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Application News

No. X274

X-Ray Analysis

Analysis of Food Contaminants by EDX and FTIR – Dental Materials –

Dental materials include diverse types of restorative materials such as metals, porcelain, ceramics, and composite resins. These materials are sometimes found in food during eating, for example, in case of inadequate adhesion with the tooth or loss of adhesion due to long-term use, and are mistakenly perceived as food contaminants. In many cases, a substance which is inadvertently mixed in a food product by a consumer is presented to the manufacturer as a contaminant originating from the production process, but claims can be held to the minimum by quickly conducting an analysis to clarify the source of the contaminant.

This article introduces an example of an analysis of a human tooth and various dental restorative materials that were presented to a food factory as contaminants by using EDX and FTIR.

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Measurement Instruments

The analyses were conducted using a Shimadzu EDX-8000 energy dispersive X-ray fluorescence spectrometer (EDX) and an AIM-9000 infrared microscope attached to an IRAffinity™-1S Fourier transform infrared spectrophotometer (FTIR). Table 1 shows the EDX and FTIR analysis conditions respectively.

Table 1 Instruments and Analysis Conditions

[EDX]	
Instrument	: EDX-8000
X-ray tube target	: Rh
Tube voltage/Tube current	: 15 kV (C-Sc, S-Ca), 50 kV (Ti-U) / Auto
Atmosphere	: Vacuum
Collimator	: 1 mmφ
Primary filter	: None (Ti-U, C-Sc), #2 (S-Ca)
Integral time	: 30 s (Ti-U, C-Sc), 60 s (S-Ca)
Sample container (Used film)	: Used (polypropylene film 5 μm)
[FTIR]	
Instruments	: IRAffinity-1S, AIM-9000
Resolution	: 8 cm^{-1}
Accumulation	: 40 times
Apodization function	: Sqr-Triangle
Detector	: MCT

Human Tooth

Fig. 1 shows a photograph of the contaminant, and Fig. 2 shows the results of a $_{6}C$-$_{92}U$ qualitative and quantitative analysis by EDX. Ca and P, which are the main components of teeth, could be confirmed. Although the element composition is similar to that of bone, the smaller content of Sr is a distinctive feature of teeth. Fig. 3 shows the results of a qualitative analysis by FTIR. Because the contaminant had a distorted shape and was extremely hard, a small amount of the sample was scraped off and a microscopic transmission measurement was conducted while holding sample in a diamond cell. The main component is calcium phosphate, and peaks originating from proteins can also be confirmed. The proteins are considered to be deposits on the tooth surface. When an adhering deposit exists, the sample is cleaned with water or ethanol, dried, and then measured. However, care is required, as there is a risk of losing the contaminant.

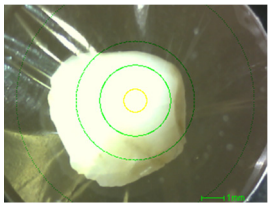
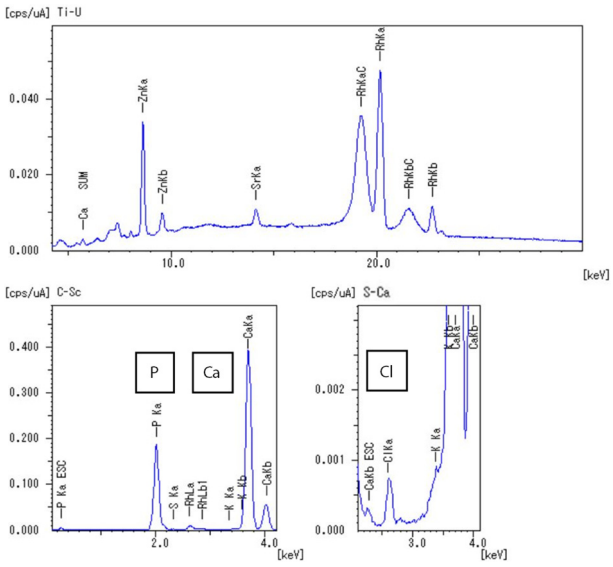


Fig. 1 Photograph of Contaminant (The Yellow Circle is the X-ray Irradiation Range: 1 mmφ)



Element	Ca	P	Cl	Zn
Quantitative value (%)	70.99	27.23	1.21	0.21
Element	K	S	Sr	
Quantitative value (%)	0.18	0.16	0.019	

Fig. 2 Qualitative and Quantitative Analysis Results: EDX

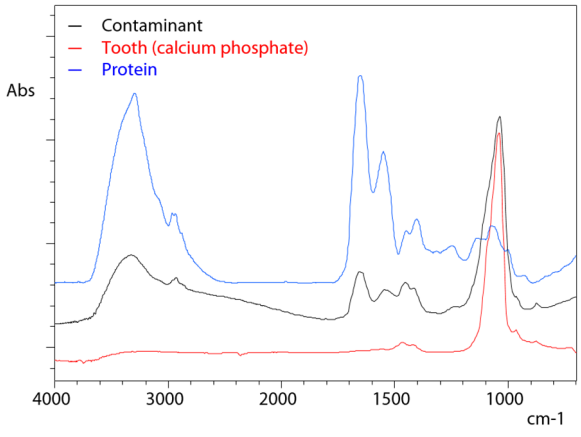


Fig. 3 Qualitative Analysis Results: FTIR

Artificial Tooth (Composite Resin)

Fig. 4 shows a photograph of the contaminant, and Fig. 5 shows the results of a $_{6}C$-$_{92}U$ qualitative and quantitative analysis by EDX. Because the main component is an organic material, a quantitative calculation was done assuming the balance is CH_2O. Fig. 6 shows the results of a qualitative analysis by FTIR. A small amount of the sample was scraped off and a microscopic transmission measurement was conducted while holding sample in a diamond cell. As the result of a library search, hits were obtained for the spectra of polymethyl methacrylate (PMMA) and polyethyl methacrylate (PEMA). Based on these results, the contaminant is considered to be an artificial tooth (composite resin).

