

Compare functions of FTIR software for structural analysis of epoxy paints on steel structures for coating fingerprinting certificate

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Abstract

This progressive article on Fourier-transform infrared (FTIR) structural analysis showcases the practicality and simplicity of the provision of **Coating Fingerprint Certificate** for 2-component epoxy coatings for the supply of polymeric coatings from local paint manufacturers as quality assurance requirement of the coatings supplied. Fingerprinting regions of FTIR for epoxy resin and hardener are proposed and the confidence level of acceptance for quality assurance and quality control (QA & QC) is suggested at $\geq 90.0\%$. We conclude that, the structural analysis by FTIR for complete **Coating Fingerprint Certificate** for epoxy resin and hardener is reproducible when **High Sensitivity Compare** feature of the FTIR software is to be strictly followed. This algorithm depends on x - (wavenumber) and y - (absorbance) vectors. This function is able to discriminate minute difference of different components as well as the compositional change of the components among samples. Besides, rejection or acceptance of the samples can be easily done by setting the threshold value at 0.90 using **High Sensitivity Compare** feature of the FTIR software. Finally, we attempt to compare inorganic fillers for polymer coatings (e.g. Rutile or Anatase of TiO_2) using FTIR, which seems to be practical as well.

Introduction

Currently, only 2-component epoxy coating (intermediate materials) was used in the evaluation of the practicality of the structural analysis by Fourier-transform infrared (FTIR) for complete **Coating Fingerprint Certificate** [1] introduced by Institute of Materials, Malaysia (IMM) Task Force on Coatings Fingerprinting. Deliveries of 1st phase of the task force (2013 – 2014) are:

1. Tentative Coating Fingerprint Certificate for 2-component intermediate materials of epoxy coatings was presented.
2. FTIR is a simple and reliable tool for the study of reproducibility (i.e. to fingerprint) of the epoxies and hardeners as well as to differentiate different types of epoxies and hardeners without any intrusion of paint formulations.
3. Fingerprinting regions of FTIR for epoxy resin and hardener are proposed and the confidence level of acceptance for quality assurance and quality control (QA & QC) is proposed at $\geq 90.0\%$.

We would like to recapitulate here, the complete **Coating Fingerprint Certificate** [1] for polymeric coatings consist of two parts, i.e. (1) physical analyses (e.g. viscosity, density, color code, non-volatile matter (by mass), weight solid of Zn metal/Total Zn etc; and (2) structural analysis by FTIR (which shall be carried out immediately after each batch of the production in the paint factory).

For this article, we are looking into the Compare functions of FTIR software for structural analysis of 2-component intermediate materials of epoxy coatings, which serve as an important tool for QA & QC for the batch-to-batch reproducibility of the epoxies and hardeners as well as for different epoxies and hardeners by estimation of correlation (r).

We shall emphasize here once more, this FTIR analysis coupled with the considerations needed using the Compare functions of FTIR software shall not be limited to 2-component epoxy coating, but has to be extended to inorganic zinc coating, epoxy-zinc coating, polyurethane coating, acrylic coating, polyester coating etc.

Experimental

FTIR sample collection

Sample collection was published in ref. [2] and is briefly sketched here. Polymeric coatings, i.e. epoxy resin (or base) and hardener (or curing agent) from local Paint Manufacturer A were analyzed. A total of 3 samples/batch or 3 samples/mixing tank with minimal of 50 g of sample mass for epoxy resin as well as hardener were supplied. Sampling of samples at the end stage of production (before packing) was done from Top, Middle and Bottom of the mixing tanks. Samples were sent for FTIR analysis within 4 days after sample collection. A total of 2 batches of samples were used for this study on the reproducibility of the results. These samples were analyzed as received.

Epoxy_ **BxT**(or **M** or **B**) y - z denotes epoxy resin of x^{th} Batch for y^{th} sample at the location of Top (or Middle or Bottom) and with the z^{th} FTIR scanning. Analogue sample coding was adopted for Hardener_ **BxT**(or **M** or **B**) y - z .

