



Foreign Body Identification in Foods via FTIR and Handheld XRF

The inspection of foods to detect foreign materials is an integral part of quality assurance programs for food companies. Technologies such as metal detectors and X-ray scanners can detect small debris embedded in food products. Once detected, an accurate material identification of these foreign bodies is critical. The root cause of the contamination must be pinpointed and eliminated before that contamination goes further downstream on the processing lines, possibly leading to product recalls. Common forms of debris can include clear plastics that hold food items during shipping; other polymers and plastics that can be present in the manufacturing plant (e.g., gaskets); glass and/or various silicates; different types of metals used in process equipment; or materials that originate outside of the manufacturing facility.

A visual inspection of the debris in question starts the process of attempting to identify unknown particulates. Spectroscopic techniques that employ infrared (IR) and X-ray fluorescence (XRF) technology can generate rapid, accurate chemical identification of the unknown debris. As a result, these techniques aid in determining the root cause of the contamination, which in turn helps prevent large recalls and saves food manufacturers money. Most importantly, finding the root cause helps generate safer foods for the end consumer.

The purpose of this note is to demonstrate how Fourier transform infrared (FTIR) spectrometry and handheld X-ray fluorescence (HHXRF) spectrometry are complementary techniques for the rapid chemical identification of different types of foreign materials in food, including organics/plastics, rubber, ceramics, glass, and metals. The FTIR analysis presented in this note was performed with the Thermo Scientific™ Nicolet™ Summit™ X FTIR Spectrometer and the handheld XRF analysis was conducted with the Thermo Scientific™ Niton™ XL5 Plus Handheld XRF Analyzer (Figure 1).



Figure 1. (left) Nicolet Summit X FTIR Spectrometer with a Thermo Scientific™ Everest™ diamond attenuated total reflectance (ATR) accessory; and (right) Niton XL5 Plus Handheld XRF Analyzer.

The Nicolet Summit X spectrometer coupled with the Everest diamond ATR configuration is a robust FTIR system that can be set up on the factory floor, expediting data acquisition. ATR is the most common mode of data acquisition for material identification with the Nicolet Summit X spectrometer; it does not require any sample preparation. A sample is simply placed on top of an infrared inactive crystal, typically diamond, and a pressure tower is used to engage the sample onto the crystal. The pressure tower has a built-in slip clutch that controls the amount of torque that the crystal can receive, which prevents damage to the ATR accessory. Furthermore, due to the hardness and chemical inertness of diamond, hard, sharp, or rough samples (e.g., glass) as well as acidic or basic samples (in the pH range of 1-14) can be placed on the sampling area with no damage to the diamond crystal. Once a sample is engaged on the crystal, data acquisition can begin to generate an infrared spectrum of the unknown component.

Data acquisition of a single spectrum is typically done in seconds (circa 20 seconds) and after acquisition, that spectrum can be compared against a database of commercial libraries of known components for material identification. In the example shown in Figure 2, the clear unknown plastic sample that was analyzed on the Nicolet Summit X spectrometer appears to be a polyethylene-based material. Users can also generate their own spectral libraries from known material within their production or work environments, and those custom libraries can be searched to identify materials and contaminants as well. Identifying the sample material helps indicate where foreign debris could be coming from (e.g., inside the plant or from an external source), allowing the user to target the debris source and prevent further contamination.

