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Application News

No.X261

X-ray Analysis

Contaminant Analysis in Food Manufacturing Process by EDX and FTIR

EDX and FTIR are widely used for analysis of foreign contaminant matter, but recently, these instruments are increasingly being utilized in tandem to conduct contaminant analysis[1]. While identification using any of these instruments and analytical methods independently is limited to some degree, using them in conjunction with one another permits a more detailed elucidation of the contaminant characteristics, thereby enhancing the validity of the respective results.
The analytical method and sample pretreatment method to be used depend on the degree to which a contaminant is to be characterized, whether or not the substance is altered or destroyed due to pretreatment, and the speed that is required to complete the analysis. Introduced here is an example of actual analysis of various types of foreign matter entered during the food manufacturing process.

Samples

Foreign matter that entered during the food manufacturing process Five types of samples: Sample 1, 2, 3, 4, 5

Pretreatment and Analysis Procedures

First, EDX measurement was conducted without conducting any sample pretreatment, and then FTIR measurement was conducted similarly without pretreatment. Next, the foreign matter was removed by rinsing, and then analyzed. This preparation procedure is outlined in the flowchart of Fig. 1.
Depending on the sample, there may be cases in which detailed analysis by ATR measurement using the FTIR main unit will be difficult due to such factors as small sample size relative to the prism, which could result in the sample being crushed, such as in the current situation, or samples consisting of a mixture, etc. It was therefore decided to conduct microscopic ATR measurement with close contact of the prism at the measurement site.

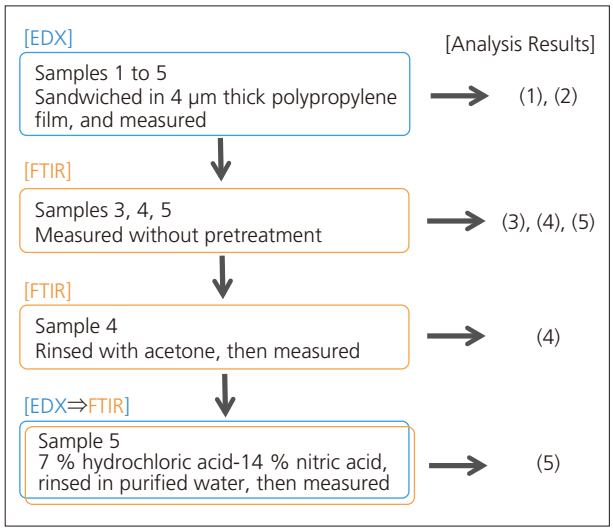
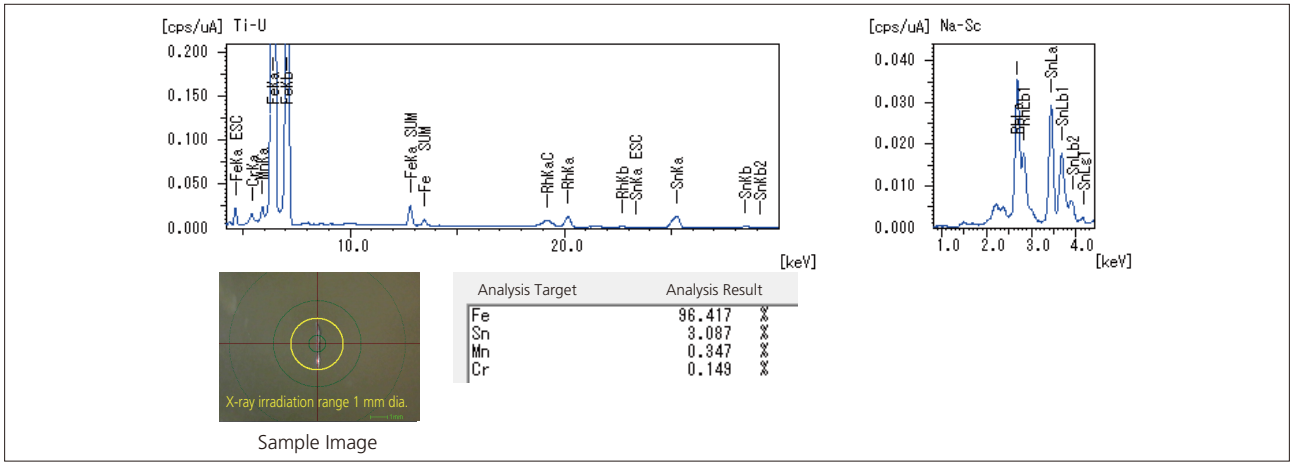


