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Application News

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Spectrophotometric Analysis

Analysis of Contaminant Adhering to Frozen Pizza

The steady stream of consumer complaints related to foods reflects continuing high concern for food safety. To address these concerns and specific contamination-related complaints, it is important to quickly and accurately report the analysis results and clearly elucidate the contamination pathway. Here, using a Fourier transform infrared spectrometer (FTIR) and an energy dispersive X-ray fluorescence spectrometer (EDX), in addition to an electron probe micro analyzer (EPMA), we present the results of analysis of a contaminant adhering to the surface of frozen pizza.

Photograph of Contaminant Adhering to Surface Frozen Pizza

The photograph in Fig. 2 shows the site of contamination on the frozen pizza. It was discovered when the package of this commercially available frozen pizza was opened. The foreign substance was subjected to complex analysis using a Fourier transform infrared spectrometer (FTIR), an energy dispersive X-ray fluorescence spectrometer (EDX), and an electron probe micro analyzer (EPMA).

Fig. 1 Photograph of Contaminant Adhering to Frozen Pizza

Analysis by FTIR

Some of the foreign substance was scraped off the frozen pizza, and after rolling it onto the diamond cell, the infrared spectrum was measured by transmission infrared microscopy. Measurements were conducted at multiple sites on the contaminant. Fig. 2 shows a photograph indicating the measurement locations, and Fig. 3 shows the overlaid infrared spectra that were obtained at the respective sites. Measurements were conducted using 30 × 30 µm focal regions. The analytical conditions used are shown in Table 1.

Table 1 Instruments and Analytical Conditions

Instruments	: IRPrestige-21, AIM-8800
Resolution	: 4 cm^{-1}
Accumulation	: 45
Apodization	: Happ-Genzel
Detector	: MCT

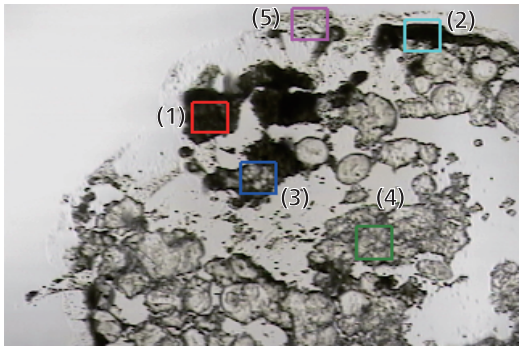

Fig. 2 Microscope Photograph of Measurement Sites

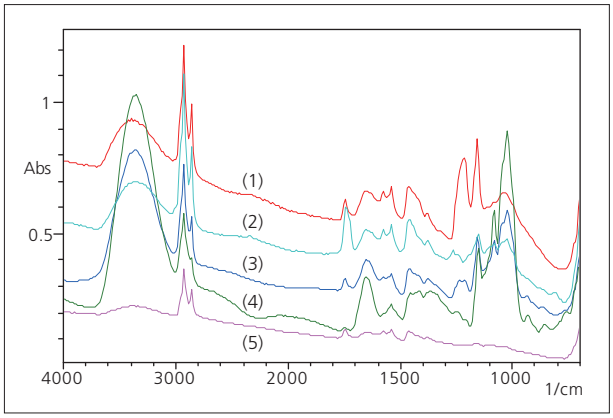

Fig. 3 Infrared Spectra of Contaminants

It is evident from Fig. 3 that the infrared spectra appear differently depending on the location. Among the spectra of Fig. 3, searches were conducted using the spectra (1), (4) and (5). The results are shown in Fig. 4 - Fig. 6.