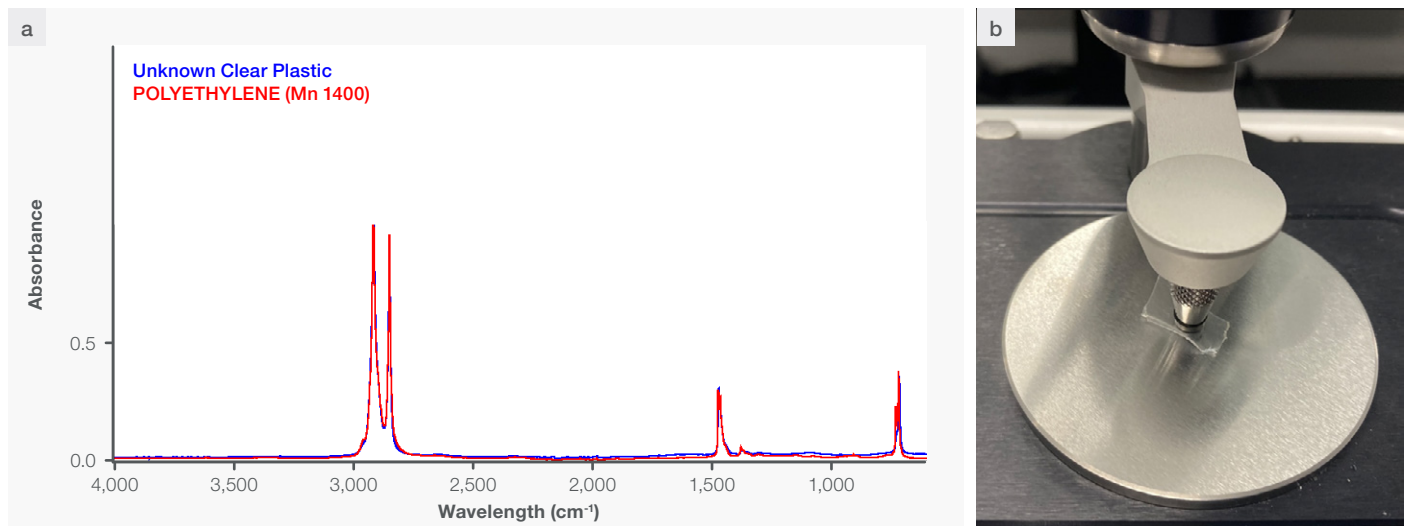


Figure 2. (a) Spectrum in Thermo Scientific™ OMNIC™ Paradigm software of unknown transparent plastic, overlaid with results of a library search, that generates a good match for polyethylene. (b) Example of a transparent plastic loaded onto the diamond ATR crystal for data acquisition.

In another experiment, two samples, one rubber and one metal, were analyzed on both the Nicolet Summit X FTIR spectrometer and the Niton XL5 Plus HHXRF analyzer; the results show why the techniques are complementary (Figure 3). For the unknown rubber sample, the FTIR spectrometer generated a workable spectrum (Figure 3a, blue curve) that the software searched against to identify it as a silicone-based rubber, in this case polydimethylsiloxane (PDMS) (Figure 3a, red curve). However, the FTIR spectrum for the stainless-steel sample had no peaks (Figure 3b) and was simply a flat line because metals are IR inactive. The Niton XL5 Plus analyzer is ideally suited to identify inorganic samples, especially metals and alloys such as stainless steel 316 (Figure 3d). While the XRF analyzer does not identify the plastic as silicone, it is able to detect elements such as silicon (Figure 3c) as well as additive metals present in the sample.

More germane to how FTIR and XRF are complementary techniques is the example that, if the unknown material was soda lime or borosilicate glass, the FTIR would generate a broad peak in the lower wavenumber region struggling with that material identification, while the XRF would be able to distinguish between those glasses based on elemental composition of silicon and calcium. The FTIR spectrometer can confirm organic-based materials, but it cannot discriminate among different metal alloys. However, the handheld XRF analyzer can distinguish metal alloys, glasses and minerals but delivers less information for polymer/rubber samples. Thus, HHXRF and FTIR measurements complement one another to identify both types of unknown materials.