Fig. 1 Pretreatment and Analysis Procedures

Analysis Result

Fig. 2 to Fig. 9 and Table 1 to Table 5 show the analysis results for each sample using EDX and FTIR, in addition to the inferred and specific attributions according to those results.

(1) Sample 1 Characteristics: Metallic luster, hard, silvery white



Sample Image

Fig. 2 Sample 1 Qualitative-Quantitative Results by EDX

Table 1 Analysis Results for Sample 1

	Measurement Result	Possible Source	Total Findings Found by EDX and FTIR
EDX	Principal component is $_{26}$Fe, next prevalent is $_{50}$Sn.	Tin-plated steel sheet, fragment of tin can	Tin-plated steel sheet, fragment of tin can (Clearly metallic according to EDX measurement only)
FTIR	Omitted (Significant peak not detected)	Possibly a metal or inorganic compound	

(2) Sample 2 Characteristics: Metallic luster, hard, silver color

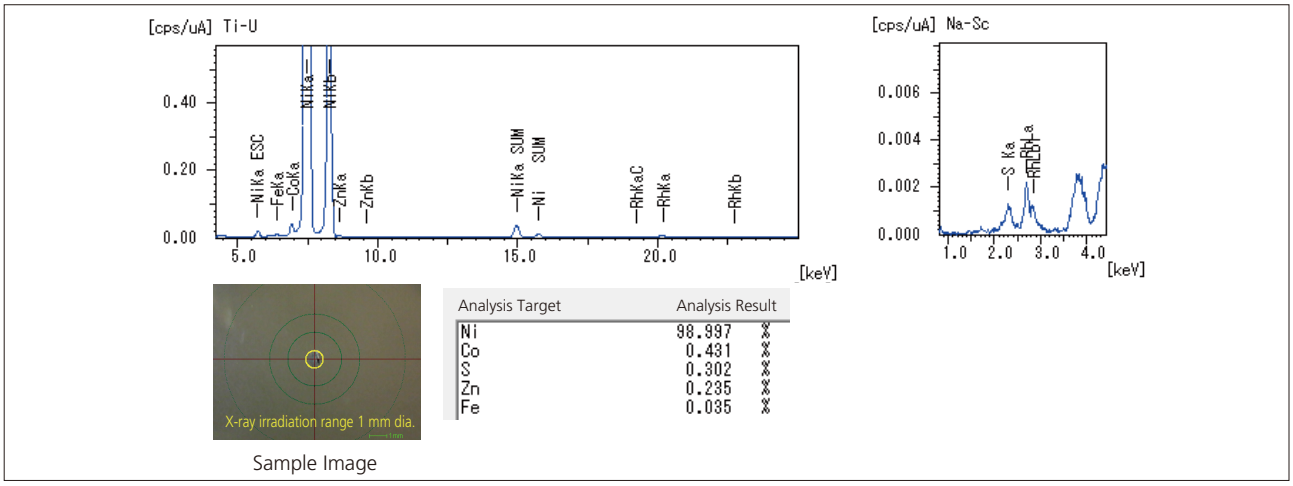


Fig. 3 Sample 2 Qualitative-Quantitative Results by EDX

Table 2 Analysis Results for Sample 2

	Measurement Result	Possible Source	Total Findings Found by EDX and FTIR
EDX	Principal component is $_{28}$Ni, other components are in small quantity.	Nickel, peeling of the nickel plating	Nickel, peeling of the nickel plating (Clearly metallic according to EDX measurement only)
FTIR	Omitted (Significant peak not detected)	Possibly a metal or inorganic compound	

