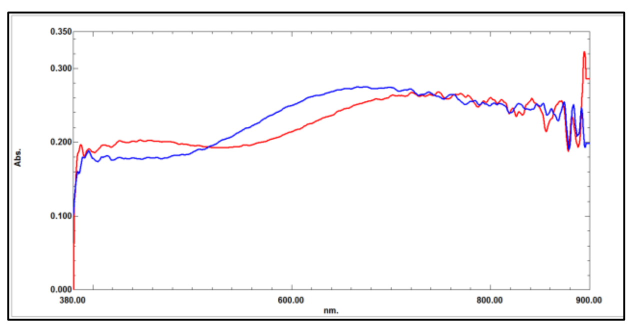
■ **Results**

A blank scan (empty slide) and scans of each filter material were acquired and are shown below. The blank scan shows low noise transmission between 380 nm and 840 nm. Below 380 nm, the glass elements in the microscope and microscope slide absorb and block throughput. Similarly, between 800 and 900 nm, the response profile of the PMT detector begins to decline and noise in the baseline begins to be apparent as the S/N is reduced. The acquired spectra for each film are also shown below. The transmission spectrum for each individual film is of high quality with good S/N. In addition, the individual film's transmission profiles are well characterized by the spectra.



Spectral scans of small filters using the Shimadzu UV-2600 Spectrophotometer fiber-connected to a standard bench optical microscope; small green filter (green), small blue filter (blue), and small yellow filter (yellow).

In a similar fashion, silicon nano-spheres (approximately 3 µm in diameter) were analyzed for their absorption spectra. The spheres were dispersed onto a glass microscope slide and spread very thin by leveling between two slides. The acquired absorption spectra of the two samples are shown below. The spectra show good S/N even with the low absorbance values. In addition, the spectra show differences between the two sphere samples.



Acquired spectra of silicon nano-spheres with different surface treatments.

■ Conclusion

The advanced Lo-Ra-Ligh gratings of the UV-2600 UV-Vis Spectrophotometer allow for the photometric range and low baseline noise required to perform accurate analysis using fiber optic systems. When the spectrophotometer is combined with a standard optical microscope, very small samples can be routinely, accurately, and successfully characterized.

First Edition: February 2016

SHIMADZU Corporation
www.shimadzu.com/an/

SHIMADZU SCIENTIFIC INSTRUMENTS
7102 Riverwood Drive, Columbia, MD 21046, USA
Phone: 800-477-1227/410-381-1227, Fax: 410-381-1222
URL: www.ssi.shimadzu.com