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Case Study of Absorption-Spectrum Transferability Relative to Substrates for Dyes in Fabrics

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14. ABSTRACT This study describes inverse modeling of absorption-spectrum transferability with respect to substrates for tailoring the NIR SWIR reflectance of dyed fabrics, which is important for optimizing reflectance of camouflaged fabrics. Inverse modeling is applied to reflectance spectra of a prototypical IR absorbing dye, which has been deposited upon different substrates, with different deposit-layer thicknesses. IR absorption features of any given IR absorbing dye will be a function of deposit-layer microstructure and dielectric response of substrate material. Therefore, one should examine the feasibility of parametrically modeling absorption-spectrum dependence on deposit micro-structure and dielectric response properties of substrates, which is relevant to simulating reflectance spectra of IR-absorbing-dye blends in fabrics, in particular, the feasibility of modeling transferability of dye-in-fabric absorption spectra, for given fabrics, to different types of fabrics, whose undyed reflectance spectra are known.						
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Introduction

In the past, night vision devices used for detection and identification of personnel targets on the battlefield have primarily utilized the visual (Vis, 380-700 nm), near-infrared (NIR, 700-1000 nm), midwave-infrared (MWIR, 3-5 μm), and longwave-infrared (LWIR, 8-12 μm) portions of the electromagnetic spectrum. Night vision devices such as the Gen 2 and Gen 3 goggles pose a greater problem to U.S. ground forces due to their affordability, availability and compact size. These devices are sensitive in the 350-1000 nm wavelength range. Naturally, the recent design of U.S. military combat uniforms has focused on mission-specific camouflage treatments in this spectral region. One such treatment embodies various techniques for dyeing, printing, and embedding NIR reflective pigments in military fabrics. Experiments with NIR absorbing dyes in fabric have demonstrated the ability to adjust the NIR reflectance of the material to match the spectral response of a specified environment. However, certain dyes and dye concentrations have a tendency to darken the fabric, changing the reflectance characteristics in the visible spectrum. Another problem concerns the fact that NIR dye treatments do not account for the short-wave infrared (SWIR, 0.9 -1.7 μm) portion of the spectrum.

Advances in detector technologies, e.g., Indium Gallium Arsenide, have allowed manufacturers to produce SWIR cameras with enhanced sensitivity and resolution in a smaller package at an affordable cost. This added versatility has increased the likelihood of SWIR devices proliferating across the battlefield in the near future. The addition of a new detection threat creates a multispectral optimization problem for camouflage designers. Without knowing the specific threat or threat combination employed by an enemy, a given camouflage treatment must protect against all known threat detection systems.

There exists a need to develop and apply short-wave infrared signature-reduction treatments to existing and future military uniforms while maintaining acceptable visible and near infrared signatures. One possible solution entails the development of a parameter space for process optimization of dyed fabrics. Given the fabric and absorbing dyes available, an optimization algorithm will predict the optimal dyeing process and fabric/dye combinations needed to match the background spectral response of a specified environment across the visual, near-infrared, and shortwave infrared regions of the spectrum. To reach the point of process optimization, different absorbing dyes, military fabrics, and parametric reflectance models must first be examined.

This study investigates inverse modeling of a specific property of IR absorbing dyes, absorption-spectrum transferability with respect to substrates for predicting NIR-SWIR reflectance of dyed fabrics, for optimizing reflectance of camouflaged fabrics. Inverse modeling is applied to reflectance spectra of a prototypical IR absorbing dye, which has been deposited upon different substrates, with different deposit-layer thicknesses. In that IR absorption features of any given IR absorbing dye will be a function of deposit-layer microstructure and dielectric response of substrate material, the feasibility of parametrically modeling absorption-spectrum dependence on deposit microstructure and substrate properties is examined. In particular, the feasibility of modeling transferability of dye-in-fabric absorption spectra to different types of fabrics, whose undyed reflectance spectra are known. The dye and fabric considered for prototype analysis are tetraaryldiamine and a camouflage textile (NYCO Ripstop) characterized by color variations, respectively. The results of this study provide validation of the inverse model, within reasonable error tolerances for practical applications, including NIR spectral characteristics in camouflage textiles, for purposes of simulating NIR spectra corresponding to various dye concentrations in host fabrics.

Inverse modeling described here applies a model of diffuse reflectance for the purpose of simulating the spectral response of military textiles containing different types of IR absorbing

dyes. This analysis lays the ground work for a prediction tool, which for given constituent materials available, i.e., dyes and fabrics, will provide for design of optimal composite systems, i.e. military combat uniforms allowing individuals to match the environmental spectral response in the visual, near-infrared and shortwave-infrared spectral bands. Theory underlying inverse modeling applied here, as well as the dye and fabric chosen for prototype analysis, are described subsequently. Formulations of the model is based on the inverse analysis approach [1,2,3,4,5], which can be based either on phenomenological or semi-phenomenological model representation, formulated for parameter adjustment with respect to specified target measurements (i.e., measurements to be modeled), as well as inclusion of material properties obtained by other measurements.

Optimizing reflectance spectra of dyed fabrics with respect to given specifications poses the problem of optimizing diffuse reflectance materials. Diffuse reflection defines a separate regime for spectroscopic analysis of material surfaces and layers. The physical processes underlying diffuse reflectance are discussed extensively in references [6,7,8,9,10,11]. Spectral analyses using parametric models of diffuse reflectance have been applied successfully for identification and characterization of soil minerals and paper containing ink. Parametric models applied for these analyses include those based on the Kubelka-Munk theory of diffuse reflectance (and formulations derived from it) [12,13,14,15], and representations based on the Beer-Lambert Law) [8]. The parametric models of diffuse reflectance presented in this study, which is for analysis and characterization of dyed fabrics, is based on reflectance-background subtraction and the Beer-Lambert Law for extinction [16,17] combines with absorption coefficients obtained by inversion of transmission spectra for dyes in solution [18].

Underlying conditions for validity of the inverse model considered in this study, designated is transferability of background-subtracted spectra with respect to different substrates. Strictly, the absorption coefficients obtained by inverse analysis of transmission spectra are to be interpreted as estimates of the dielectric response of individual dye molecules. This is supported by studies of solvent effects on absorption spectra, which have shown that spectra of dissolved dyes depend on the nature of dye-solvent interactions. In particular, that wavelengths for maximum absorption, spectral shifts, and other spectral features depend on dye-solvent interactions [19,20,21]. These permittivity functions, however, should be considered as “reasonable” estimates of dielectric response. This follows from the general dependence of absorption spectra on dye-solvent interactions, which tends to preserve dominant spectral features, regardless of changes in intensity maxima, wavelengths for maximum absorption, and shape of absorption band (see [21] for example). Following the inverse modeling approach, by inclusion of adjustable scaling factors, e.g., a_s defined below, a parametric model can be structured for statistical variability with respect to extinction processes associated with dyed fabrics, which includes those depending on interactions of dye molecules with those comprising a fabric, as well as heterogeneous dye-molecule distributions within a fabric. Accordingly, the statistical nature of the parametric model, requires only reasonable estimates of dye absorption coefficients that preserve dominant spectral features. That is to say, with respect to the parametric model, absorption coefficients assume the role of deterministic constraints, rather than specified material properties.

Transferability with respect to substrates follows in that the spectra of individual molecules, although perturbed by being embedded within a host bulk environment, still retain their signature identity to some level of estimation. Specifically, molecules retain their spectral identity within different environments, but will have their properties affected in accordance with the strength of the interactions experienced. The particular properties of interest here are IR spectra of molecules. Of the ordinary three phases of matter, the strength of interactions with the environment will increase in changing from gas to liquid to solid. In any of these environments the interaction affects will grow with increasing pressure, which brings molecules into closer interaction. As regards the

effect of increasing temperature, within a fixed phase, higher temperature will affect the occupation of vibration levels, and enhance a tendency towards a change of harmonic levels to anharmonic levels. The important point to be emphasized, however, is that molecules retain their IR spectral properties through the veil of all such changes.

Materials and Methods

Dyes. This study examines the transferability of reflectance characteristics for tetraaryldiamine dye (see [22]), which is considered prototypical of IR absorbing dyes, with respect to substrates (see Figure 1A).

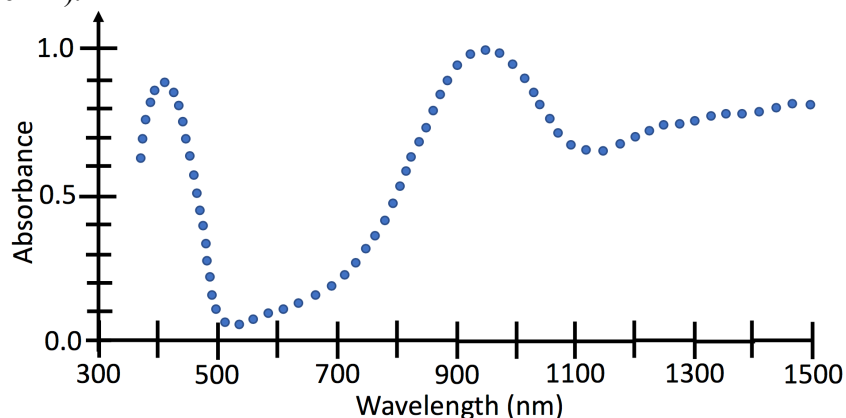


Figure 1A. Absorbance spectrum of prototype IR absorbing dye.

Fabric. The base fabric considered for dye application was a 50% Nylon, 50% Cotton blend Ripstop fabric, i.e. 50/50 NYCO Ripstop. Ripstop fabrics are woven fabrics with reinforcement threads interwoven at regular intervals in a crosshatch pattern (see [23]). The fabric was characterized as having a rough surface, finite and non-uniform thickness, heterogeneous composition, and a four color, AOR1 desert camouflage pattern design (see Figure 1B).

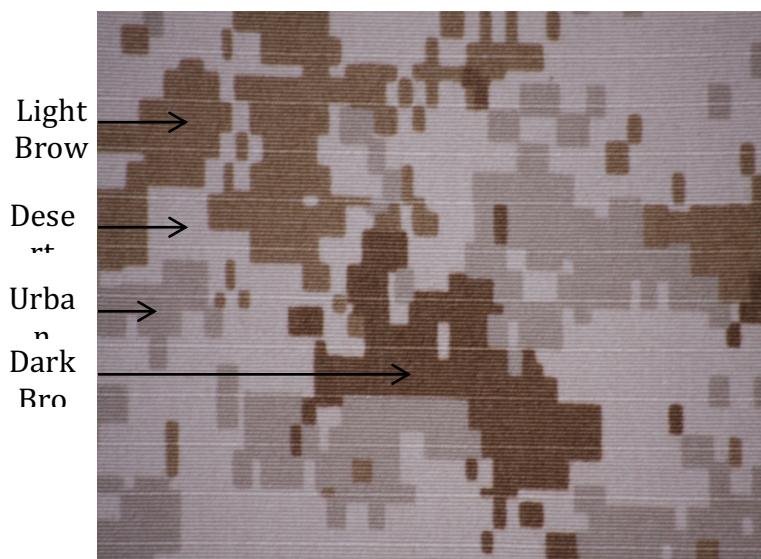


Figure 1B. Macroscopic sample of base fabric considered for dye application, 50% Nylon, 50% Cotton blend Ripstop fabric, i.e. 50/50 NYCO Ripstop.

Dyed-Fabric Sample Preparation. For the prototype dye and corresponding solvent, a stock solution of 30 g L⁻¹ was made. Serial dilutions of the stock solution was prepared, consisting of 3.0, 1.5, 0.3, 0.15 and 0.03 g L⁻¹. One square yard of 50/50 NYCO Ripstop fabric was cut from the roll and placed under a Freeman Schwabe cutting press. A 2"x2" Fremont steel rule cutting die was used in combination with the mechanical press to make 2"x2" fabric samples. Dye-fabric samples were prepared by pipetting 1 mL of each dye solution onto the center of each 2"x2" fabric piece. The pipetting speed was adjusted as to avoid pooling of the dye solution on the fabric surface. Dye-fabric samples were given 24 hours to dry before total reflectance measurements were made.