FTIR analysis

To fingerprint polymeric coatings, ASTM D7588-11 [3] standard is followed. As mentioned before [4], there is lack of guide in ASTM D7588-11 for the interpretation of FTIR

spectra, *i.e.* the practical approaches on estimation of the degree of similarity (or correlation) (r) between two FTIR spectra for the same or different polymeric coatings.

Spectroscopic studies were performed on the intermediate materials of polymeric coatings, *i.e.* epoxy resin and hardener independently. FTIR analysis was carried out using the Attenuated Total Reflection accessory (ATR) on Nicolet iS5 (Madison, UK). FTIR spectra were recorded in the transmittance mode over the range of 600 - 4000 cm^{-1} by averaging 32 scans at a maximum resolution of 4 cm^{-1} . Triplicate analysis for each sample was carried out, where a fresh sample was used for each analysis. The material of ATR crystal is Diamond coated with ZnSe germanium. The spectra of FTIR were analyzed by OMNIC Software Suite (Madison, UK).

Quality control of the intermediate materials

Absorbance spectra were baseline corrected. The “average” FTIR spectrum of sample from **Top**, **Middle** and **Bottom** of the mixing tank was adopted as the reference spectrum (c.f. ref. [2].) The degree of similarity, which is termed as *correlation* (r), of a spectrum was generated by comparing the spectra of the samples to that of the reference using the *Normal Compare* OR *High Sensitivity Compare* features of the FTIR software. Degree of similarity is directly proportional to quantities of r , *i.e.* $r = 1$ represents complete matching of the sample spectrum to that of the reference spectrum.

The standard compare algorithms commonly used in infrared (IR) spectroscopy are well suited to identify unknown materials or to discriminate between materials that are significantly different, *i.e.* correlation compare algorithm of the FTIR software depends on x -vector (wavenumber / cm^{-1}) only. However, these standard algorithms often lack the sensitivity required when the materials being compared only exhibit very minute differences, *e.g.* for routine quality assurance and quality control of batch-to-batch production of the intermediate materials of the epoxy paints.

The Compare QCheck feature of the OMNIC Software (belongs to *Normal Compare* feature of the FTIR software), is a single scale x -correlation. This compare function depends mainly on the “structural analysis” of the component(s) of the intermediate materials of epoxy or hardener. This means, this compare function is not sensitive to compositional change of the similar structure(s) (or slight different structures) of epoxy or hardener. Very often, $r \rightarrow 1$ is observed even if there is significant variation of composition change of epoxy or hardener. This function is only useful for qualitative check to discriminate between obviously different materials.

On other hand, the high sensitivity function in OMNIC QCheck (belongs to *High Sensitivity Compare* feature of the FTIR software) effectively provides better sensitivity when the degree of similarity between samples is high. This algorithm depends on x - (wavenumber) and y - (absorbance) vectors. This function is able to discriminate minute difference of different components as well as the compositional change of the components among samples. For this study, quantities r (from 0 to 1) were estimated firstly for spectrum with wavenumbers from (i) 600 - 4000 cm^{-1} , and subsequently from (ii) 1000 - 1300 cm^{-1} (C-O-C) & (iii) 700 - 900 cm^{-1} (C-O-C) for epoxy resin; and (iv) 1000 - 1400 cm^{-1} (C-N) for hardener.

Results and discussion

Generation of reference FTIR spectrum from **Top**, **Middle** and **Bottom** of the mixing tank were sketched in ref. [2]. After automatic baseline correction, the spectra in transmittance mode shall be converted to absorbance mode. The three spectra from **Top**, **Middle** and **Bottom** were “averaged” using the commercial FTIR software for the generation of reference spectrum for Epoxy1 and Hardener1 (refer Figure 1).