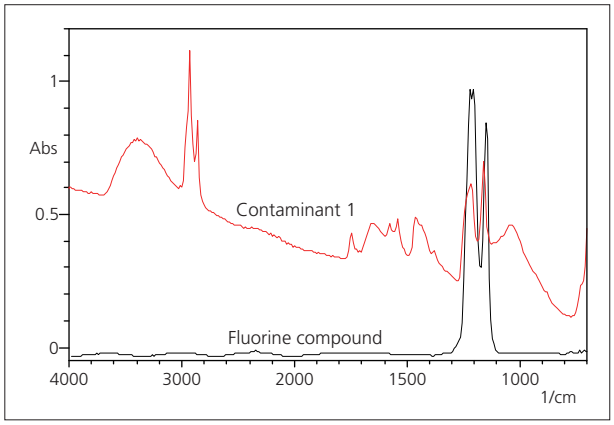

Fig. 4 Search Result 1

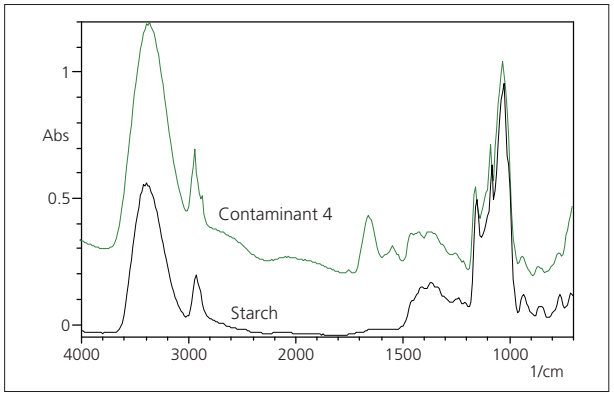

Fig. 5 Search Result 2

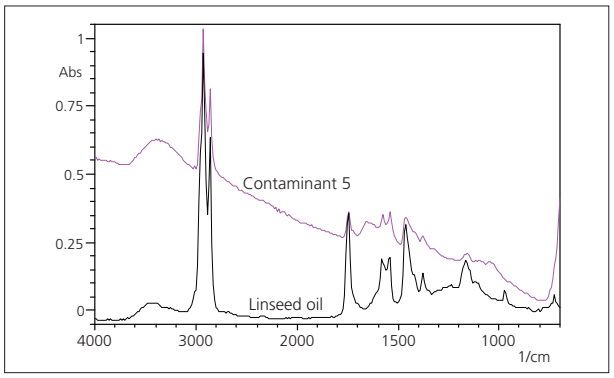

Fig. 6 Search Result 3

As can be seen from the respective search results, there were search hits on a fluorine compound, starch, and linseed oil. Of these, the starch and linseed oil are expected components of the pizza dough. The fluorine compound is an industrial product, and since it is also used in cooking utensils, it is apparently a contaminant derived from an external source.

Analysis by EDX

Analysis of the intact contaminant adhering to the frozen pizza was conducted using a 3 mm analysis diameter. Fig. 7 shows the qualitative results comparing the normal and contaminated parts of the frozen pizza. In addition, a comparative profile of the normal and contaminated sites of Fig. 7 was calculated, and quantitative analysis of the detected elements was conducted by the FP method. The obtained results are shown in Table 2.

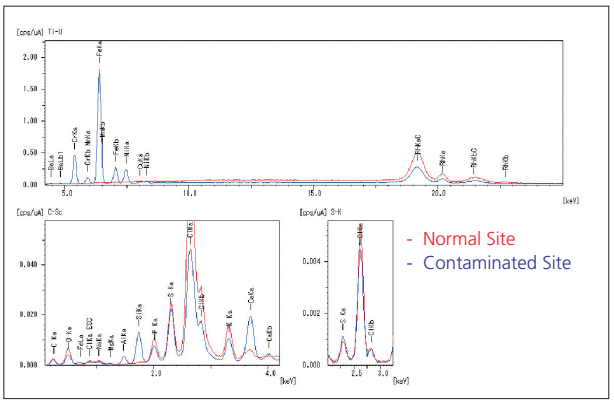

Fig. 7 $_{6}$C-$_{92}$U Qualitative Results for Contaminated and Normal Sites

Table 2 Quantitative Results for Contaminated Site by FP Method (%)

Fe	Cr	Ni	Si	Ca	Al	Ba	Mn
59.42	12.07	8.97	8.91	4.69	3.55	1.72	0.79

From the results shown in Fig. 7 in which Fe, Cr and Ni were detected in the contaminant as principal components, and considering the quantitative results, the contaminant is presumed to consist of stainless steel (SS). Further, since Al, Si, Ca and Ba were also detected, it is possible that a ceramic or other pigment-containing material may also be included. Since Na, P, S, Cl, K and Ca were also detected at the un-contaminated site, these are assumed to be of food origin. As for the F that is derived from the fluorine compound and was detected by FTIR, it was not clearly detected here, probably due to the small relative mass within the analysis radius and the overlap with the Fe$L\alpha$ line.

Analysis by EPMA

After scraping off a portion of the contaminant and coating its surface with gold, we conducted mapping of each element within a micro-region measuring 400 × 400 µm. Fig. 8 shows the mapping results for the principal elements that were detected.
The SS constituents Fe and Cr that were detected by EDX were detected over a wide range. In addition, the F that was detected by FTIR was detected in a localized perimeter. Thus, the results are consistent with the obtained FTIR and EDX results.

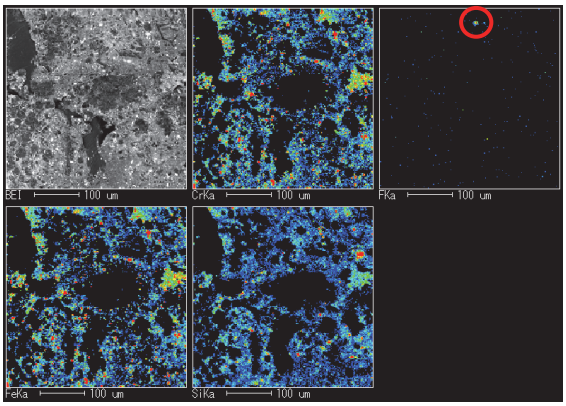

Fig. 8 Results of Elements Mapping for Contaminant Part

Conclusion

Complex qualitative analysis using FTIR, EDX and EPMA was conducted to identify a contaminant adhering to the surface of frozen pizza, and the results indicated detection of SS constituents and a fluorine compound. EPMA mapping results indicated that the SS constituents (Fe, Cr) were scattered over a region of approximately 10 µm.
On-site verification in the actual manufacturing process is essential to identify the source of contamination, but from the photograph of Fig. 1, the contaminant appears to be some type of burnt substance. The above analysis suggested that the contaminant consists of burnt cooking oil mixed with a fluorine compound originating from a cooking utensil or manufacturing machine, in addition to SS powder.

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