Dye-on-Aluminum Sample Preparation. Dye-on-Al samples were prepared by pipetting 1 mL of each dye solution onto the centers of 2" x 2" x 0.126" Aluminum coupons. Again, the pipetting speed was adjusted as to avoid pooling of the dye solution on the metal surfaces. Dye-on-Al samples were given 24 hours to dry before total reflectance measurements were made.

Spectral Measurements of Dyed-Fabric and Dye-on-Al Samples. Measurements of total reflectance were made using a UV/Vis/NIR/SWIR spectrophotometer (Perkin-Elmer® Lambda™ 1050, Waltham, MA, USA) in combination with UV WinLab (V6.0.3, Perkin Elmer) software. Reflectance was measured from 250 nm to 2500 nm at 5 nm stepping increments. All measurements were 8 degree hemispherical in total reflectance collection mode. The Lambda 1050 incorporated a double beam, 150 mm integrating sphere housing a photomultiplier tube (PMT) detector for the UV-Vis (175 – 860 nm) region and an Indium Gallium Arsenide (InGaAs) detector for the NIR-SWIR (860 - 2500 nm) region. Radiation sources included a deuterium lamp for operation in the UV (175 – 319 nm) and a tungsten halogen lamp for use in the Vis-NIR-SWIR (319 – 3000 nm). A light source change at 319.2 nm and a detector change at 860.8 nm occurred automatically during monochromator slewing. For UV-Vis wavelengths, the slit width was fixed at 4 nm. For NIR-SWIR wavelengths, the slit width was set to "Servo" mode, which automatically adjusted the slit width during scanning to maintain constant energy at the detector.

Inverse Analysis of IR Reflectance Spectra for Dyed Fabrics

Parametric models provide a means for inverse analysis of reflectance characteristics of a given system consisting of a given material, or combination of materials. These models can be defined in terms of adjustable parameters, dielectric properties (e.g., optical constants), and measured spectra (reflectance and transmission) [24,25,26]. For reflection from a dyed fabric of finite and nonuniform thickness, whose material composition is heterogeneous, it is plausible that parametric modeling of diffuse-reflectance spectra can be based on the Beer-Lambert law for extinction, (i.e., attenuation of of radiant flux traversing a medium by scattering and absorption [16]) and reflectance-background subtraction. A parametric model for inverse spectral analysis, combining the Beer-Lambert law and reflectance-background subtraction, is given by

$$R_{d+s}^{(p)}(\lambda) = R_s(\lambda) \exp\left[-\alpha_s A_d^{(r)}(\lambda)\right] \quad (1)$$

$$A_d^{(r)}(\lambda) = -\ln \left[\frac{R_{d+s}^{(r)}(\lambda)}{R_s^{(r)}(\lambda)} \right] \quad (2)$$

$$Z_R = \sum_{n=1}^N w_n \left(R_{d+s}^{(p)}(\lambda_n) - R_m(\lambda_n) \right)^2 \quad (3)$$

where $R_{d+s}^{(p)}$ is the predicted reflectance for dye on substrate, R_s measured reflectance of substrate of interest, and a_s is an adjustable scaling factor. $A_d^{(r)}$ is the reference absorbance determined by inverse analysis using defined by Eq. (2), and is an estimate of dye absorbance to within an adjustable scale factor. $R_{d+s}^{(r)}$ and $R_s^{(r)}$ are measured reference spectra, which are reflectances for substrates with and without deposited dye, for a given dye deposited dye concentration. The use of (λ) indicates quantities that are wavelength dependent. The formal procedure underlying inverse analysis based on Eqs.(1)-(3) entails adjustment of the scaling factor a_s of the reference absorbance $A_d^{(r)}$, (determined using Eq. (2)). This approach defines an optimization procedure where the reflectivity spanning the range of wavelengths is adopted as the quantity to be optimized. Constraint conditions are imposed on the reflectivity by minimizing the objective function defined by Eq. (3), $R_m(\lambda_n)$ is the measured or target reflectivity for wavelength λ_n . The quantities w_n ($n=1, \dots, N$) are weight coefficients that specify relative levels of influence associated with constraint conditions $R_m(\lambda_n)$.

The parametric model defined by Eqs. (1) and (2) adopts a reference absorbance spectrum determined using reflectance-spectrum background subtraction for modeling reflectivity spectra of dyes in fabric. Equation (2) follows from the assumption that the contribution to the reflectivity due to the dye can be factored out from that of the combined system of dye and fabric. This relation follows from the Beer-Lambert law and formalism of the scattering-matrix [16]. Validation of the parametric model is that, for the dye-fabric systems considered, there is agreement between predicted and measured reflectance spectra for a dye in fabric, i.e., sufficient transferability of spectra.

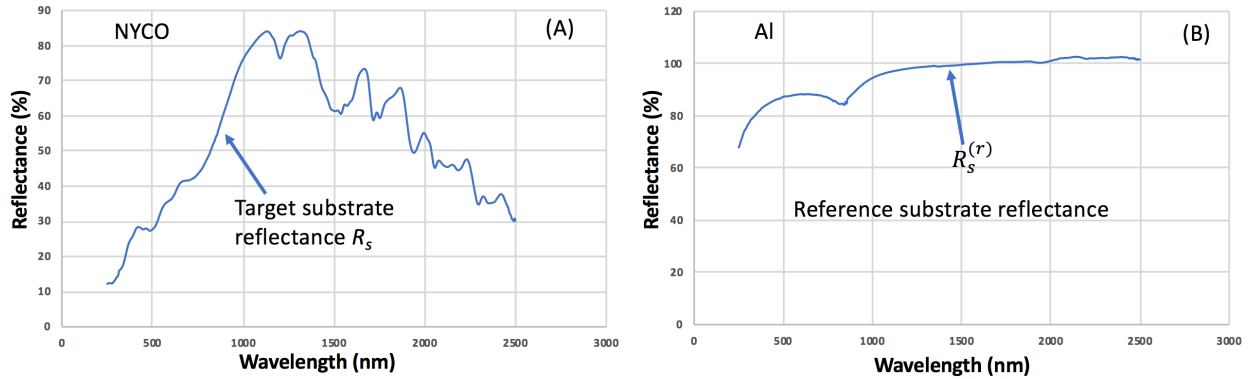


Figure 2. Reflectance spectra of (A) target and (B) reference substrates, NYCO Ripstop and Aluminum, respectively.

Validation of Absorption-Spectrum Transferability for Modeling Dyed-Fabric Reflectance

In this section modeling of diffuse reflectance spectra for a dyed fabric is presented, providing model validation and demonstrating modeling of reflectance spectra based on absorption-spectrum transferability. The parametric model defined by Eqs. (1) and (2), whose formulation is based on the Beer-Lambert Law and background-subtracted reflectance, was examined for feasibility of

characterizing the diffuse reflectance of dyed fabrics. Consistent with the Beer-Lambert law, diffusely reflected radiation is related to changes in scattering, absorption, and thickness of a dyed fabric layer. The layer is assumed infinitely thick, leaving reflectance in terms of the relative proportions between absorption and scattering. With respect to the Beer-Lambert law, due to the complex nature of scattering, and the uncertainty with respect to dyed fabric microstructure, the combined influence of absorption and scattering are represented by the absorbance function $A_d^{(r)}$ to within a scale factor. Accordingly, the function $A_d^{(r)}$ defined by Eq.(2) represents the combined influence of absorption and all other underlying physical processes contributing to reflectance within the dyed fabric layer.

A case study is presented for modeling reflectance spectra using the inverse model Eqs. (1) and (2). Shown in Fig. 2 are reflectance spectra for NYCO fabric (see Fig. 1) and aluminum, which are adopted as target and reference substrates, respectively, and quantities R_s and $R_s^{(r)}$ defined by Eqs. (1) and (2), respectively. Shown in Figs. 3 and 4 are reflectance spectra $R_{d+s}^{(r)}$ for different concentrations of dye deposited on aluminum, and their background normalization $R_{d+s}^{(r)}/R_s^{(r)}$ with respect to reflectance from Al, respectively. Shown in Fig. 5 are comparisons of measured and modeled reflectance spectra for different concentrations of dye deposited on NYCO fabric. This comparison of measured and modeled spectra demonstrate reasonable transferability of spectra with respect to substrates Al and NYCO fabric. The scaling factor a_s defined with respect to the inverse model defined by Eqs. (1) and (2), is an adjustable parameter accounting for average dye concentration, statistical variations in fabric thickness, dye distribution in fabric, and variations in concentration. In that the concentration of deposited dye was the same for both target and reference spectra, the values of a_s for all modeled spectra shown in Fig. 5 was 1.

The inverse model defined by Eqs. (1) and (2) suggests a metric for transferability based on a similarity function f_s defined by the relation

$$\frac{R_{d+s1}^{(r)}(\lambda)}{R_{s1}^{(r)}(\lambda)} = f_s(\lambda) \frac{R_{d+s2}^{(r)}(\lambda)}{R_{s2}^{(r)}(\lambda)}, \quad (4)$$

where $s1$ and $s2$ designate different substrates. A qualitative analysis based on Eq.(4) follows by comparison of normalised (background subtracted) spectra shown in Figs. 4 and 6, for different concentrations of dye deposited on aluminum and NYCO, respectively

In general, application of the inverse model defined by Eqs. (1) and (2) must consider the inherent statistical nature of diffuse reflectance from a dyed fabric. In particular, agreement between modeled and measured spectra should not be interpreted in the same sense as, for example, thin-film reflectance spectroscopy, where interegrated samples are characterized by smooth surface (and thus specular reflection), homogeneous composition, and well-defined thicknesses. In contrast, dyed fabrics are characterized, inherently, by rough surfaces (thus diffuse reflectance), heterogeneous compositions, and nonuniform thicknesses. It follows from the inherent statistical nature of reflection from a dyed fabric that some criterion for relaxed parameter optimization should be applied for modeling spectra.

Conclusion

This study examines the diffuse reflectance characteristics of a dyed fabric with respect to transferability of reflectance spectra relative to different substrates using parametric models. The results show good agreement between modeled and experimental reflectance for a prototypical

NIR absorbing dye in military fabric as a function of concentration and wavelength. The goodness of fits between modeled and experimental reflectance provide validation of the parametric models, which are based on the concept of spectrum transferability relative to substrate, for simulating diffuse reflectance of dyed fabrics. These results should contribute to a parametric model space for process optimization of dyed fabrics for SWIR applications.