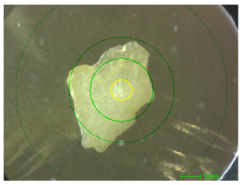
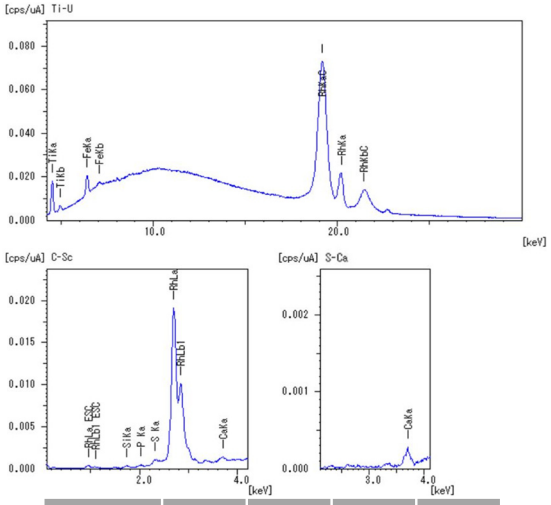


Fig. 4 Photograph of Contaminant (The Yellow Circle is the X-ray Irradiation Range: 1 mmφ)



Element	Ti	Si	S	Ca
Quantitative value (%)	0.032	0.023	0.017	0.013
Element	**P**	**Fe**	**CH_2O**	
Quantitative value (%)	0.011	0.006	99.90	

Fig. 5 Qualitative and Quantitative Analysis Results: EDX

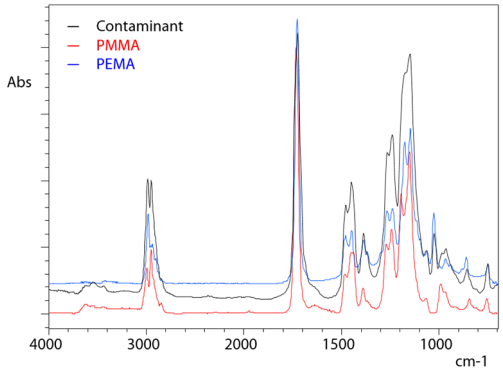


Fig. 6 Qualitative Analysis Results: FTIR

Artificial Tooth (Metal)

Fig. 7 shows a photograph of the contaminant, and Fig. 8 shows the results of a $_{6}C$-$_{92}U$ qualitative and quantitative analysis by EDX. The main components are Ag, Sn, and Zn. No significant peaks were detected in the FTIR analysis. Based on these results, the contaminant is thought to be an artificial tooth (metal tooth filling = silver crown).

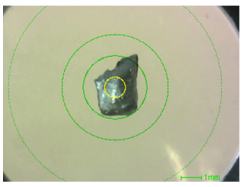
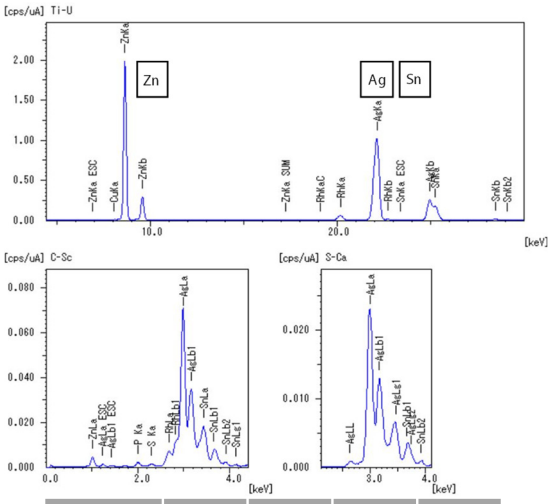


Fig. 7 Photograph of Contaminant (The Yellow Circle is the X-ray Irradiation Range: 1 mmφ)



Element	Ag	Sn	Zn	P
Quantitative value (%)	61.81	22.08	15.59	0.38
Element	**S**	**Cu**		
Quantitative value (%)	0.088	0.054		

Fig. 8 Qualitative and Quantitative Analysis Results: EDX

Conclusion

Contaminant in the form of a human tooth and various dental restorative materials were analyzed by using EDX and FTIR. Due to the diversity of dental materials, which include hybrid materials containing organic and inorganic components, metal materials, and others, a combined analysis technique using EDX and FTIR is extremely effective. Shimadzu Corporation also supplies the EDX-FTIR integrated analysis software EDXIR-Analysis™, which enables integrated analyses using both EDX and FTIR data. Use of the EDXIR-Analysis program in combination with the instruments mentioned in this article is recommended.

First Edition: Nov. 2019

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