(3) Sample 3 Characteristics: No metallic luster, brittle, brownish red

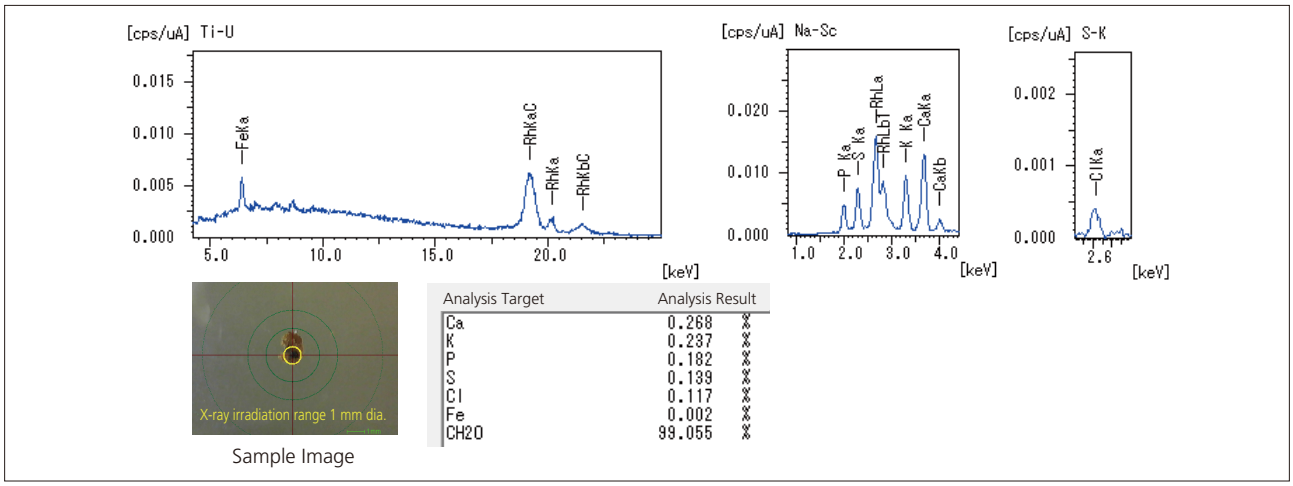


Fig. 4 Sample 3 Qualitative-Quantitative Results by EDX

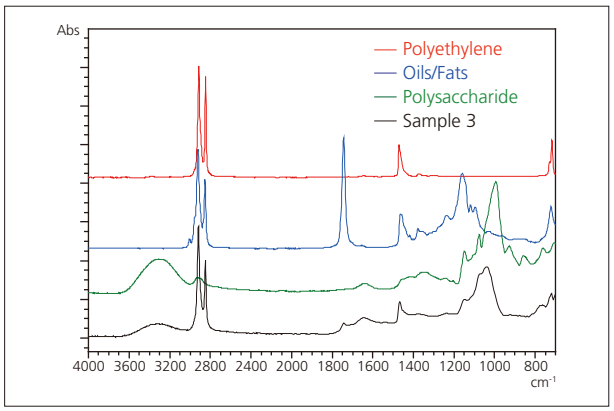


Fig. 5 Infrared Spectrum and Search Results for Sample 3 by FTIR

Table 3 Analysis Results for Sample 3

	Measurement Result	Possible Source	Total Findings Found by EDX and FTIR
EDX	Detected $_{19}$K, $_{20}$Ca, and other food components. Principal component is $_{9}$F and below. (RhKαC is big.[2])	Food clump	Polyethylene with attached food components
FTIR	Polyethylene, oils and fats, polysaccharides	Polyethylene with attached oils/fats and polysaccharides	