Acknowledgement

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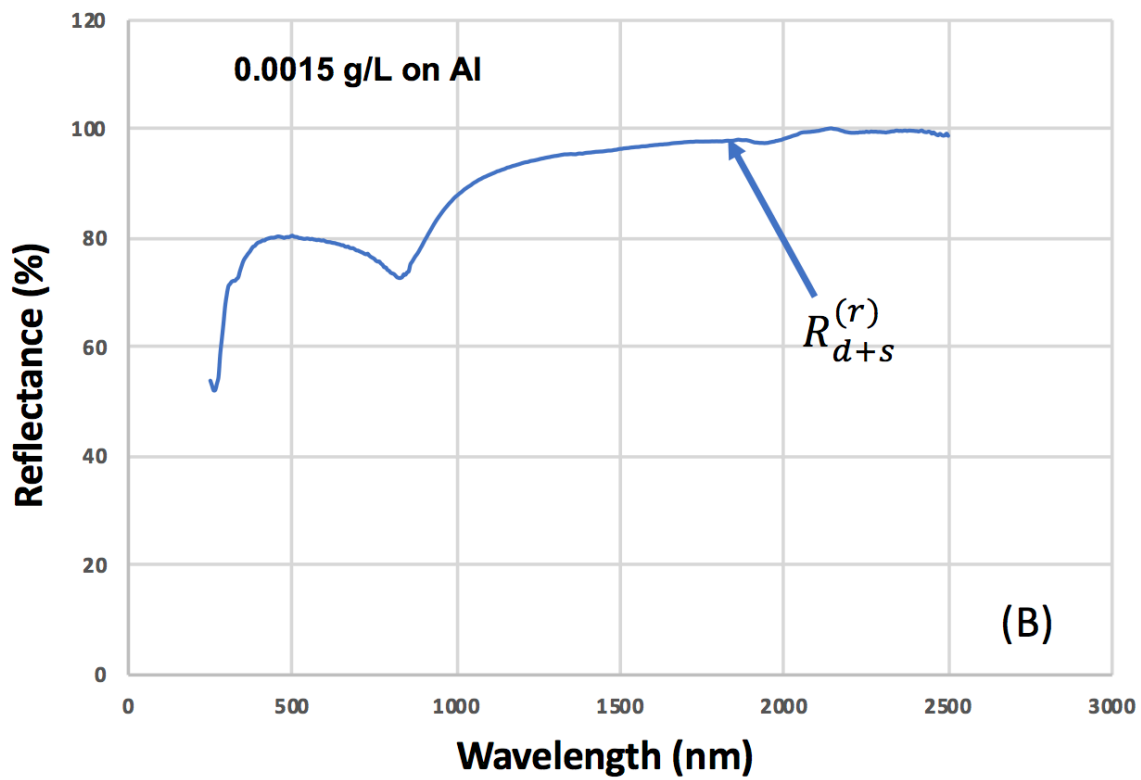
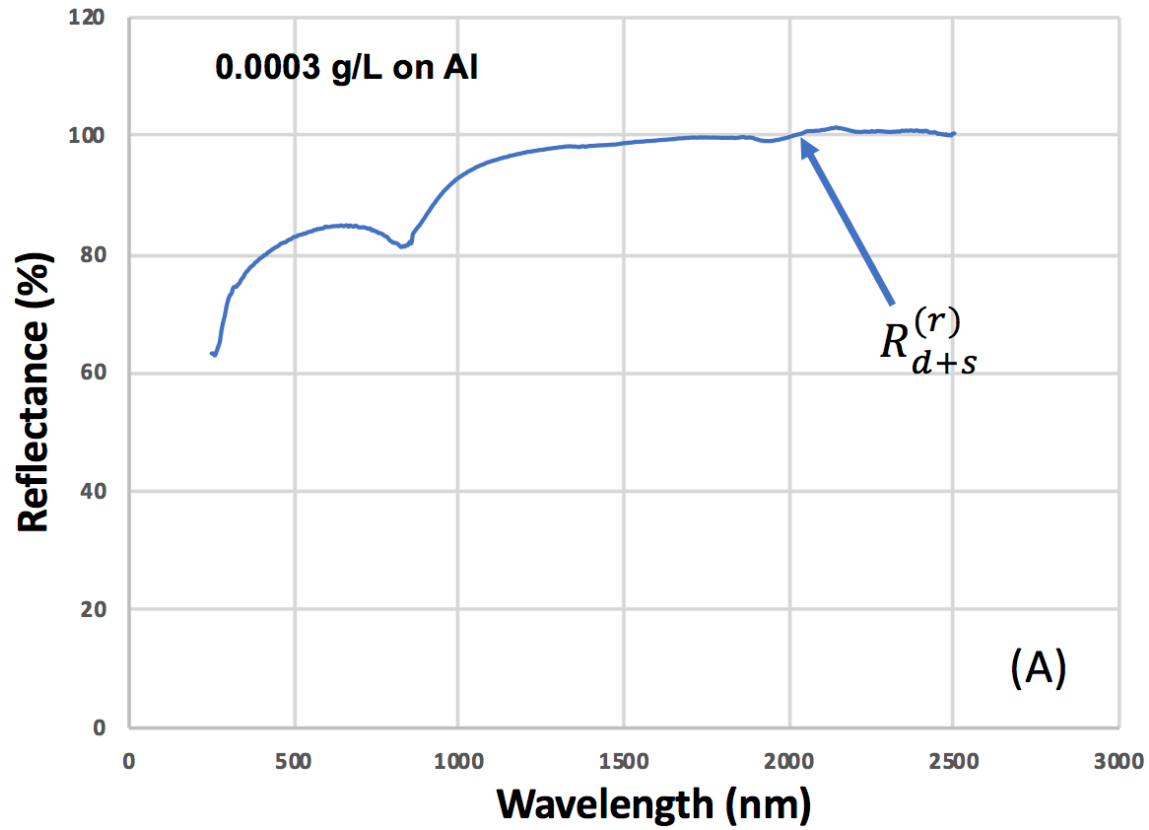


Figure 3 (A) and (B). Reference reflectance spectra for dye on Aluminum.

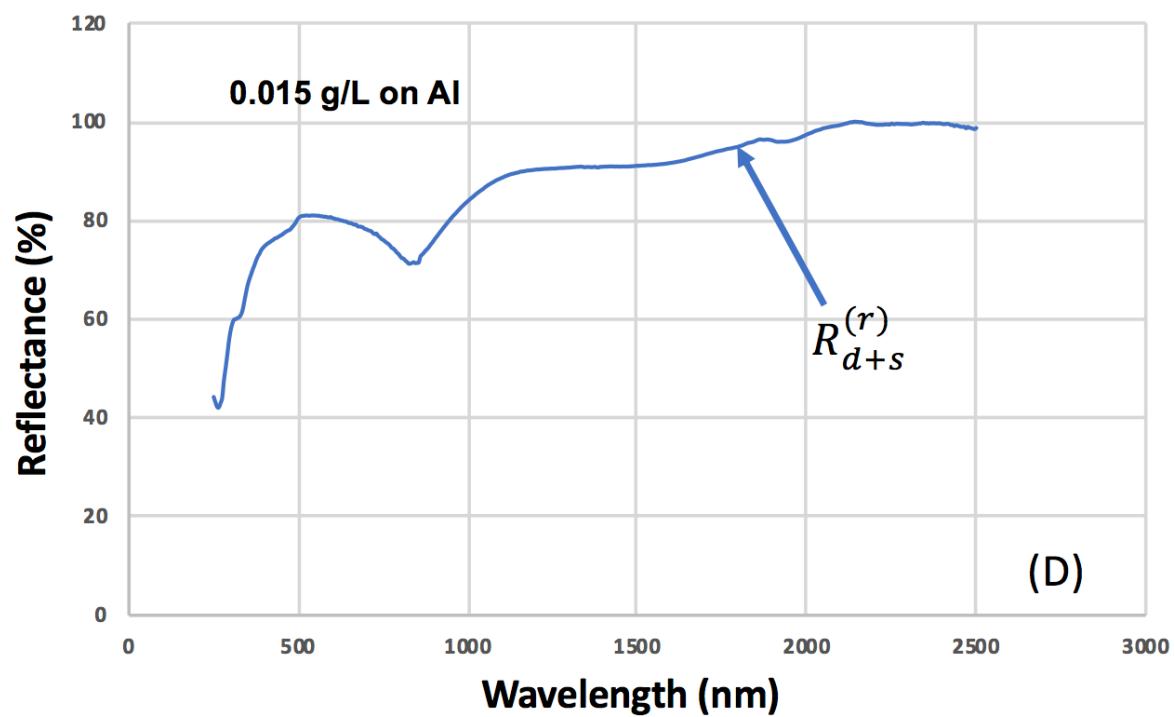
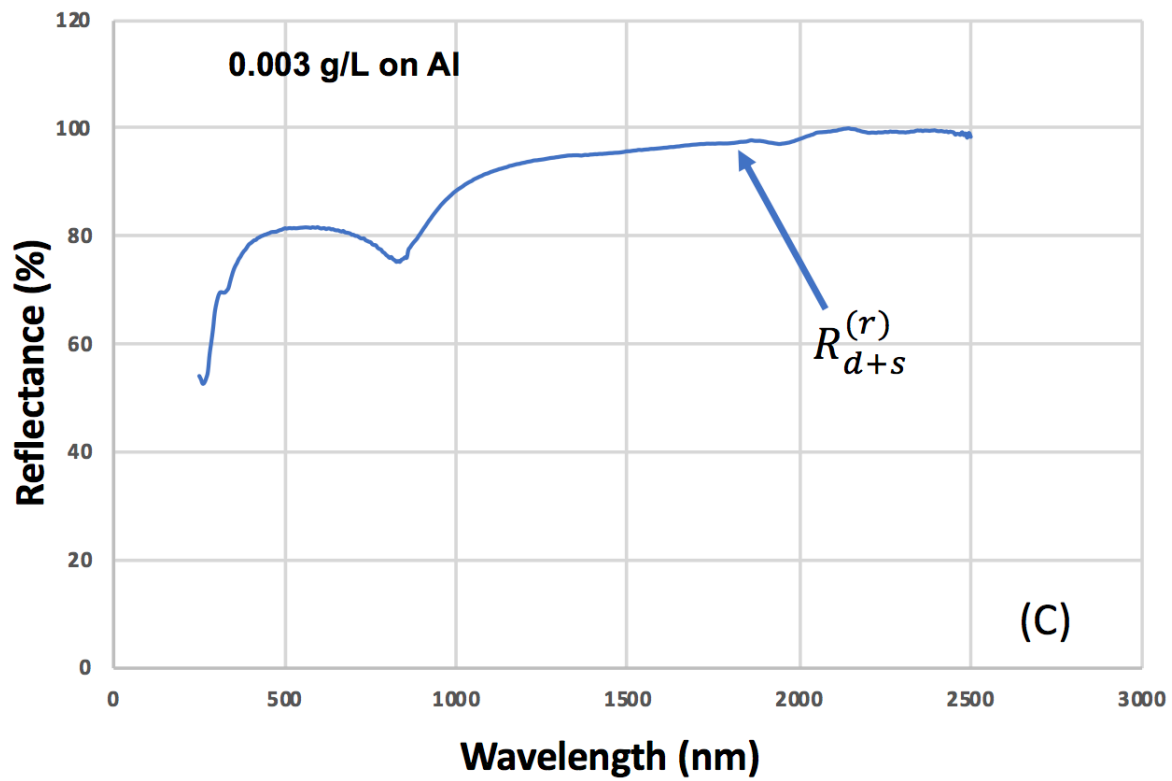


Figure 3 (C) and (D). Reference reflectance spectra for dye on Aluminum.