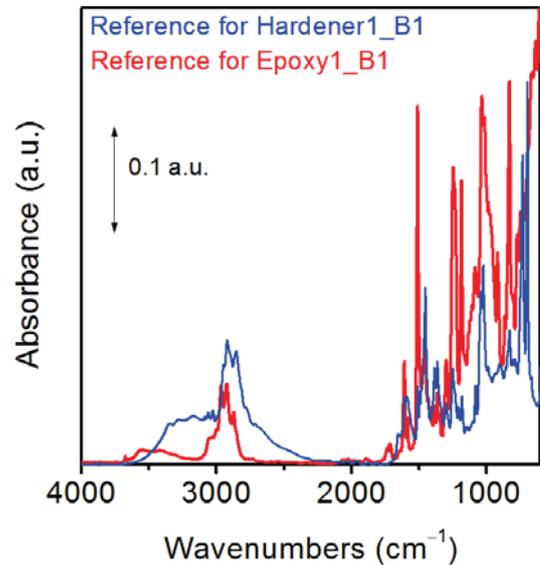


Figure 1 Reference spectra for Epoxy1 and Hardener 1

Homogeneity of epoxies and hardeners at Top, Middle and Bottom of the mixing tanks and reproducibility of the epoxies (or hardeners) of Batch 2 as compared to Batch 1 by estimation of r

The degree of similarity (r) (in term of paint formulation), of Epoxy1_B1 (or Hardener1_B1) collected from **Top**, **Middle** and **Bottom** of the mixing tank was generated by comparing the spectra of the sample to that of the reference spectrum (Epoxy1_B1 or Hardener1_B1) using the *Normal* and *High Sensitivity* features of the FTIR software. Quantities r for Epoxy1 and Hardener1 and are tabulated in Tables 1 and 2.

Table 1A Estimation of r for Reference Epoxy1_B1 to Epoxy1_B1T(or M or B) for 1st FTIR scanning with *High Sensitivity Compare* feature (reprint with permission from ref. [2])

Sample Code	r	r	r	Reference Spectrum
	600 – 4000 cm^{-1}	1000 – 1300 cm^{-1} (C-O-C)	700 – 900 cm^{-1} (C-O-C)	
Epoxy1_B1B1	0.9960	0.9996	0.9997	Reference for Epoxy1_B1
Epoxy1_B1M1	0.9910	0.9990	0.9996	
Epoxy1_B1T1	0.9871	0.9990	0.9990	
Epoxy1_B2B1	0.9970	0.9990	0.9990	
Epoxy1_B2M1	0.9940	0.9970	0.9990	
Epoxy1_B2T1	0.9930	0.9990	0.9970	

Table 1B Estimation of r for Reference Epoxy1_B1 to Epoxy1_B1T(or **M** or **B**) for 1st FTIR scanning with Normal Compare feature

Sample Code	r 600 – 4000 cm ⁻¹	r 1000 – 1300 cm ⁻¹ (C-O-C)	r 700 – 900 cm ⁻¹ (C-O-C)	Reference Spectrum
Epoxy1_B1B1	0.9996	1.0000	1.0000	Reference for Epoxy1_B1
Epoxy1_B1M1	0.9997	0.9999	0.9999	
Epoxy1_B1T1	0.9995	0.9998	0.9999	
Epoxy1_B2B1	0.9980	0.9992	0.9992	
Epoxy1_B2M1	0.9981	0.9992	0.9993	
Epoxy1_B2T1	0.9983	0.9993	0.9995	

The quantities r in this study are correlated to the paint formulation. It is relatively common to set $r \geq 0.90$ as the acceptable tolerance in order to suggest the similarity of different samples or different batches of similar samples. All epoxies and hardener are homogenous in the mixing for Epoxies or Hardener 1, 2 and 3 for Batches 1 and 2 at different locations of **Top**, **Middle** and **Bottom** of the mixing tanks. Besides, quantities $r \geq 0.90$ are recorded for Epoxies (or Hardeners) 1, 2 and 3 when Batch 1 was compared to Batch 2 at different locations of **Top**, **Middle** and **Bottom** of the mixing tanks.

