

Application News

No. A488

Spectrophotometric Analysis

Analysis of Inorganic Additives in Resin by FTIR and EDX

Additives enhance the quality of products by improving the functionality, workability, and stability of the materials used in the product. They are utilized in a wide range of products, including electronics, foods, pharmaceuticals, cosmetics, plastics, etc., and play an important role by adding value to products.

The approach taken in the analysis of additives is dependent on whether the additive is organic or inorganic. Organic additives are typically identified by first extracting the additives using a suitable pretreatment procedure, and after chromatographic separation of the extracted components, a suitable analytical instrument is used for qualitative analysis. On the other hand, comprehensive identification of inorganic additive components is typically based on the results obtained using elemental analysis, infrared spectroscopy or morphologic observation, etc¹⁾.

Here, we introduce examples of FTIR analysis to obtain useful information regarding some typical inorganic additives, in addition to an example of analysis of a resin containing an inorganic additive using FTIR and EDX.

■ Analysis of Inorganic Additives by FTIR

FTIR is used mainly for organic analysis, but useful information can also be obtained by applying FTIR to the analysis of some inorganic additives.

Here, we conducted single-reflection ATR measurement using a diamond prism. Table 1 shows the analytical conditions using FTIR, and the infrared spectra and peak positions of the four substances used as additives (aluminum silicate, aluminum hydroxide, magnesium silicate, and calcium carbonate) are shown in Fig. 1 to 4. A spectral characteristic of inorganic additives is the appearance of a relatively wide peak in the lower wavenumber region. In addition, as is clearly evident in Fig. 1 to 3, a characteristic peak is sometimes present in the higher wavenumber region. In such cases, qualitative identification is possible by FTIR alone.

Table 1 Analytical Conditions

Instruments	: IR Tracer-100, Quest, Diamond
Resolution	: 4.0 cm ⁻¹
Accumulation	: 40
Apodization	: Happ-Genzel
Detector	: DLATGS

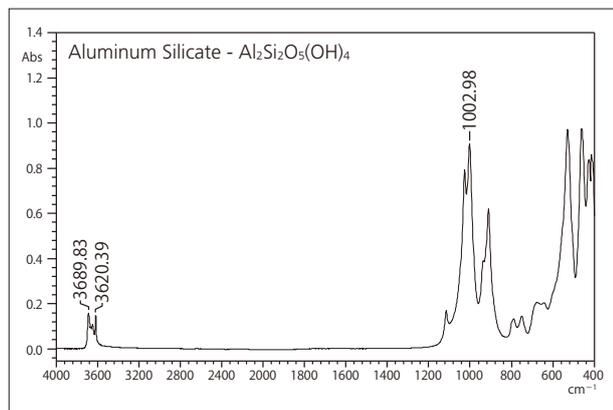


Fig. 1 IR Spectrum and Peak Position of $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$

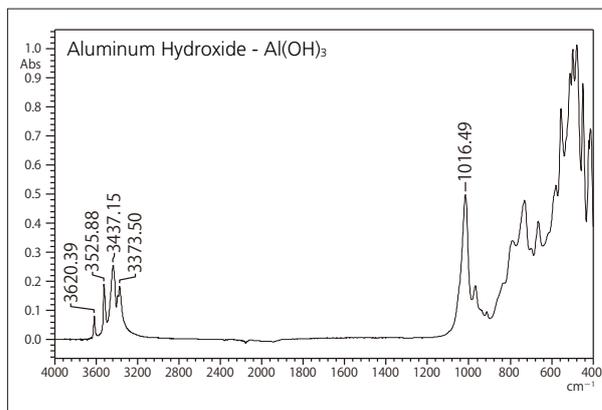


Fig. 2 IR Spectrum and Peak Position of $\text{Al}(\text{OH})_3$

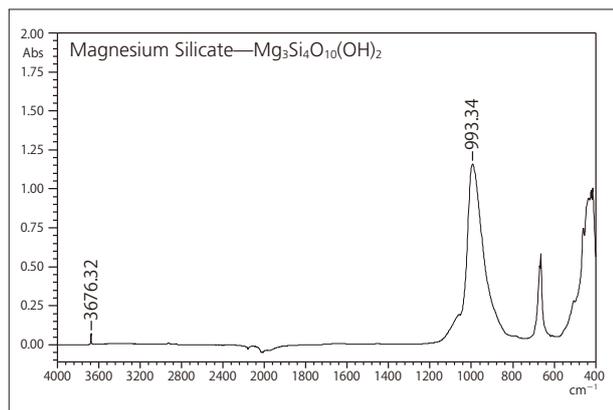


Fig. 3 IR Spectrum and Peak Position of $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$

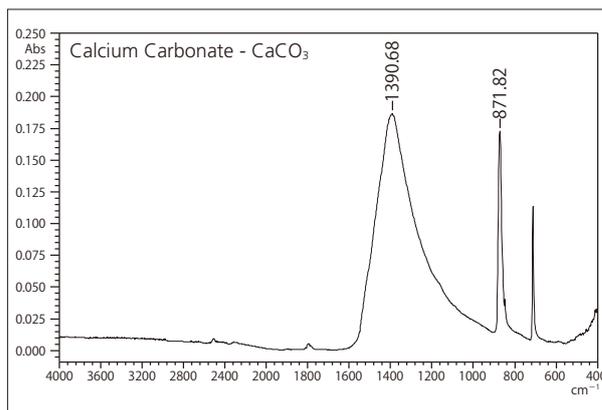


Fig. 4 IR Spectrum and Peak Position of CaCO_3

■ Analysis of Inorganic Additives in Resin

FTIR was used for the analysis of a connector cover as a sample that contains an inorganic additive. Fig. 5 shows a photograph of the sample, and Fig. 6 shows the results of analysis.