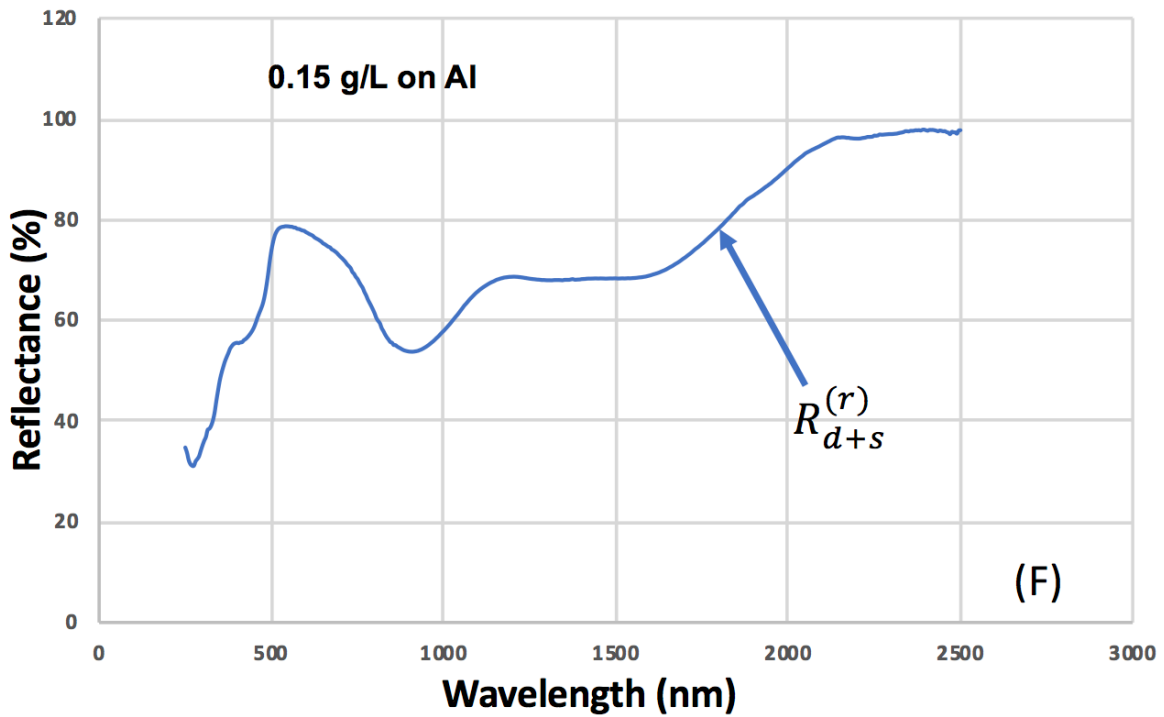
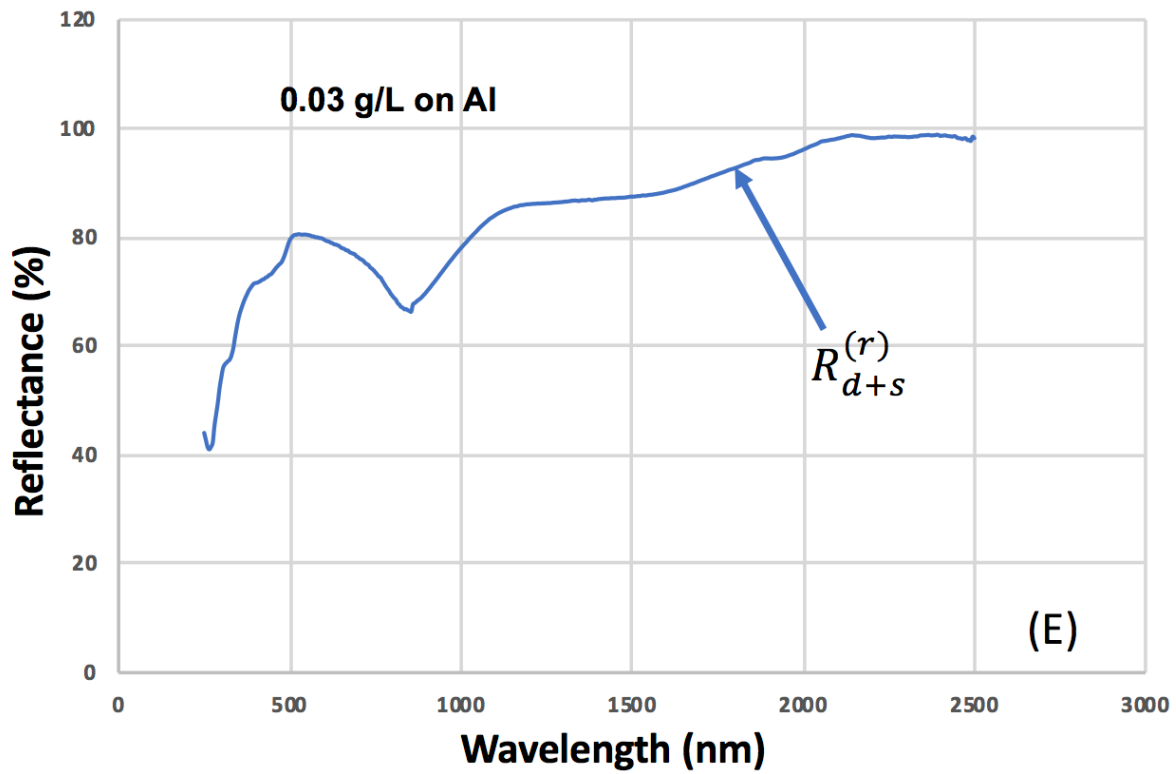


Figure 3 (E) and (F). Reference reflectance spectra for dye on Aluminum.

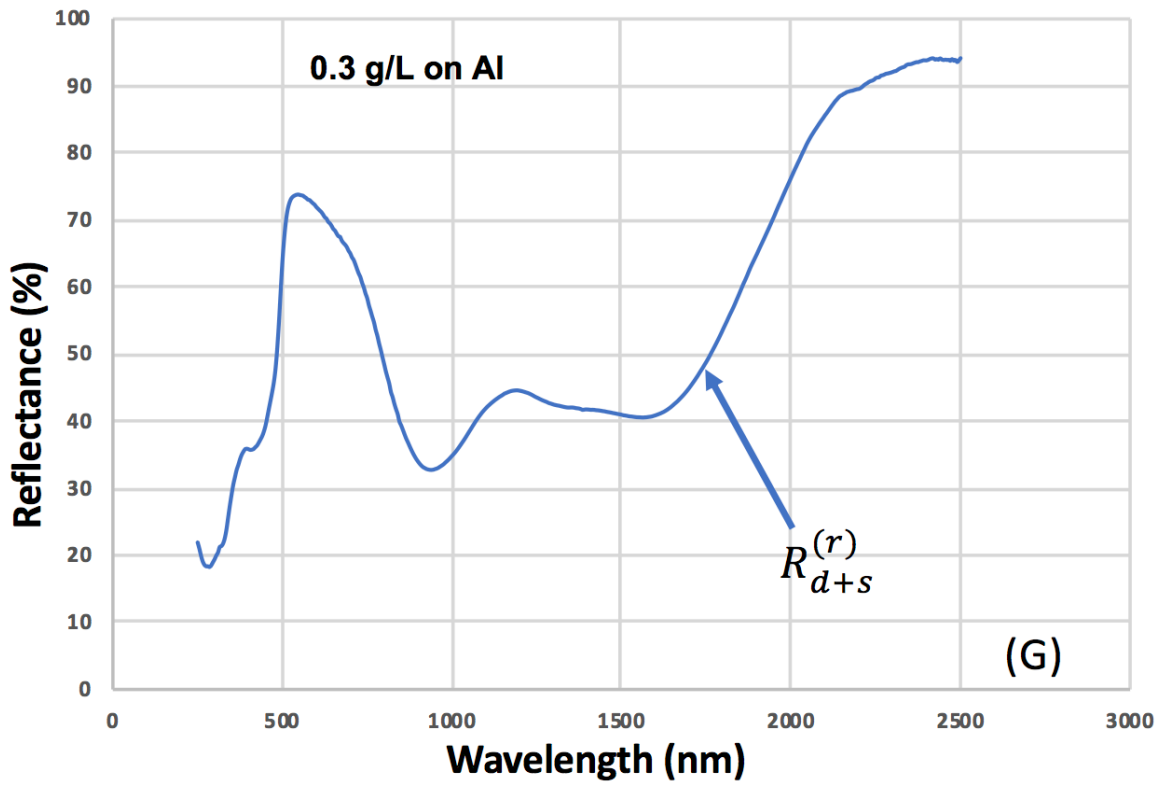


Figure 3 (G). Reference reflectance spectra for dye on Aluminum.

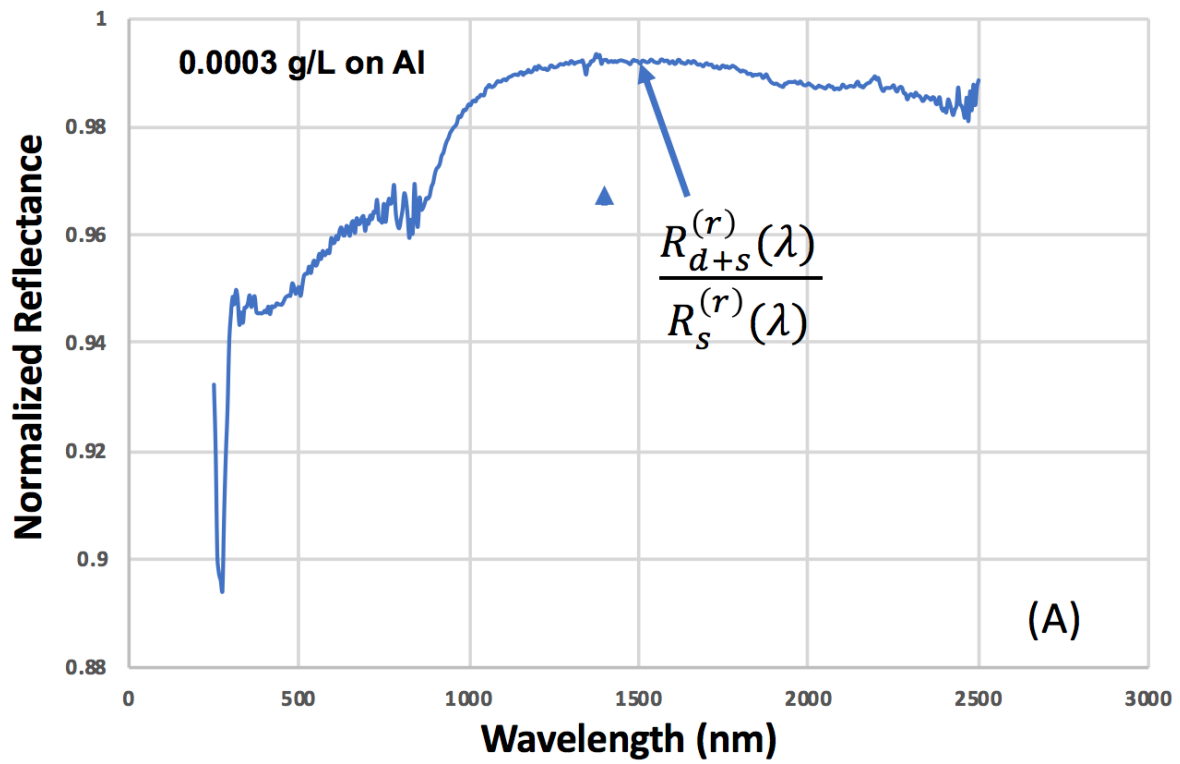


Figure 4 (A). Background subtracted reference spectra for dye on Aluminum.