In all cases, $r_{\text{High Sensitivity}} < r_{\text{Normal Sensitivity}}$ are noted. In some cases, $r_{\text{Normal Sensitivity}} = 1.0000$ are recorded, which are rather unrealistic, *i.e.* 100% matching between the Reference spectrum and the sample. Hence, we propose here, $r_{\text{High Sensitivity}}$ is more suitable to be used for checking the homogeneity of epoxies and hardeners at **Top**, **Middle** and **Bottom** of the mixing tanks and the batch-to-batch reproducibility of the epoxies and hardeners

Table 2A Estimation of r for Reference Hardener1_B1 to Hardener1_B1T(or **M** or **B**) for 1st FTIR scanning with **High Sensitivity Compare** feature (reprint with permission from ref. [2])

Sample Code	r 600 – 4000 cm ⁻¹	r 1000 – 1400 cm ⁻¹ (C-N)	Reference Spectrum
Hardener1_B1B1	0.995	0.9998	Reference for Hardener1_B1
Hardener1_B1M1	0.993	0.999	
Hardener1_B1T1	0.993	0.999	
Hardener1_B2B1	0.998	0.9997	
Hardener1_B2M1	0.994	0.9995	
Hardener1_B2T1	0.994	0.9996	

Table 2B Estimation of r for Reference Hardener1_B1 to Hardener1_B1T(or **M** or **B**) for 1st FTIR scanning with Normal Compare feature

Sample Code	r 600 – 4000 cm ⁻¹	r 1000 – 1400 cm ⁻¹ (C-N)	Reference Spectrum
Hardener1_B1B1	0.9996	1.0000	Reference for Hardener1_B1
Hardener1_B1M1	0.9998	1.0000	
Hardener1_B1T1	0.9997	1.0000	
Hardener1_B2B1	0.9994	0.9997	
Hardener1_B2M1	0.9989	0.9997	
Hardener1_B2T1	0.9977	0.9993	

To estimate the r for Epoxy1 as compared to Epoxy2, Epoxy3 and PU; and Hardener1 as compared to Hardener2, Hardener3 and NCO

Epoxy1, Epoxy2, Epoxy3 and poly(urethane) (PU) are with different paint formulations. Analogues to epoxy, Hardener1, Hardener2, Hardener3 and isocyanate (NCO) are with different paint formulations. Tables 3 and 4 clearly demonstrate that FTIR analysis is a simple tool to differentiate different types of epoxies, between epoxy & PU, different types of hardeners; and between hardener & NCO with $r < 0.90$ when **High Sensitivity Compare** feature is used.

Again, In all cases, $r_{\text{High Sensitivity}} < r_{\text{Normal Sensitivity}}$ are noted. When Epoxy1 is compared to Epoxy2 and Epoxy3, $r_{\text{Normal Sensitivity}} \geq 0.90$ are recorded, which are unacceptable because Epoxy1, Epoxy2 and Epoxy3 are having structural and compositional differences. Hence, we emphasize here, $r_{\text{High Sensitivity}}$ is more suitable to be used for the comparison of different types of epoxies and hardeners.

Table 3A Estimation of r for Reference Epoxy1_B1 to Epoxy2, Epoxy 3 and PU with **High Sensitivity Compare** feature (reprint with permission from ref. [2])

Sample Code	r 600 – 4000 cm ⁻¹	r 1000 – 1300 cm ⁻¹ (C-O-C)	r 700 – 900 cm ⁻¹ (C-O-C)	Reference Spectrum
Epoxy2_B1	0.5	0.5	0.5	Reference for Epoxy1_B1
Epoxy3_B1	0.6	0.8	0.6	
PU_B1	0.05	0.07	0.06	

Table 3B Estimation of r for Reference Epoxy1_B1 to Epoxy2, Epoxy 3 and PU with Normal Compare feature

Sample Code	r 600 – 4000 cm ⁻¹	r 1000 – 1300 cm ⁻¹ (C-O-C)	r 700 – 900 cm ⁻¹ (C-O-C)	Reference Spectrum
Epoxy2_B1	0.90	0.93	0.90	Reference for Epoxy1_B1
Epoxy3_B1	0.96	0.98	0.94	
PU_B1	0.11	0.14	0.12	