Fig. 5 Connector Cover

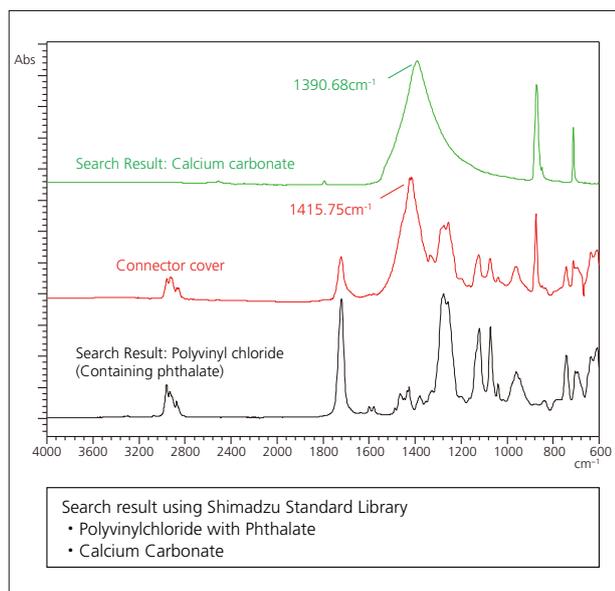


Fig. 6 IR Spectra and FTIR Search Results

From the infrared spectra and search results of Fig. 6, the principal component of the connector cover was determined to be polyvinylchloride (PVC). Also, the peak in the vicinity of 1415 cm^{-1} in the infrared spectrum suggests the presence of calcium carbonate (CaCO_3).

However, a comparison of the connector cover peak at 1415 cm^{-1} with the peak at 1390 cm^{-1} of calcium carbonate alone indicates a peak position shift of 25 cm^{-1} . Therefore, there is clearly insufficient basis to irrefutably conclude that calcium carbonate is included as an additive from the infrared spectrum alone.

Thus, we followed up this result by conducting EDX analysis to reconcile this discrepancy. The analytical conditions used are shown in Table 2, and the qualitative analytical results are shown in Fig. 7. Table 3-1 and Table 3-2 show the quantitative analytical results by the FP method²⁾.

Table 2 EDX Analytical Conditions

Instrument	: EDX-7000
X-ray Tube	: Rh target
Voltage / Current	: 15 kV(Na-Sc), 50 kV(Ti-U) / Auto
Atmosphere	: Vacuum
Measurement Diameter	: 10 mmφ
Integration Time	: 100 sec

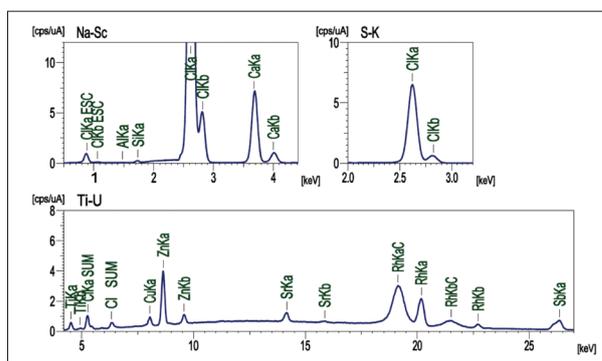


Fig. 7 Results of Qualitative Analysis of Connector Cover by EDX

Table 3-1 Results A of Quantitative Analysis of Connector Cover by EDX

Element	Cl	Ca	Sb	Zn	Ti	Si	Al	Cu	Sr
Quantitation Value	72.56	26.11	0.40	0.31	0.23	0.21	0.096	0.054	0.030

Table 3-2 Results B of Quantitative Analysis of Connector Cover by EDX

Element	$\text{C}_2\text{H}_3\text{Cl}$	CaCO_3	SiO_2	Sb_2O_3	TiO_2	ZnO	Al_2O_3	CuO	SrO
Quantitation Value	73.14	25.55	0.31	0.30	0.25	0.25	0.13	0.046	0.024

As clearly indicated in Table 3-1, chlorine (Cl) and calcium (Ca) are the principal constituent elements. This is consistent with the polyvinyl chloride results obtained by FTIR, supporting the presence of calcium carbonate. Table 3-2 shows the quantitative analytical results for specific compounds identified from the results of both FTIR and EDX. It should be noted that other detected elements are assumed to be oxides³⁾. Thus, the combination of FTIR and EDX has provided sufficient evidence that calcium carbonate is present as an additive.

■ Conclusion

By applying a combination of FTIR and EDX, we were able to more accurately identify the additives included in an actual sample. Such an analysis is applicable to contaminant analysis and confirmation testing, and should be considered an effective means for conducting such confirmation testing in a wide range of fields, including electrical and electronic, chemical, pharmaceutical, and foods, etc.

- 1) Toshikatsu Nishioka, Tatsuya Housaki: (2011) "A Guide on Plastic Analysis," Maruzen Publishing Co., Ltd.
- 2) Hiroto Ochi, Hideo Okashita: Shimadzu Review, 45 (1-2), 51 (1988)
- 3) Sachio Murakami, et.al: Shimadzu Review, 69 (1-2), 133 (2012)