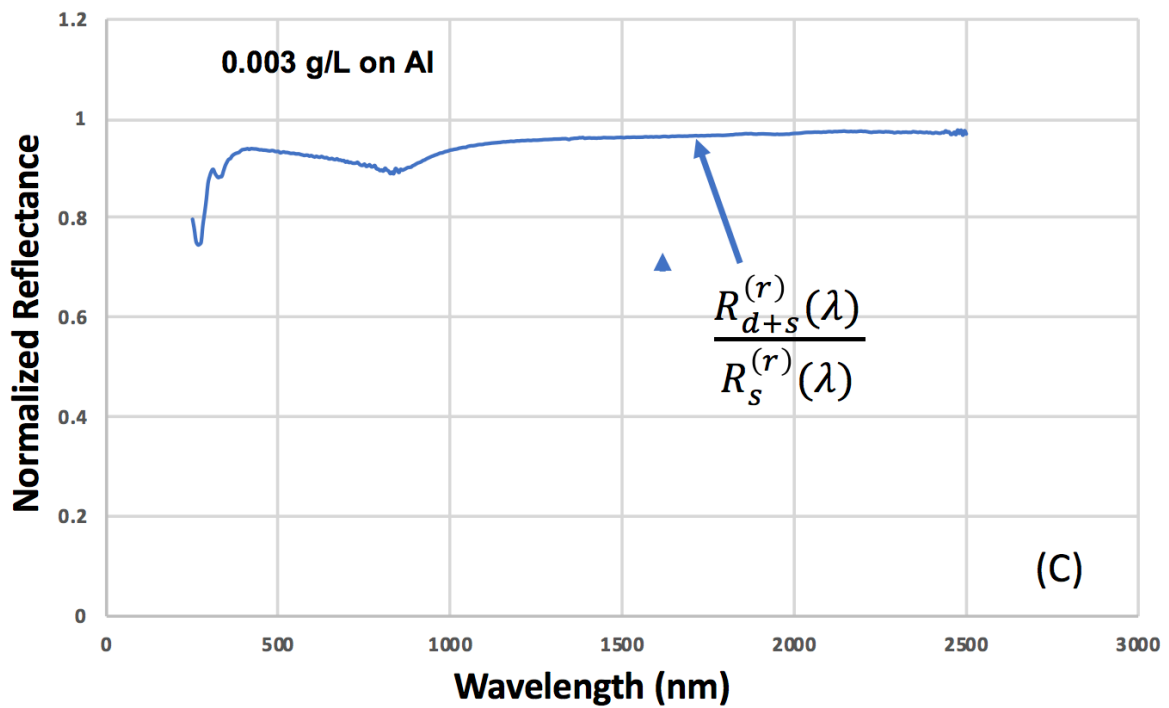
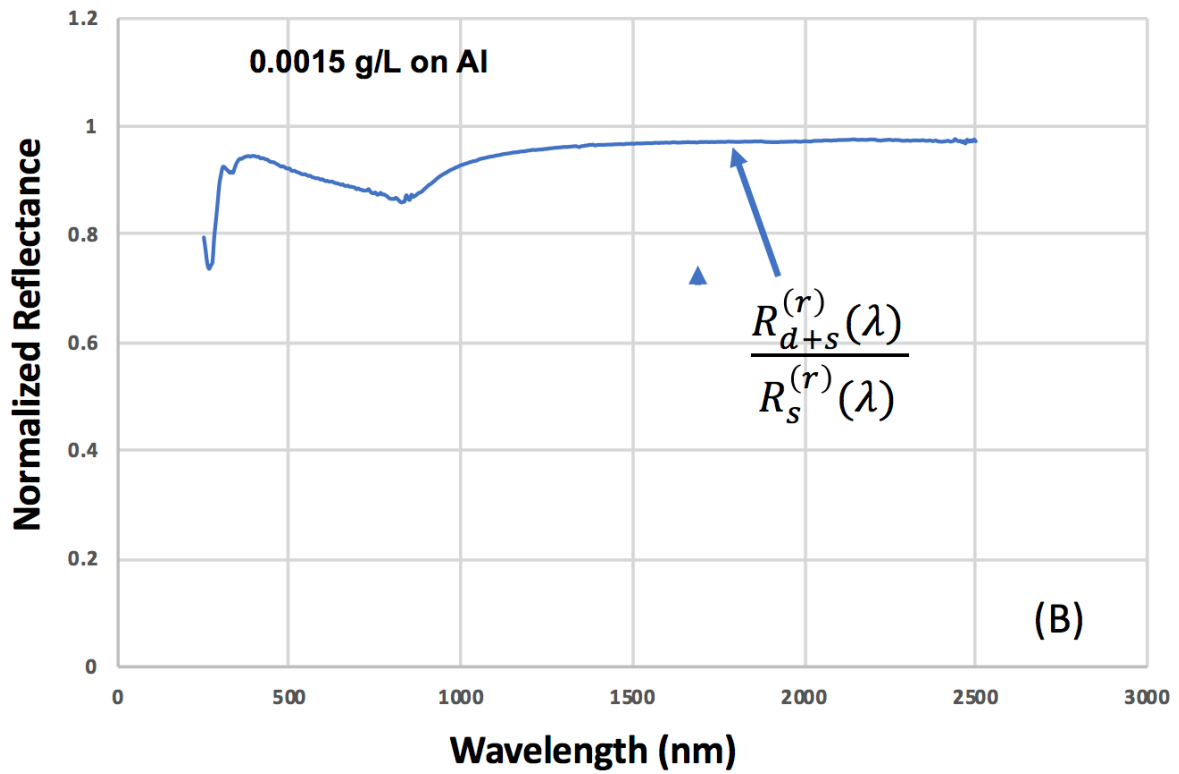


Figure 4 (B) and (C). Background subtracted reference spectra for dye on Aluminum.

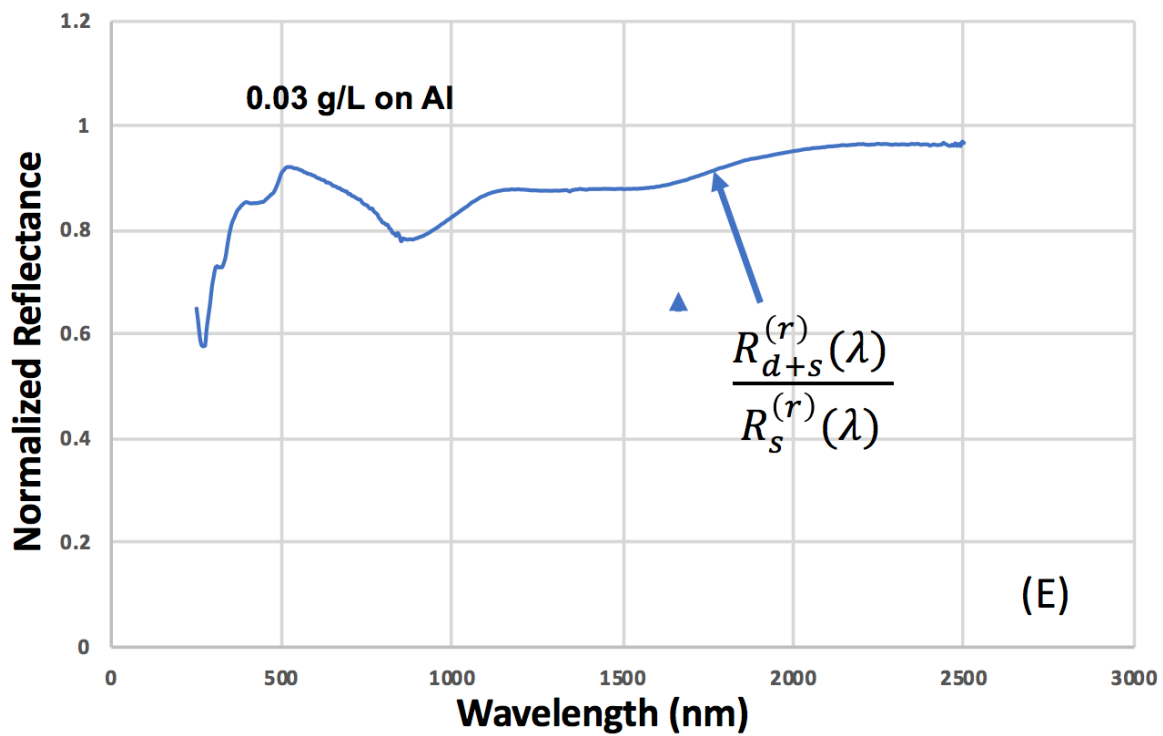
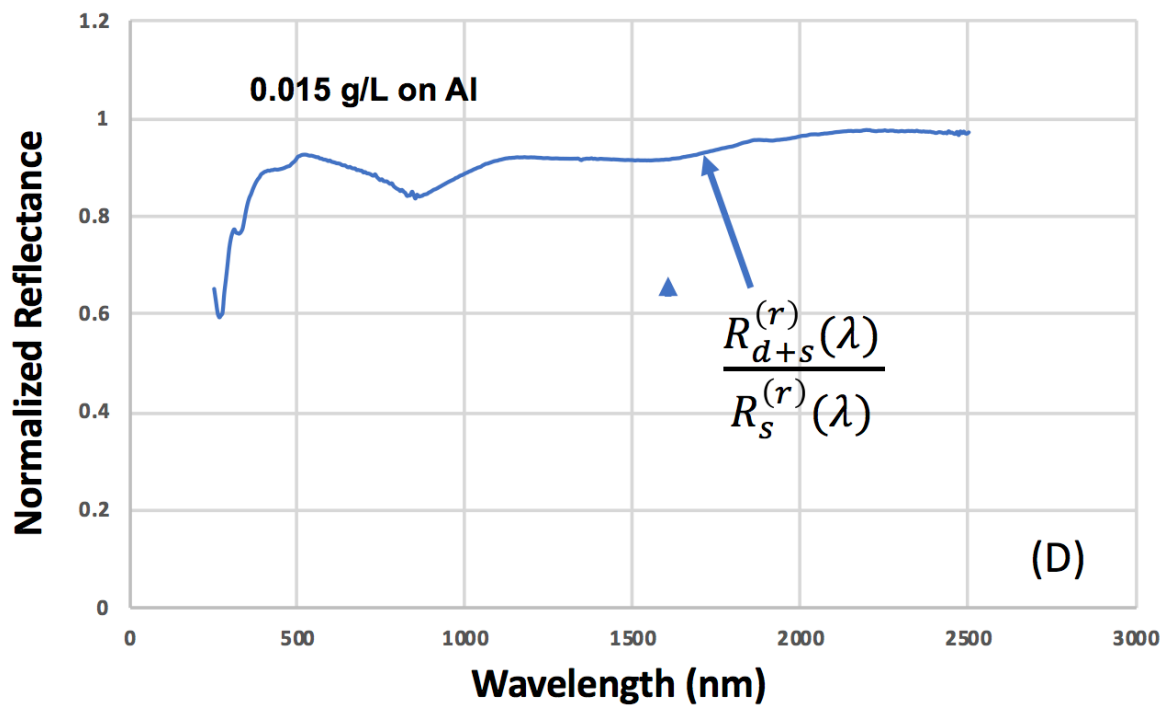


Figure 4 (D) and (E). Background subtracted reference spectra for dye on Aluminum.