Table 4A Estimation of r for Reference Hardner1_B1 to Hardener2, Hardener3 and NCO with **High Sensitivity Compare** feature (reprint with permission from ref. [2])

Sample Code	r 600 – 4000 cm^{-1}	r 1000 – 1400 cm^{-1} (C-N)	Reference Spectrum
Hard-ener2_B1	0.3	0.2	Reference for Hard-ener1_B1
Hard-ener3_B1	0.3	0.1	
NCO_B1	0.04	0.002	

Table 4B Estimation of r for Reference Hardner1_B1 to Hardener2, Hardener3 and NCO with Normal Compare feature

Sample Code	r 600 – 4000 cm^{-1}	r 1000 – 1400 cm^{-1} (C-N)	Reference Spectrum
Hardener2_B1	0.5	0.4	Reference for Hard-ener1_B1
Hardener3_B1	0.6	0.2	
NCO_B1	0.08	0.004	

Setting the threshold to reject or to accept one sample using High Sensitivity Compare feature

For routine QA & QC check of FTIR, which shall be carried out immediately after each batch of the production in the paint factory, can be done easily with the assistance from the commercial FTIR software. As in this case, when the **High Sensitivity Compare** feature is opted and the threshold can be set at 0.90 (equivalent to $r \geq 0.90$) (c.f. Figure 2), we can immediately **REJECT** Epoxy2, Epoxy3 and PU when these samples are compared to Epoxy1 (c.f. Figure 3) by simply referring to the **PASS** or **FAIL** results displayed.

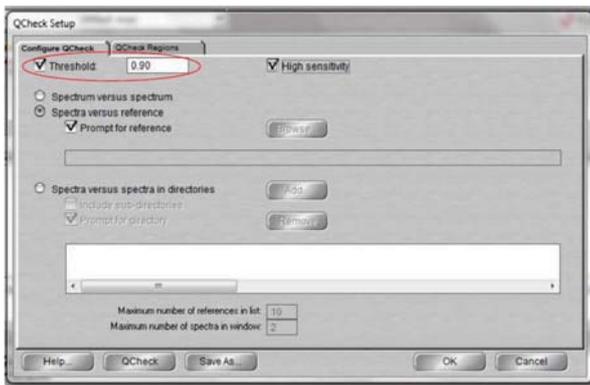


Figure 2 Setting the **threshold** to reject or to accept one sample using **High Sensitivity Compare** feature

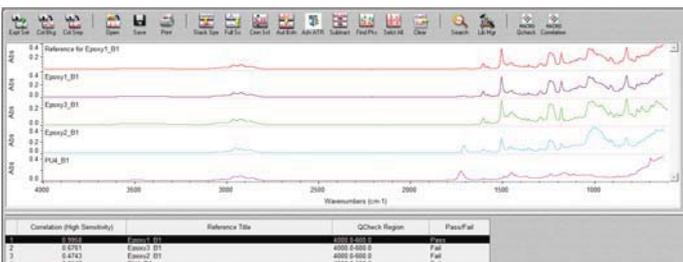


Figure 3 **PASS** or **FAIL** results displayed using **High Sensitivity Compare** feature by setting the **threshold** at 0.90

Comparing the inorganic fillers for polymer coatings

Titanium dioxide (TiO_2), the inorganic pigment, is commonly added into protective polymeric coatings. TiO_2 is commercially available in two crystal forms - **Anatase** and **Rutile**. The **Rutile** pigments are preferred over **Anatase** pigments for protective coatings, because they scatter light more efficiently, are more stable, and are less likely to catalyze photodegradation. **Rutile** is more expensive than **Anatase**. Hence, it may happen that the **Rutile** may be substituted by **Anatase** if reformulation of paints is attempted. Identification of **Anatase** or **Rutile** or the mixtures of both is often made by X-ray analysis (Test Method: ASTM D 3720 [5]), where the testing cost is rather high.