(4) Sample 4 Characteristics: Non-metallic luster, hard, black

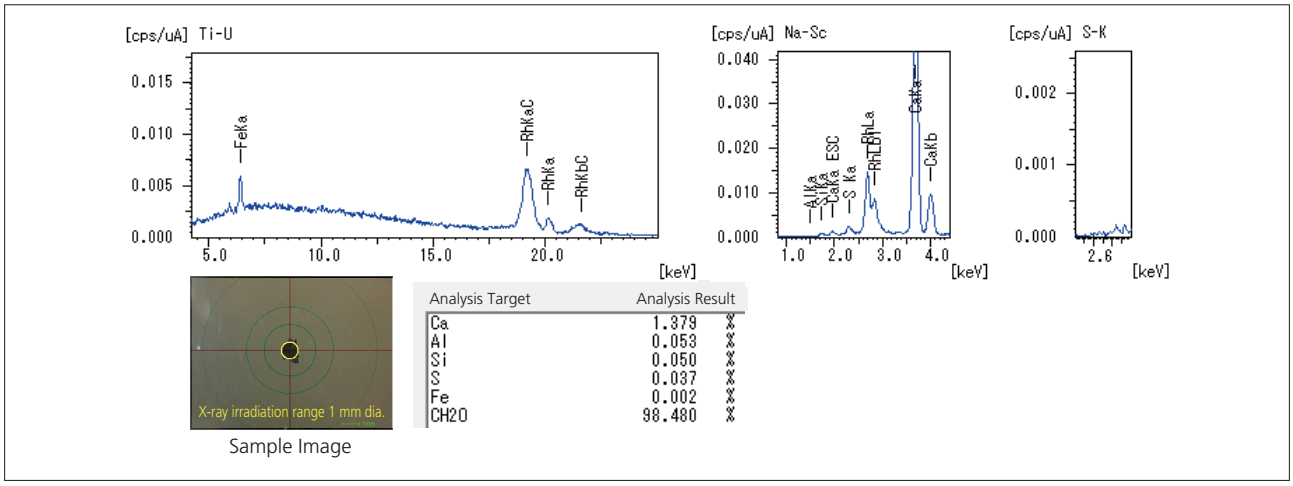


Fig. 6 Sample 4 Qualitative-Quantitative Results by EDX

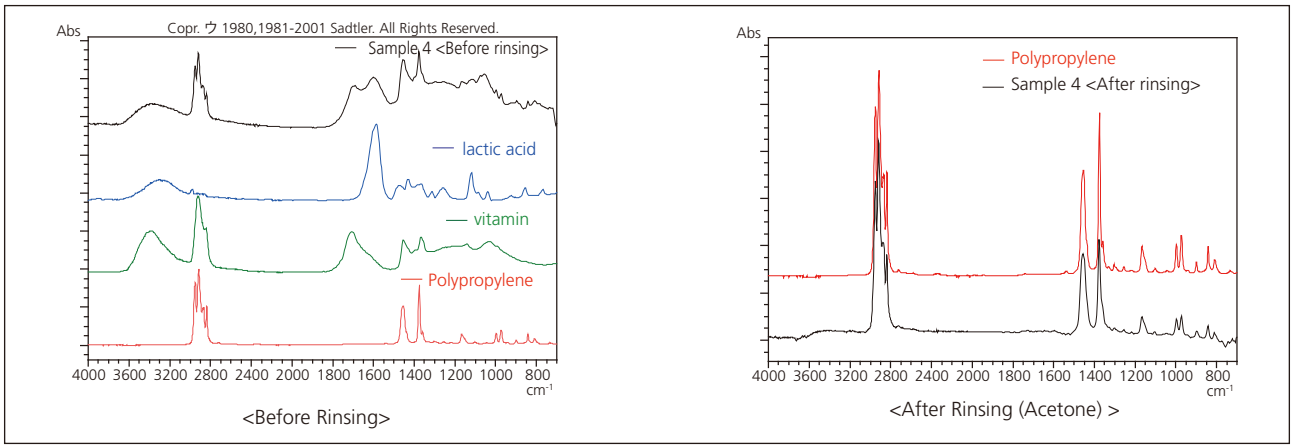


Fig. 7 Sample 4 Infrared Spectra and Search Results by FTIR

Table 4 Analysis Results for Sample 4

	Measurement Result		Possible Source	Total Findings Found by EDX and FTIR