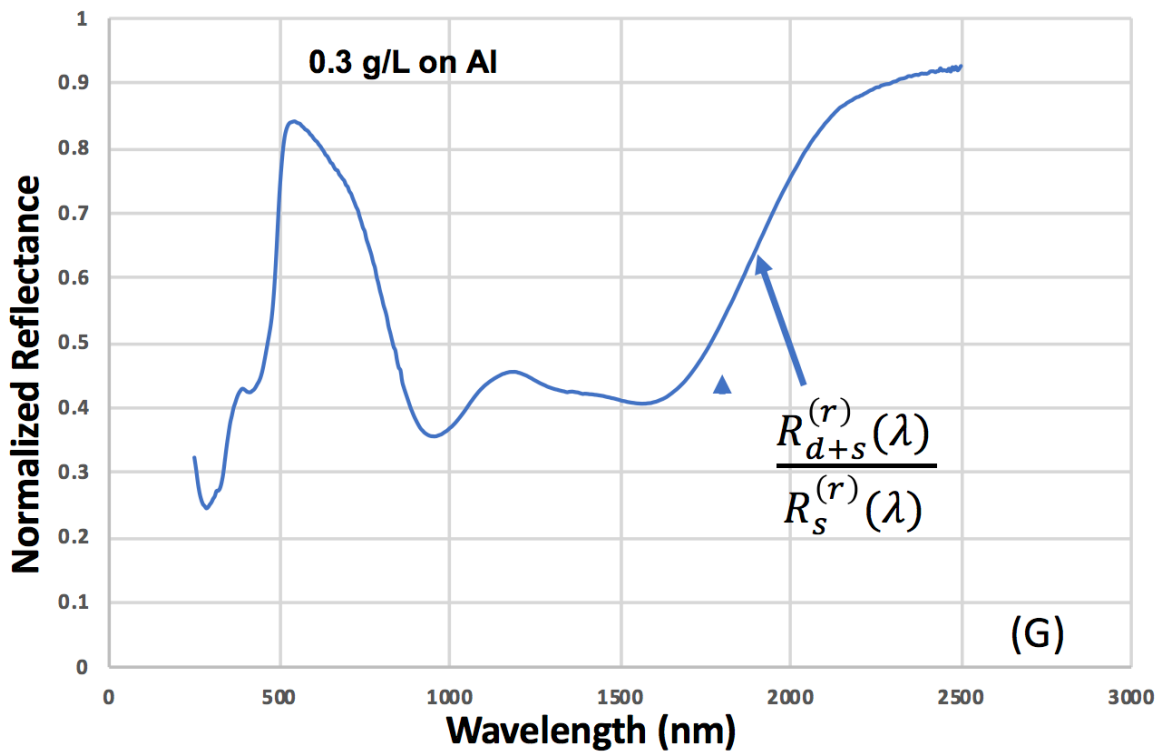
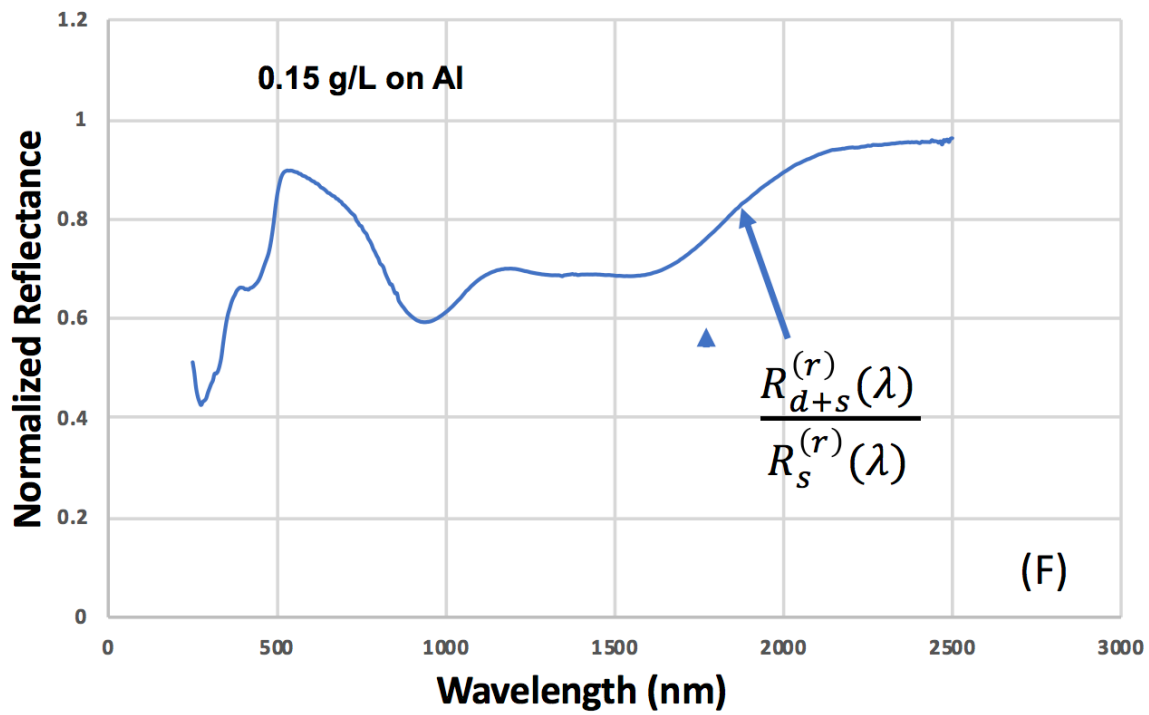


Figure 4 (F) and (G). Background subtracted reference spectra for dye on Aluminum.

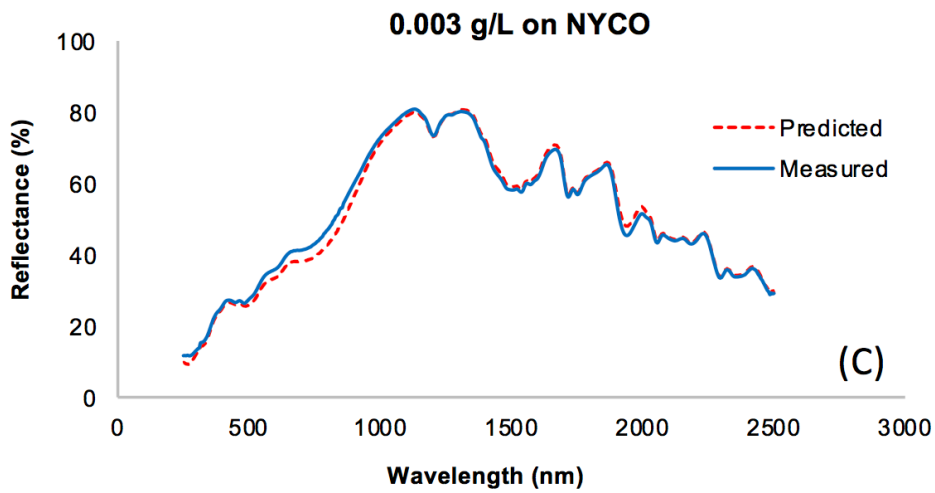
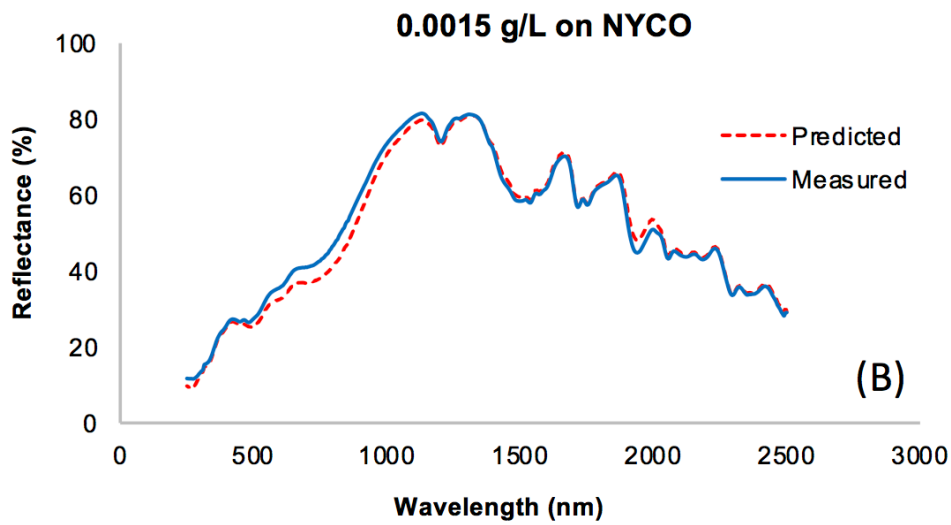
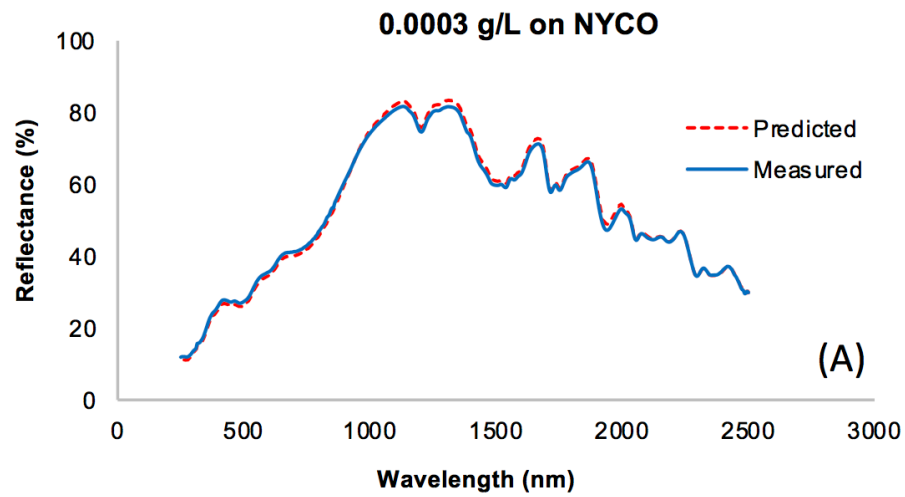


Figure 5 (A), (B) and (C). Measured and modeled reflectance spectra for different concentrations of dye deposited on NYCO fabric.

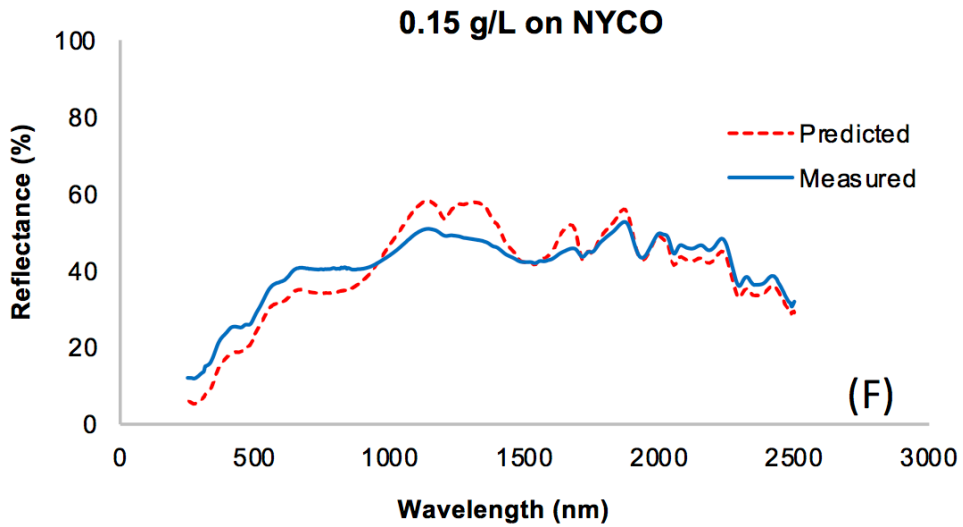
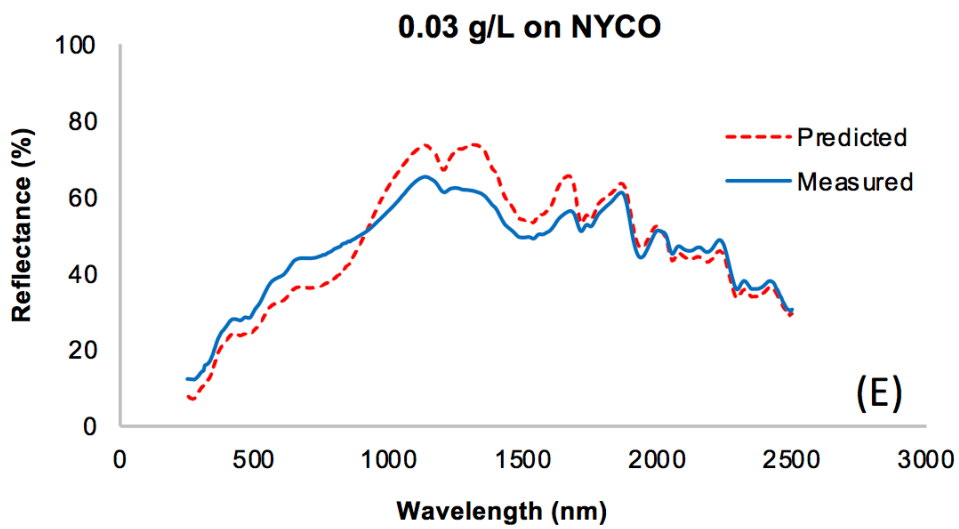
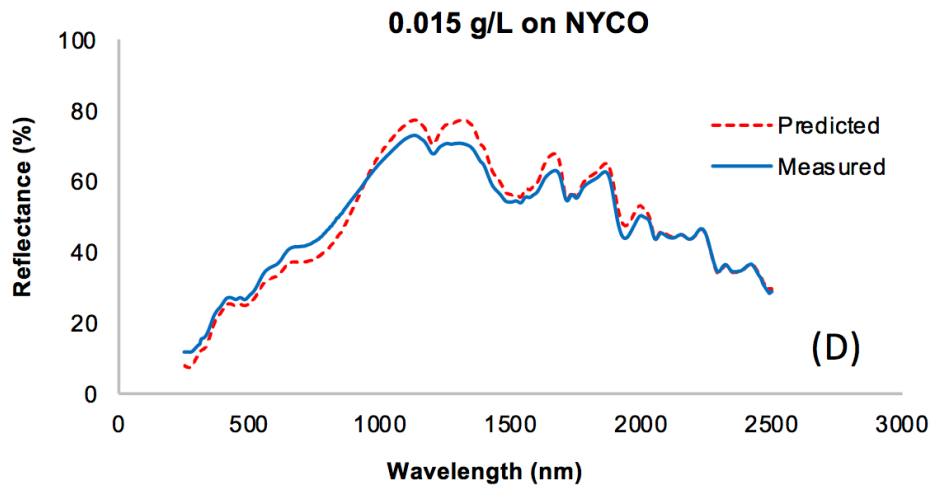


Figure 5 (D), (E) and (F). Measured and modeled reflectance spectra for different concentrations of dye deposited on NYCO fabric.

