Agilent 4300 Handheld Portable FTIR

Coatings Analysis: Non-Destructive Testing (NDT) of an Industrial 2K Epoxy Resin-coated panel undergoing accelerated weathering using the ASTM G155 protocol

Application Note

Authors

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Introduction

It is crucial to understand the effect of environmental factors on the performance and lifecycle of paints, coatings, and protective films. One means of accomplishing this goal is to use aging chambers designed to accelerate and control the key parameters. This application note used the Agilent 4300 Handheld FTIR in conjunction with the most common class of aging chamber, called a weatherometer, to measure changes in an industrial 2K epoxy resin finish paint formulation coated onto an industry-standard Q-coupon. We show that the FTIR diffuse spectra effectively detect subtle chemical changes in the coatings as a factor of exposure time and conditions. To accomplish this work, a single Q-panel was weathered, and replicate diffuse reflectance spectra were recorded at intervals of 0, 3, 6, 10, 14, 21, 28, 35, 42, 49, and 56 days. The aging chamber was paused for 10 minutes to allow for multiple measurements of the panel before re-engagement of the weathering cycle.

Figure 1. Agilent 4300 Handheld Portable FTIR (Fourier Transform Infra-Red) spectrometer and the selection of available interfaces.

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The 4300 Handheld FTIR coupled with the diffuse interface is particularly well-suited for this application since it enables the coated coupon to be Non-Destructively analyzed, thus enabling the remeasurement of a single coupon over the weathering period. The collected data can then be statistically analyzed using the multivariate analysis technique called partial least squares. Information gleaned from this work can be correlated to handheld FTIR measurements in a field-based, outdoor weathering farm with the potential to monitor batch performance or formulation potential.

Key benefits for paints and coatings analysis

Chemical information on coatings

The Agilent 4300 FTIR technology yields detailed chemical information on virtually all coating ingredients including binders, extenders, solvents, and additives, as well as the vast majority of organic or inorganic pigments and fillers. The diffuse IR spectra contain features directly related to changes in the coating as a function of weathering, both in the enclosed weathering chamber, or in an outdoor real-time weathering test facility. For this application, both ATR and diffuse interfaces were assessed. Only the diffuse interface was able to provide the data quality, data consistency, and true Non-Destructive analysis required. Therefore, only the diffuse interface results will be discussed in this application note.

Fast, Non-Destructive sample measurement

Each measurement takes <30 seconds (64 scans, 8 cm^{-1} resolution), and no sample preparation is required. Since the panel is measured directly and Non-Destructively, the same panel can be re-examined, thus eliminating the burdensome, costly need for a large set of samples to cover the testing intervals required by conventional destructive testing. This also eliminates errors introduced by using multiple panels.

Intuitive and readily field portable

The 4300 Handheld FTIR system weighs only 2 kg. The software and on-board methods allow less experienced users to get reliable results and rapidly become proficient. The ruggedness and ergonomics of the 4300 Handheld FTIR makes it ideal for supporting R&D and QA/QC efforts, as well as for on-site measurements of coated items regardless of size, including bridges, buildings, transportation vehicles, and so forth.

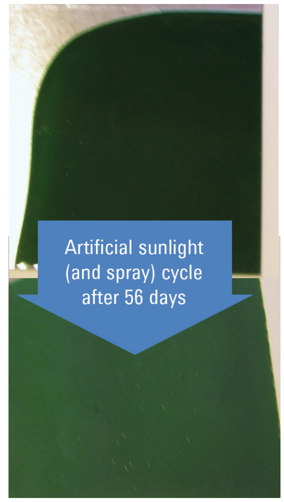

Figure 2. Two-part industrial epoxy resin finish coating sprayed onto a Q-panel, showing the visible effects of accelerated aging in a weatherometer before (unaged, top) and after 56 days (bottom: visible pin-holing).

Results and Discussion

Simulated weathering

An industrial-grade, 2K epoxy resin formulation coating was assessed in a weathering chamber. The conditions were adjusted according to ASTM G155.

Parameter	Value
Light intensity	55 W/m^2 at 340 nm
Black panel temperature	70 °C
Air temperature	47 °C
Humidity	50 %
Continuous 2-step cycle (see Figure 3)	

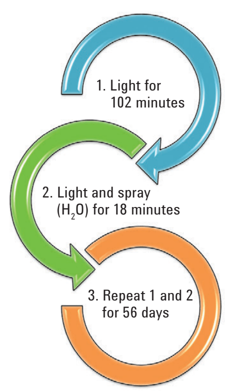

Figure 3. Two-step cycle used in the weathering process. The conditions are the environmental equivalent to sunlight at zenith at Florida's latitude and longitude. The acceleration of changes arises as the sun is not allowed to set for the whole test regime.