EDX	Detected $_{20}$Ca and other food components. Principal component is $_{9}$F and below.		Food clump, resins, etc.	Polyethylene with attached food components
FTIR	Before rinsing	Polypropylene, lactic acid, vitamins	Food components (lactic acid, vitamins, etc.) adhering to polypropylene	
	After rinsing	Polypropylene		

(5) Sample 5 Characteristics: Some metallic luster, hard, black silver color

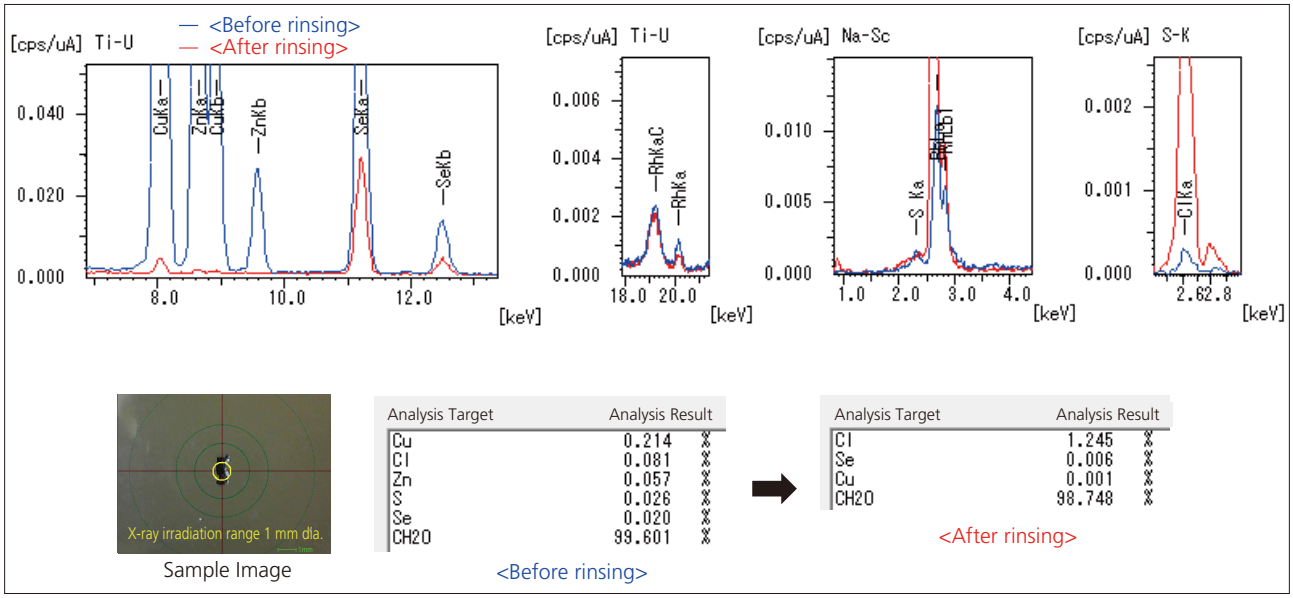
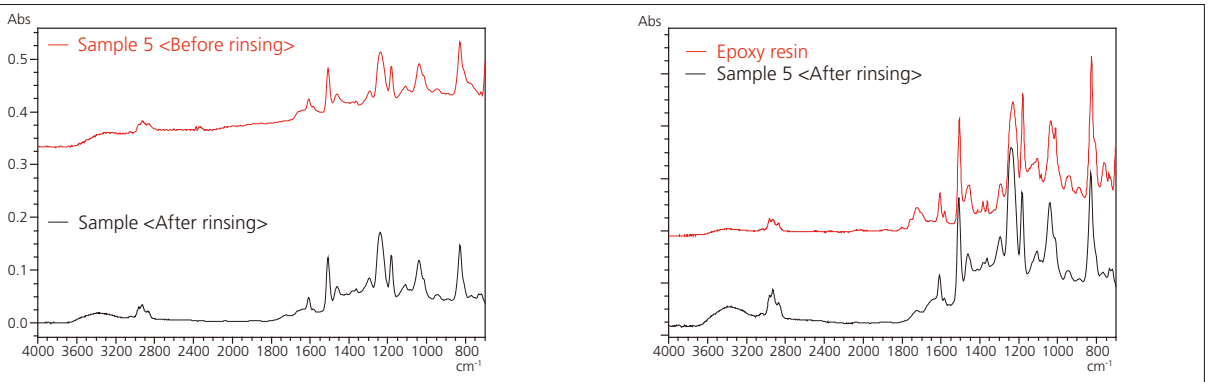