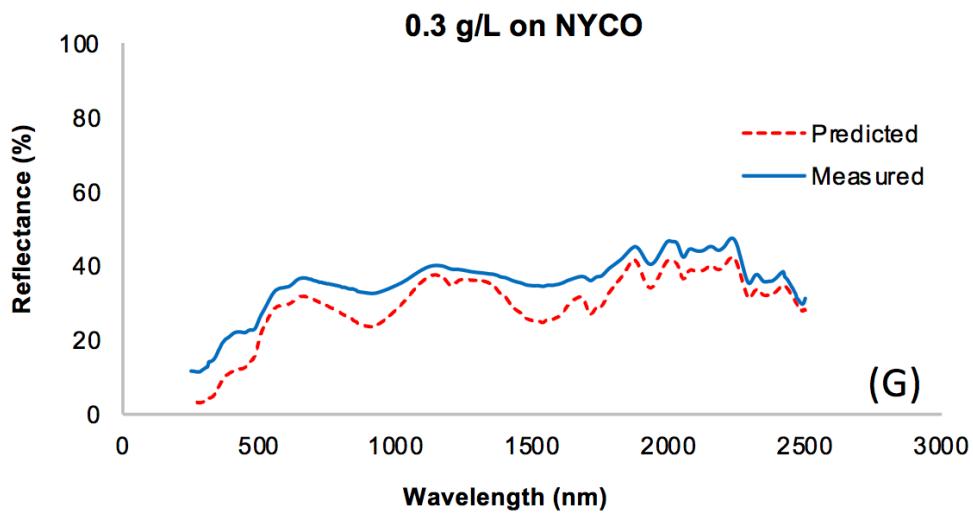


Figure 5 (G). Measured and modeled reflectance spectra for different concentrations of dye deposited on NYCO fabric.

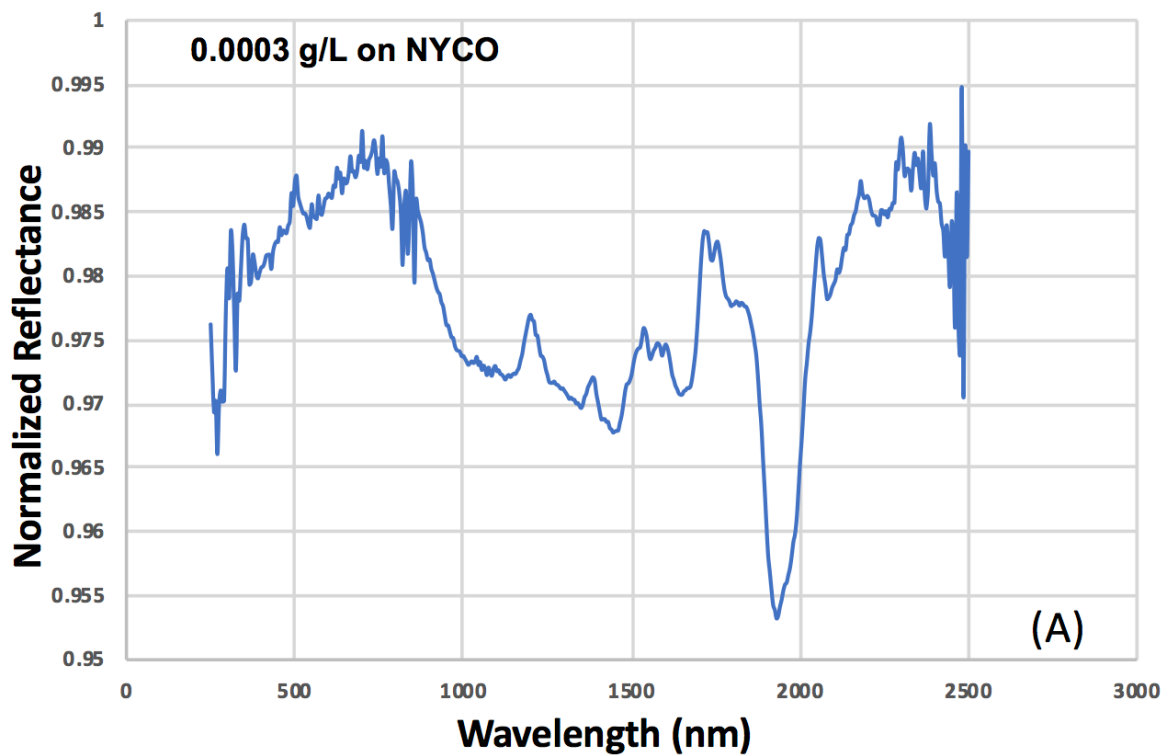


Figure 6 (A). Background subtracted spectra for dye on NYCO.

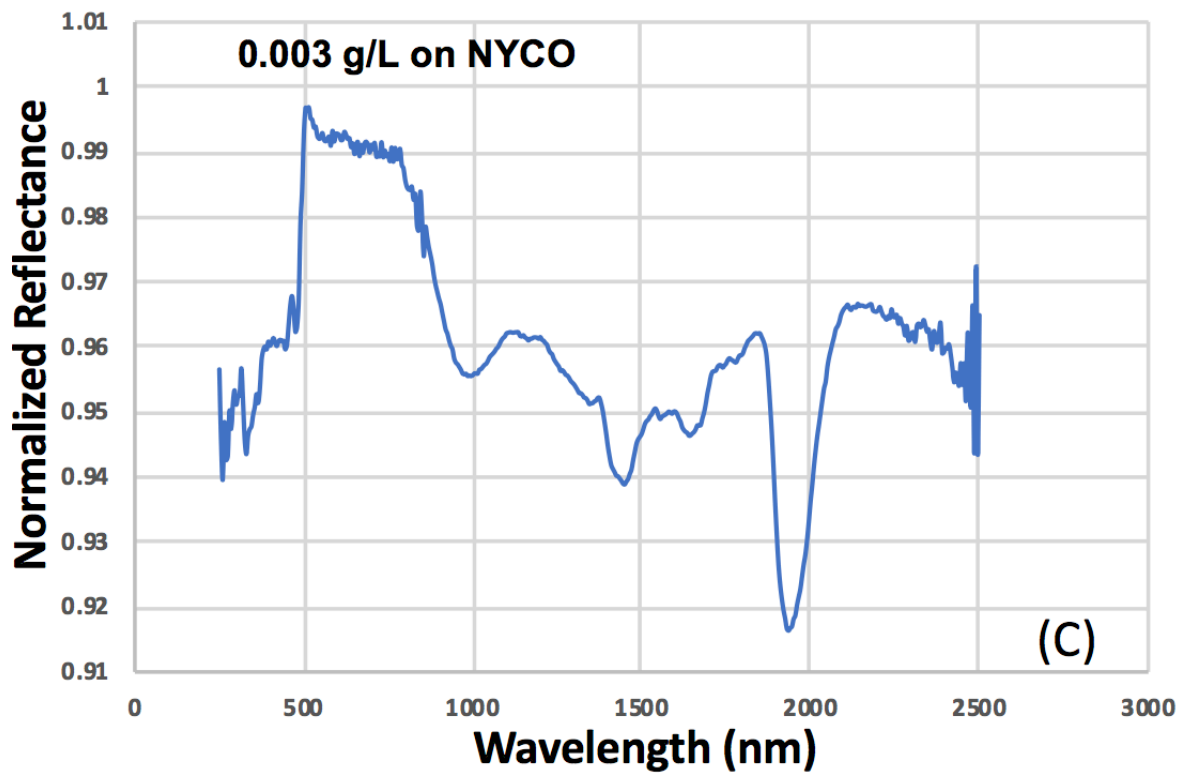
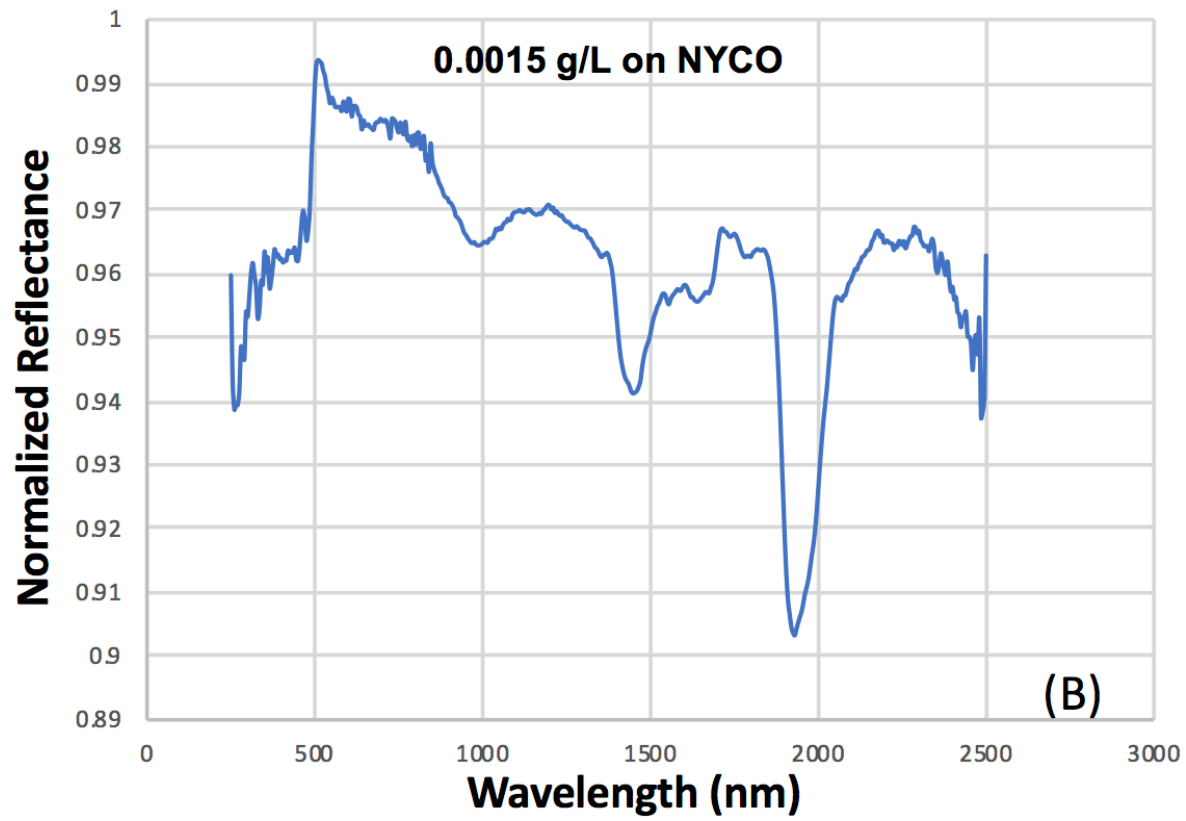


Figure 6 (B) and (C). Background subtracted spectra for dye on NYCO.

