Evaluation of the Cure State and Water Content at Carbon Fiber containing Composite Surfaces

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Abstract A technique called 'Near Infrared diffuse reflectance spectroscopy' is able to monitor the moisture content in resin rich fiber reinforced composite surfaces and the cure state of resin at the surface of a resin rich fiber reinforced composite. Measurement of water in composites made from 934 resin and T300 fibers was addressed using both normalized absorption spectroscopy and using a "Chemometrics" second derivative partial least squares spectrum analysis. (Chemometrics is the procedure of relating measurements made on a chemical system via application of mathematical or statistical methods). We will show that interpretation of a diffuse reflectance near IR spectrum is more complex than interpretation of a transmission near IR Spectrum, with the result that a partial least squares (Chemometrics) analysis gives better results than a straightforward normalized Beer type plot. Calibration curves have been produced to relate diffuse reflectance near IR spectra to water content for uptake and desorption of water in medium and high performance epoxy resins, high performance adhesives, and carbon fiber reinforced composites. Calibration curves have also been produced to relate the near IR diffuse reflectance spectrum to cure state in high performance adhesives and carbon fiber reinforced composites.

1.Introduction

Composite materials used in the aerospace industry need to be tested for extent of cure and water content. While there are numerous methods employed to determine moisture content and degree of cure in polymeric materials, they have been limited by the inconvenience of sample preparation and the invasive nature of the procedure. It is vital that a technique for monitoring water content and cure state in polymers and composite materials be developed without resorting to destructive and time consuming methods that cannot be used for on-site analysis.

The reliability of near-infrared spectroscopy as a rapid, nondestructive and expedient method has been boosted by the introduction of chemometric data evaluation techniques, diffuse reflectance measurements, and light-fiber optics. It is a low-cost method that requires no sample preparation and with the advent of light-fiber cables, it is now possible to take spectral data without physically removing parts from a structure or samples from a processing line.

2. Experiment, Results, Discussion and Significance

Second derivatives of the near IR spectra were obtained to eliminate baseline deviations and then subjected to partial least squares (PLS) analysis to build a calibration curve.

Two goals were achieved with the help of the calibration curves i.e, primarily water content in epoxy resins for water uptake and desorption in medium and high performance resins and in high performance adhesives and carbon fiber reinforced composites and secondly the degree of cure in high performance epoxy adhesives and high performance carbon fiber reinforced composites.

Water in composites gives rise to an IR peak at 5215 cm^{-1} (**Figure 1**). This peak is free from interference from resin hydroxyls and provides a quantitative measure of water. **Figure 2** shows the calibration curve obtained from partial least squares (PLS) analysis of spectral data. Actual percentage of water values are acquired from agravimetric method.

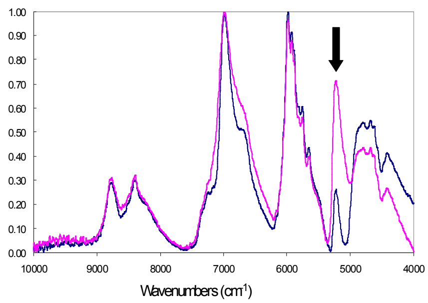

Figure 1. water absorption peak at 5215 cm^{-1}

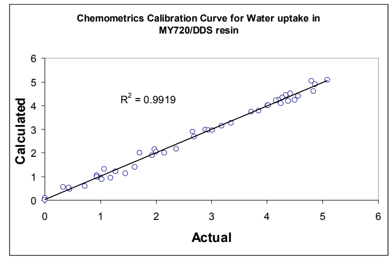

Figure 2. chemometric calibration curve for water uptake of MY720-DDS resin

Epoxy groups produce a strong and well defined combination band at 4530 cm^{-1} as shown in **Figure 3**. The calibration curve in **Figure 4** are derived from PLS analysis of near IR spectra. The actual values of degree of cure are determined from differential scanning calorimetry

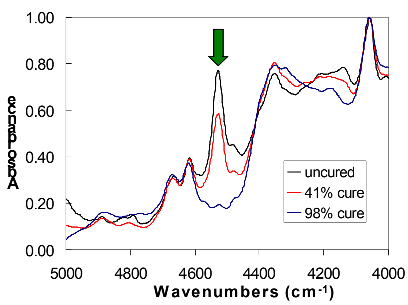

Figure 3. epoxide peak at 4530 cm^{-1}

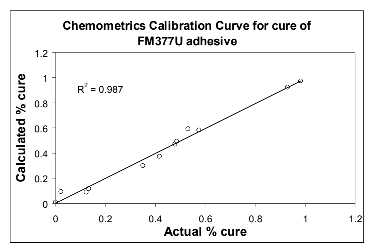

Figure 4. chemometric calibration curve for cure of FM377U adhesive

Both calibration curves (water content and degree of cure) show a good correlation between actual and calculated values as indicated by the R^2 value. These can then be an effective quantitative technique to determined water content and cure state in composite materials

3. Conclusion

Diffuse reflectance near infrared spectrometry was shown to effectively quantify water content and degree of cure in polymeric and composite materials without the hassles of sample preparation and time consumption. Furthermore because the method is nondestructive in nature it can be applied to on site analysis of parts such as in the aviation industry where these materials are mostly used.

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