By using commercial FTIR software, we are able to compare **Rutile** and **Anatase** TiO_2 spectra as depicted in Figure 4. The quantity $r^{\text{High Sensitivity}} = 0.3$ (or $r^{\text{Normal Sensitivity}} = 0.6$) when **Rutile** is compared to **Anatase**.

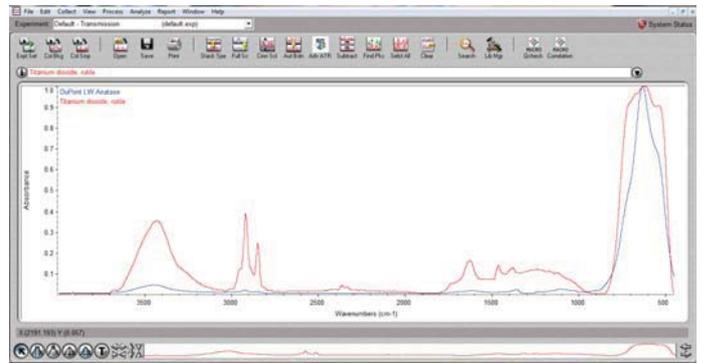


Figure 4 FTIR spectra extracted from the Polymer Library of OMNIC software for **Rutile** and **Anatase** TiO_2

Conclusion

We conclude here:

1. The structural analysis by FTIR for complete **Coating Fingerprint Certificate** for epoxy resin and hardener is simple and reproducible when **High Sensitivity Compare** feature of the FTIR software is to be strictly followed.
2. Rejection or acceptance of the samples can be easily done by setting the threshold value at 0.90 using **High Sensitivity Compare** feature of the FTIR software.
3. Comparing the inorganic fillers for polymer coatings (e.g. **Rutile** or **Anatase** of TiO_2) is also practical by using FTIR.

We note here again, this FTIR analysis shall not be limited to 2-component epoxy coating, but has to be extended to inorganic zinc coating, epoxy-zinc coating, polyurethane coating, acrylic coating, polyester coating etc. FTIR can be used to fingerprint all these types of coatings. Progressive reports of the FTIR fingerprinting studies on these types of coatings will be published in the forthcoming issues of *Materials Mind*.

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fingerprint regions for epoxy and hardener.

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Note: all the documents related to the background of "Coating Fingerprint Certificate" can be viewed at <http://www.iomm.org.my/v1/index.php/fingerprint>

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Biodata



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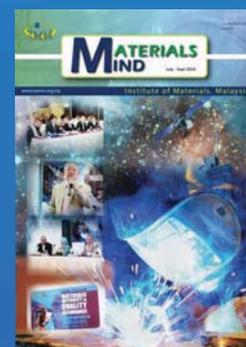
CONTENTS

IMM Council Members	4-6
International Journal of the Institute of Materials, Malaysia (IJIMM)	5
IMTCE2016 Promo Ads	8-11
IMTCE2016 Sponsorship Package	12-14
10 th International Materials Technology Conference & Exhibition (IMTCE2016) 1 st Organizing Committee Meeting	17
From Condom to Condominium	17
Serba Dinamik-IMM : Organizing the 1st Vibration Specialist Training Program Based on ISO14836	18
Report on: Materials Integrity and Quality Assurance Forum	19
Report on: SSPC Protective Coatings Inspector Program (Level 1 and Level 2)	20
Report on : IMM Task Force on Fingerprinting Polymer Coatings (Phase 1: 2013-2014)	21
Report on : IMM Task Force on Fingerprinting Polymer Coatings (Phase 2: 2014-2016)	22
Advertorial : DNV-GL	22
Introducing the Crew at MTE	23
Conclusion of IMM Task Force For Coatings Fingerprinting PHASE 1 (2013 -2014)	24
Technical Article 1 : Compare functions of FTIR software for structural analysis of epoxy paints on steel structures for coating fingerprinting Certificate	25-29

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