Fig. 8 Sample 5 Qualitative-Quantitative Result by EDX



<Before and After Rinsing (7 % hydrochloric acid-14 % nitric acid) >

<After Rinsing (7 % hydrochloric acid-14 % nitric acid) >

Fig. 9 Sample 5 Infrared Spectra and Search Result by FTIR

Table 5 Analysis Results for Sample 5

	Measurement Result		Possible Source	Total Findings Found by EDX and FTIR
EDX	Before rinsing	Principal components are $_{9}$F and below, large amounts of $_{29}$Cu, $_{30}$Zn, $_{34}$Se.	Copper alloy, resin composite material, zinc, selenium additives	Zinc and selenium food additives adhering to epoxy resin coated on copper thin film
	After rinsing	Principal components are $_{9}$F and below, with $_{29}$Cu, $_{30}$Zn nearly absent due to rinsing, and a small amount of residual $_{34}$Se.	Film	
FTIR	Before rinsing	Epoxy resin (with the presence of metals, etc. suggested due to rising of the infrared baseline)	Composite material consisting of epoxy resin and metal	
	After rinsing	Epoxy resin (no rise in the baseline in infrared spectrum)	Epoxy resin	

- Regarding the EDX quantitative analysis results
  · Organic material is represented by CH_2O, and was balanced.
  · Abundant, small quantity, etc. are relative reference values. (In order to collectively set plating, film and deposits, etc.)

Conclusion

The analysis results by both EDX and FTIR permitted approximate identification of metals, resins, and their compounds or complex materials associated with contaminants introduced during the food product manufacturing process without the need for pretreatment. Further, by conducting relatively simple pretreatment of samples, detailed identification is also possible depending on the sample. In terms of speed and ease, these analytical techniques are quite effective.

[References]

1) Shimadzu Application News No. A452
2) Izumi Nakai (Editor), A Practical Guide for X-ray Fluorescence Analysis, Asakura Publishing, 90 (2006)

Analytical Conditions [EDX]

Instrument	: EDX-7000
Elements	: Na-U
Analytical Group	: Qualitative-quantitative
Detector	: SDD
X-Ray Tube	: Rh target
Tube Voltage [kV]	: 15, 50
Current [μA]	: Auto
Collimator [mm ϕ]	: 1 or 3
Primary Filter	: Non, #2
Atmosphere	: Vacuum
Integration Time [sec]	: 50 /ch
Dead Time [%]	: Max. 30

Analytical Conditions [FTIR]

Instruments	: IRTracer-100, AIM-8800
Resolution	: 8 cm^{-1}
Accumulation	: 40
Apodization	: Sqr-Triangle
Detector	: MCT

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