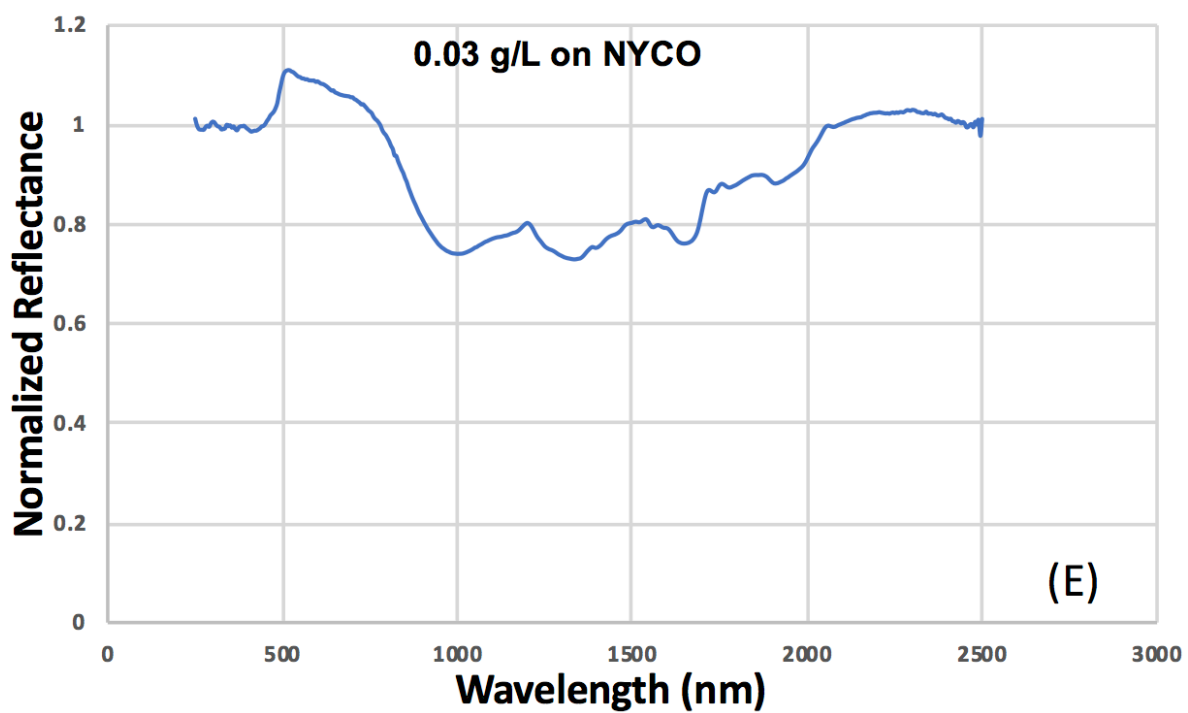
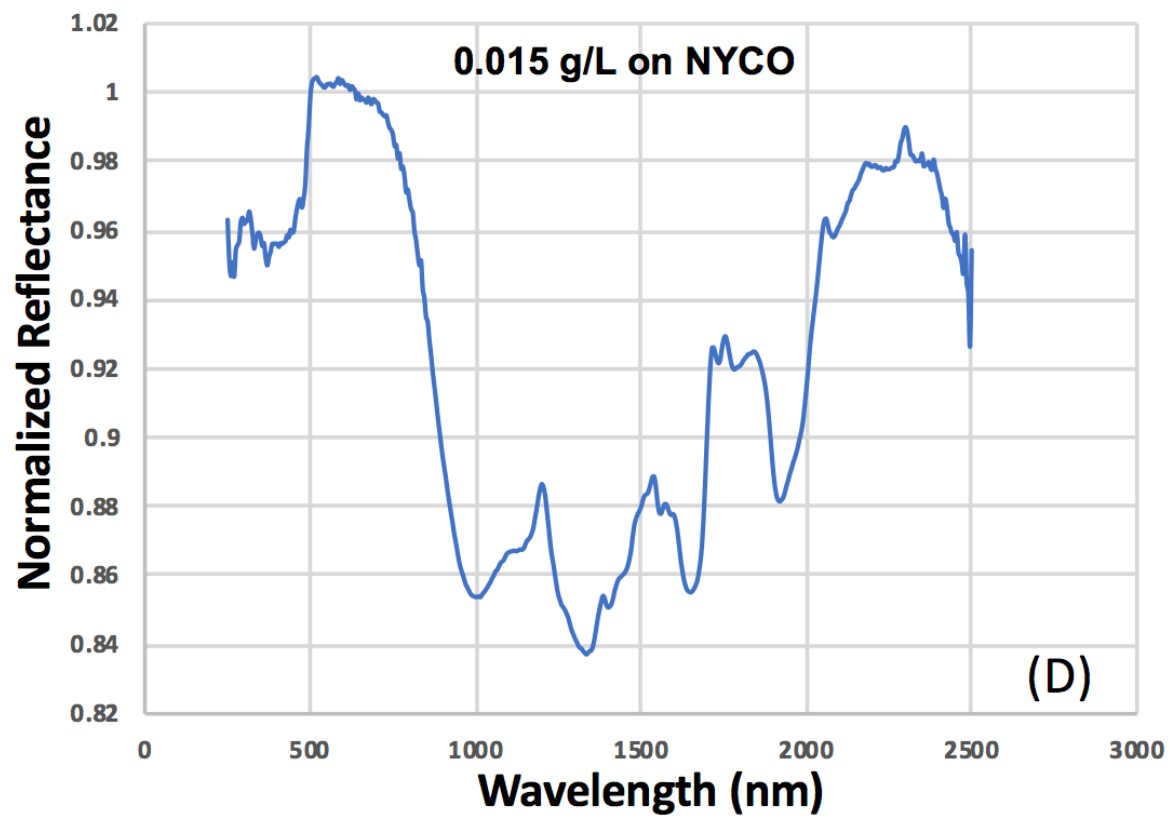


Figure 6 (D) and (E). Background subtracted spectra for dye on NYCO.

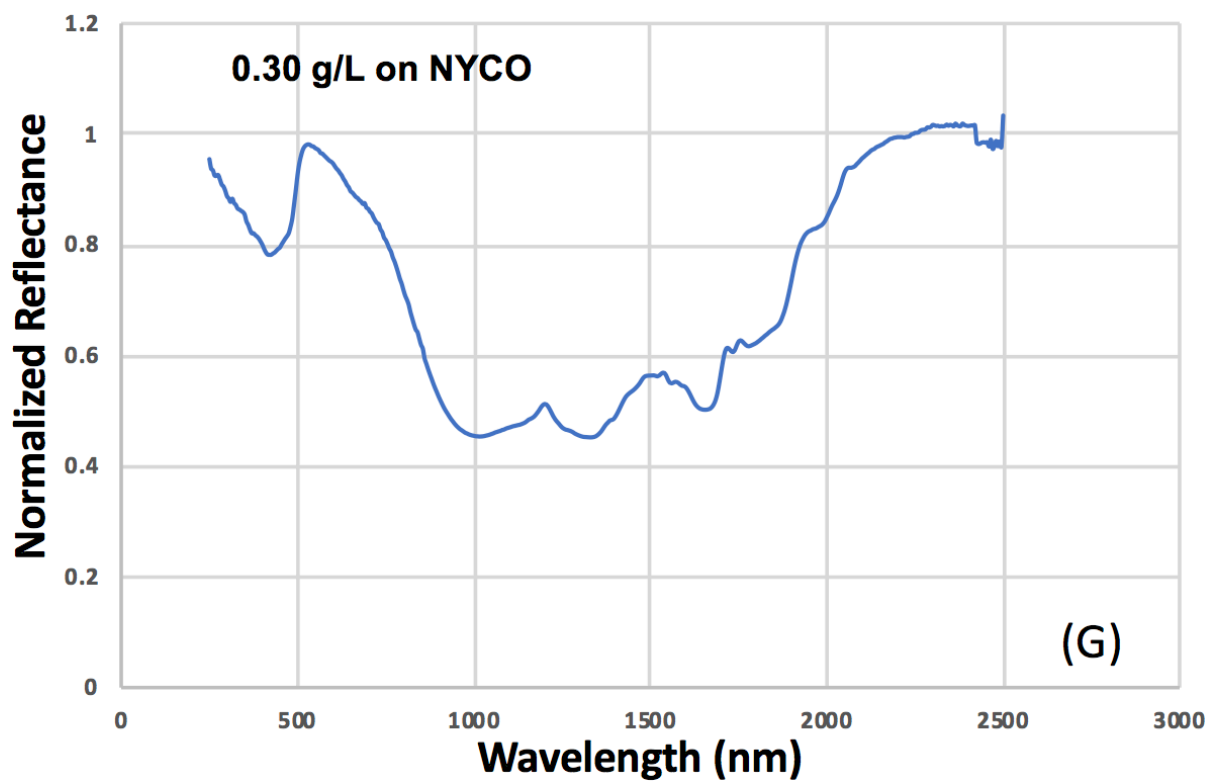
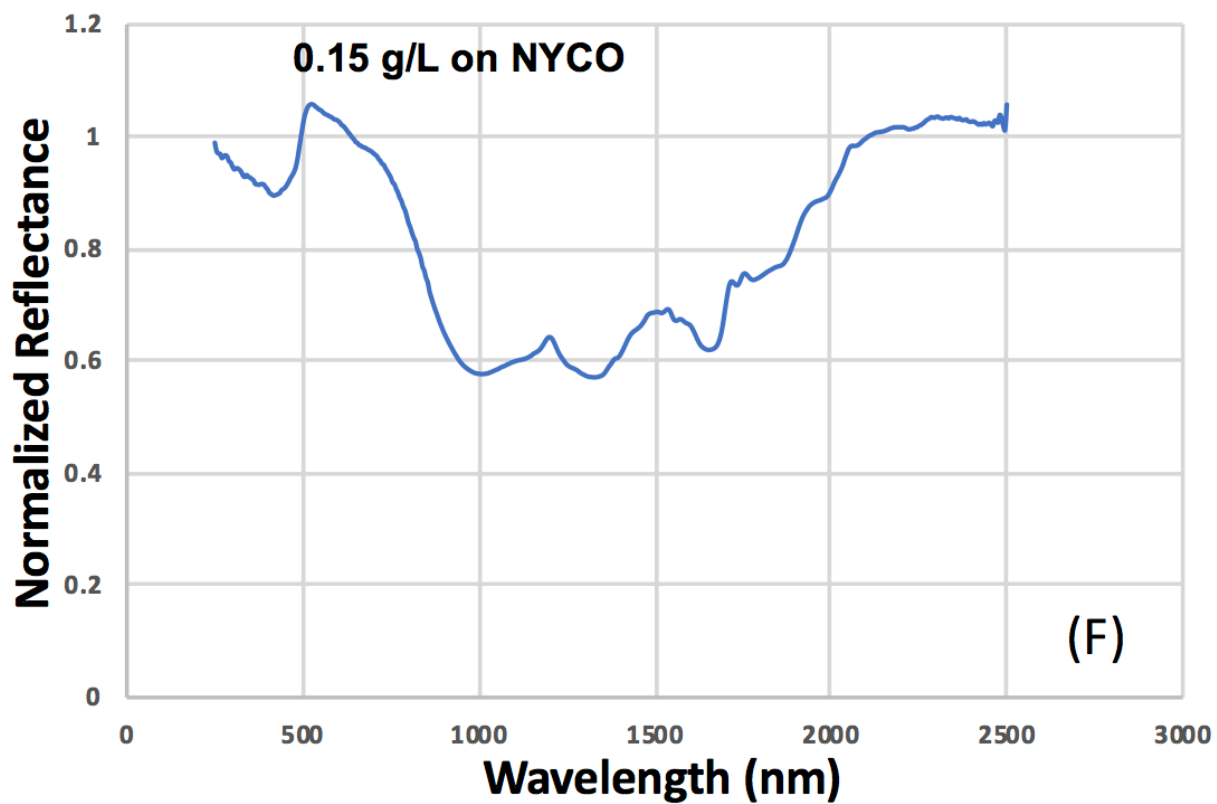


Figure 6 (F) and (G). Background subtracted spectra for dye on NYCO.

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