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EPOXY CHEMISTRY UNVEILED: NEW INSIGHTS IN PROTECTIVE COATING CHARACTERIZATION AND CORROSION CONTROL THROUGH FOURIER-TRANSFORM INFRARED SPECTROSCOPY (FTIR)

FTIR spectroscopy

Polymerization monitoring

Materials chemical characterization

Internal corrosion is a well-known phenomenon that significantly impacts many pipeline operating companies world-wide. To prevent short-time failure and enhance corrosion resistance, several epoxy-based internal coatings have been developed. Fusion-Bonded Epoxy (FBE) resins constitute a highly remarkable type of coating material that have been extendedly used since 1960's decade and continues to be the most utilized protective system as a result of its excellent adhesion to steel, good chemical resistance and other characteristics of interest.[1]

To ensure an appropriate performance of the FBE coated pipelines when exposed to fluids and operating conditions of interest in the field, different mechanical, physical and chemical test are carried out by **CIC laboratories**. This assessment includes the evaluation of properties such as blistering tendency, thickness, porosity and adherence measurements.

To evaluate the chemical resistance of coating materials, FTIR spectroscopy is, perhaps, the most versatile non-destructive analysis technique and provides detailed information on structural changes of the polymeric matrix.

As in all types of spectroscopy, FTIR involves the interaction (quantized absorption) of electromagnetic radiation with matter within a wavelength range from 2500 to 20000 nm (4000 to 500 cm^{-1}). Photon energies associated with this part of the infrared spectrum are capable of inducing vibrational excitation of covalently bonded atoms providing a "spectrum" as result.

Recent implementation of (H)ATR FTIR analysis by **CIC** as a robust quality control test for coating materials (and other samples), permits a rapid correlation of the changes associated to intensities and absorption frequencies in the IR spectrum, therefore confirming if there were significant chemical modifications involved, that might negatively impact the desired corrosion protective function of the polymer.

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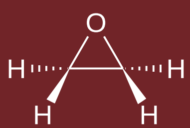
Case study: chemical resistance evaluation of a FBE internal coating sample

Prior to field conditions exposure, the ATR FTIR spectrum (4000 - 600 cm^{-1}) of a FBE sample, correctly applied on the surface of the appropriate metallic test panel, was recorded to identify the major organic chemical groups within the polymeric structural backbone. As expected, three major IR absorptions were discriminated[2,3] and correlated to the molecular structure of a typical epoxy-amine resin (Figure 1).

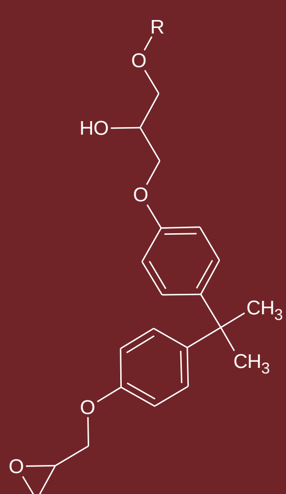


Epoxide ring structure

- **3038 cm^{-1}**. C-H residual oxyrane ring stretch vibration.
- **1033 cm^{-1}**. C-O aliphatic stretch vibration.
- **895 cm^{-1}**. CH_2 rocking vibration of residual oxyrane.

Figure 1. Non-exposed FBE FTIR spectrum

After exposure (well fluid, 3000 psi and 50ºC ± 2ºC) for 72 hours, a new spectrum was acquired on the coating surface. Comparison of the spectra confirmed the preservation of molecular integrity with minor changes associated to further polymerization (curing process) of unreacted epoxy residues and amines (Figure 2). An intensity increase of the 1033 cm^{-1} band together with a 895 cm^{-1} absorption decrease, comprise strong evidence of the above mentioned. This spectroscopic observation was later confirmed by Barcol hardness measurements, changing from 43 to 67.



Residual bisphenol A type epoxy

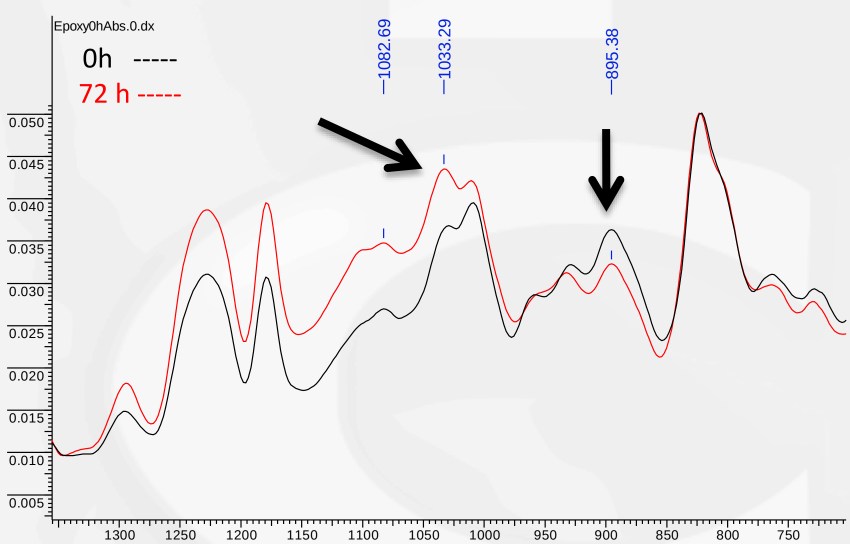


Figure 2. Comparative FTIR spectra

Thus, the performed FTIR analysis revealed a desirable chemical behavior of the FBE resin under the evaluated exposure conditions: improvement of its protective properties by increasing the degree of crosslinking on the coating surface. This chemical change will prevent the appearance of early defects that might compromise the metallic substrate, making it suitable for field application.

Consequently, FTIR analysis constitutes a rapid, reliable and accurate spectroscopic tool to evaluate the behavior of all types of organic coating materials when exposed to a specific medium, allowing real time evaluation of chemical corrosion susceptibility.

CIC specialists are currently exploring and developing other applications of FTIR that include accurate identification and quantification of prohibited (hazardous) polymer additives, quantification of the extent of curing processes and stability evaluation of oil industry chemical additives, among others.

References

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Corporación para la Investigación de la Corrosión