The chemical changes that occur during the accelerated weathering test regime (0–56 days, 11 intervals, A-K) are initially subtle, with little performance loss. With an increasing dose of light radiation and moisture, the changes are not only greater, but also begin to affect the performance of the material. For visual clarity, some of the complex changes on a reduced set of the data is shown; note there are numerous areas that change, and some of these are displayed in Figure 4.

- Induction period changes (mild, mainly chemical): 0–21 days
- Mid-term period changes (significant, both chemical and physical changes): 21–35 days
- End-of-life changes (extensive), where physical changes dominate and environmental stress cracking is evident: 35–56 days

Two individual multivariate partial least squares (PLS) models were developed to cover the induction-to-medium-term, and medium-to-long-term (Figure 5) cases.

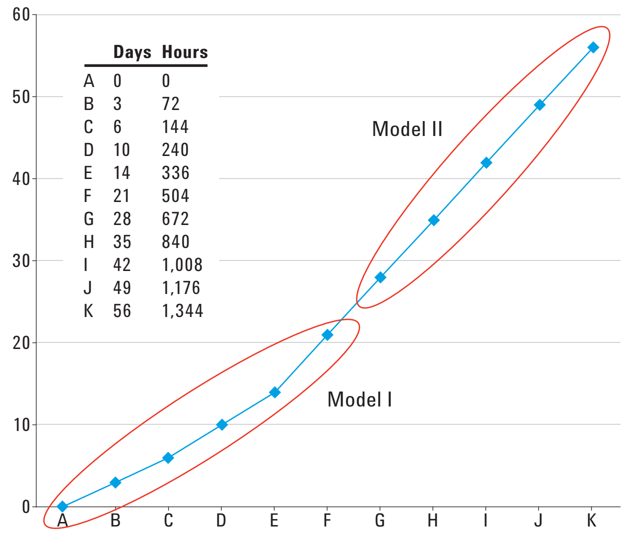

Figure 5. FTIR PLS Model I covers changes in the induction-to-middle exposure period. Model II covers the changes in the medium-to-end-of-life exposure period.

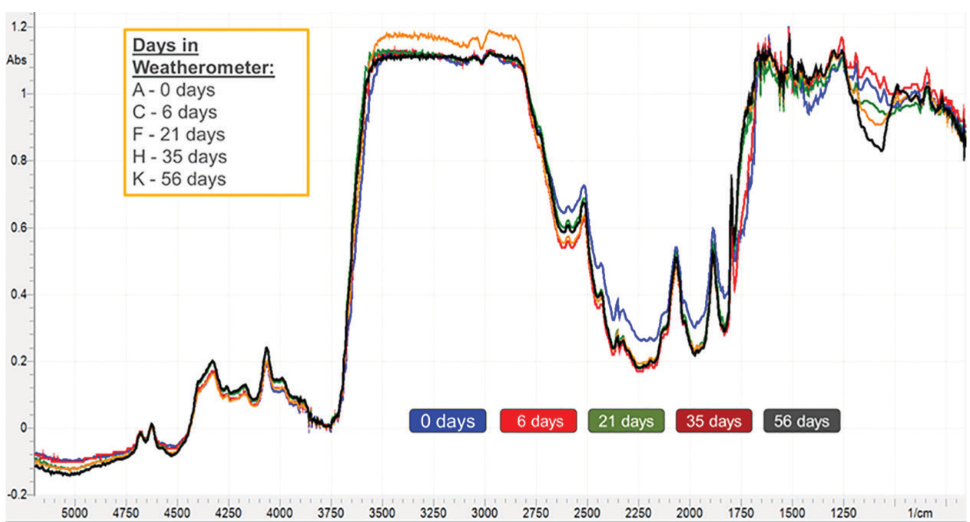

Figure 4. Selected diffuse spectra showing the different chronological changes.

The collected >100 spectra were split into calibration training data and validation data (80:20). The resultant PLS models for the industrial two-part epoxy paint finish (Figure 6) demonstrate high linearity and predictive ability.

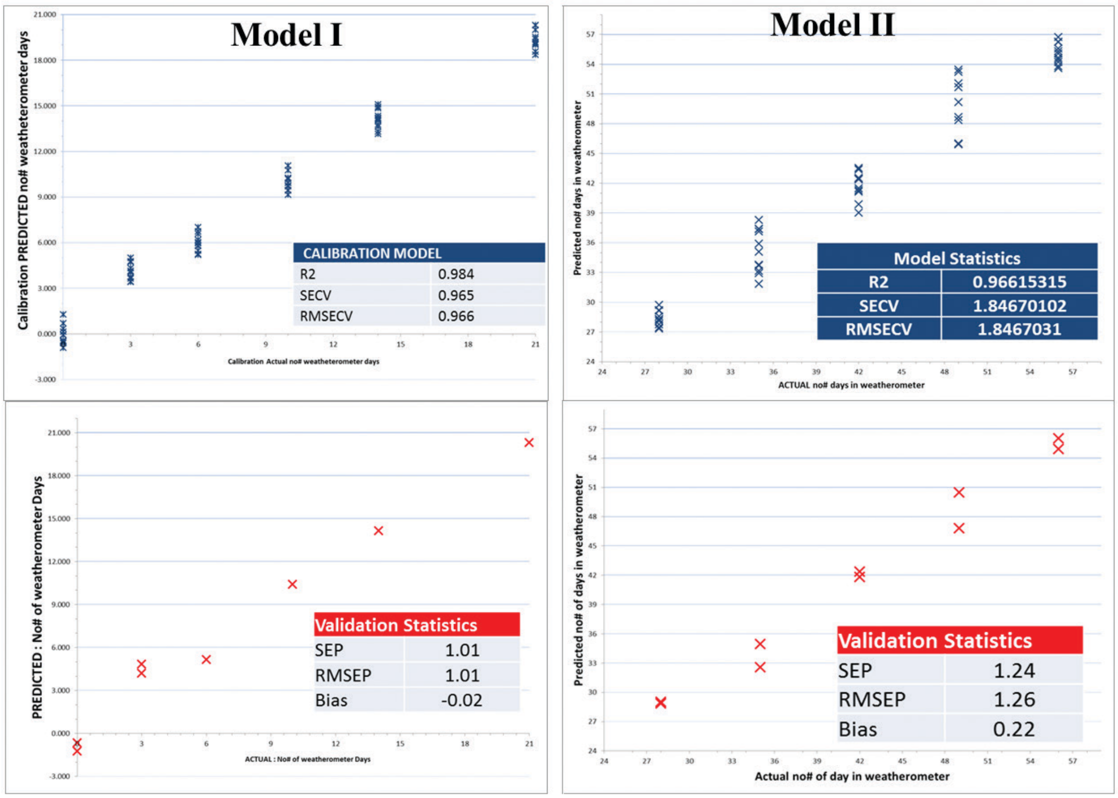

Figure 6. Model I, 0–28 days, and Model II, 28–56 days calibration (top left and right), and independent validation (bottom left and right) actual versus predicted plots.

Conclusion

We used a weatherometer-class aging cabinet and an Agilent 4300 Handheld FTIR in a 56-day accelerated aging study of an industrial 2K epoxy paint finish. This work provides an understanding of the IR spectral response to weathering a baseline coating formulation. Both chemical and physical changes invisible to the naked eye were elucidated by means of analysis of the spectra. By changing various constituents of the coatings matrix, the performance of different formulations were tested, or individual additives and their effects targeted. As a consequence of our Non-Destructive sample analysis using the handheld FTIR, only one coupon was needed for each formulation. Therefore, the performance of many different formulations were ascertained in one complete aging cycle, within the confines of a single cabinet. Moreover, other types of aging chambers specifically designed for high temperatures, outer space, aggressive chemicals, corrosive environments, salt water, and their combination would equally benefit from this type of Non-Destructive spectroscopic information-rich analysis and modeling.

In summary, we have shown:

- Successful model creation and validation using an Agilent Cary 4300 Handheld FTIR system for the accelerated aging of an industrial grade epoxy coating (Model I: 0–28 ±1.01 days, Model II: 28–56 ±1.25 days)
- Changes in the diffuse reflectance IR spectra that cover the 0–56 (accelerated) days aging regime. Similar changes can be applied to other coating types, glossy or matt
- A means for practical condition monitoring of in-service paint to end-of-life state, which can aid in prolonging performance of the underlying asset
- The opportunity to quickly examine whole sets of potential formulations in a standard weathering cabinet to ascertain the composition most resistant to chemical change
- The ability to discern subtle, initial chemical changes that can improve test cycle times for in-lab weathering studies or in-field, real-time testing
- The ability to monitor long-term real-time aging studies *in-situ* Non-Destructively with the choice of increasing the sampling intervals to suit the spectral, chemical, and physical changes without sacrificing actual painted coupons

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