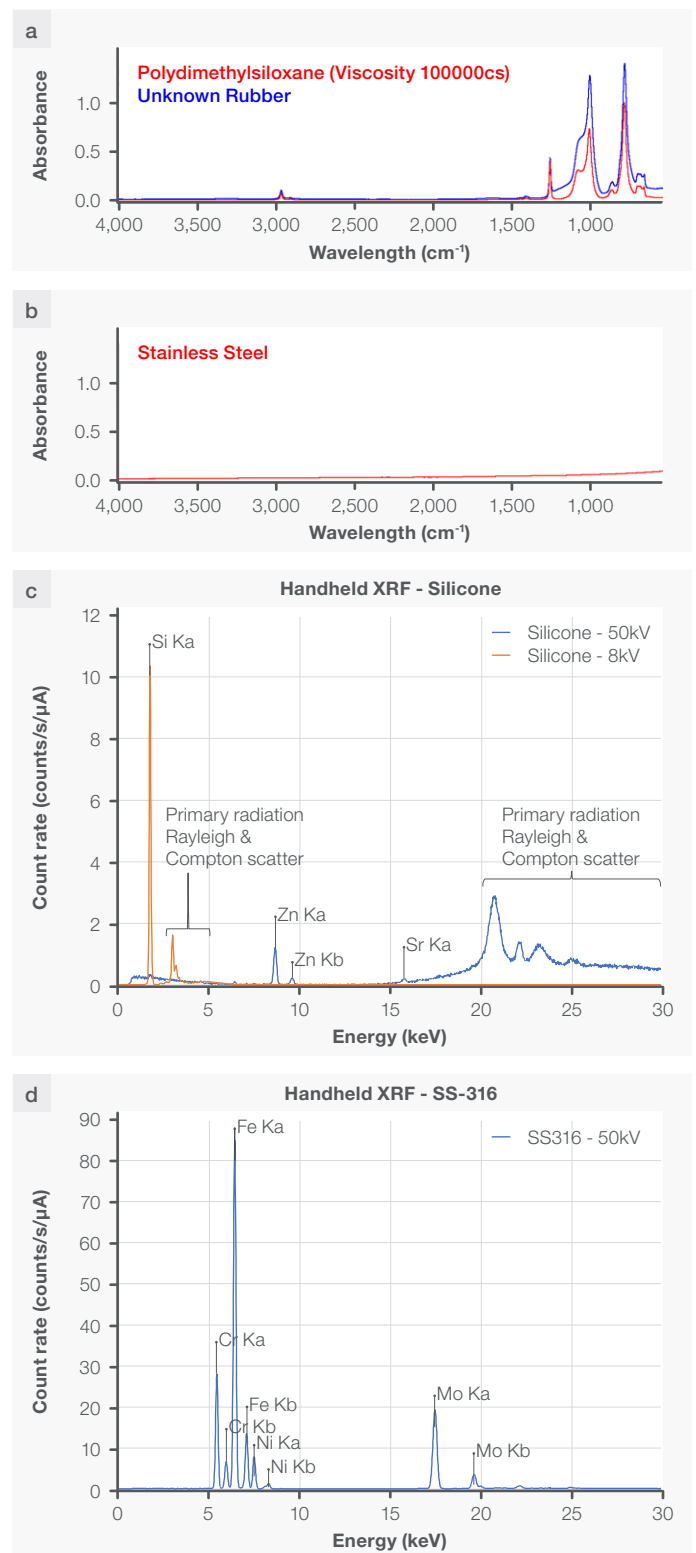


Figure 3. FTIR and XRF data on the same plastic and metal samples. The FTIR instrument can produce a reading on (a) silicone rubber but not on (b) stainless steel. The XRF analyzer detects (c) silicone and zinc in the rubber sample without being able to identify the type of rubber, but it does identify (d) the metal sample as stainless steel 316 grade.

The Niton XL5 Plus is a compact and lightweight (approx. 3 lbs.) handheld XRF analyzer that can be used directly on the production floor or in other parts of the plant. Modern HHXRF analyzers can detect and quantify elements with atomic numbers from 12 (magnesium) to 92 (uranium) in various types of materials such as metals, metal alloys, ceramics, glass, minerals, or plastics. With the elements being quantified, HHXRF can identify substances when comparing the composition of the measured sample with tabulated values of substances. This approach, called identification via grade table, is used to identify metals by comparing the measured composition with tabulated values for more than 500 standardized alloys including steel or stainless steel, as well as alloys of aluminum, copper, nickel, cobalt, titanium, tin, lead, or tungsten. Another approach to identify substances using HHXRF is comparing spectra of an unknown substance with spectra of known substances. This approach is called identification via spectral fingerprints. HHXRF analyses are generally completed within seconds for metal and alloy identification, to a few minutes for trace analyses. The user either aims the analyzer and depresses the trigger to analyze large specimens or uses a test stand (Figure 4a) to analyze small sized samples. The analysis is fully nondestructive, and results of analysis in units of mass concentrations are displayed in real time on the LCD screen of the analyzer (Figure 4b).

While handheld XRF can analyze various types of foreign bodies detected in foods, including glass, ceramics, stones, bones, or plastics, it will generally provide the most useful information in the case of metals for which it will both measure the concentration and identify the alloy grade. The workflow of foreign body identification (FBI) using HHXRF typically includes mapping and acquiring data from all parts of the processing line that might possibly contaminate products, starting with parts made of metal and eventually parts made of ceramics and/or glass. The user can compare the composition of foreign debris detected in food products with the composition of potential or suspected sources of contamination. This approach is efficient when different alloy grades are used in a processing line and will help the user to pinpoint the contamination within the plant or rule out an internal source of contamination. For example, debris from a sieve mesh made of stainless steel 316 can easily be differentiated from debris originating from a frozen food block flaker blade made of stainless steel grade 430.



Figure 4. (a) The Niton XL5 Plus Handheld XRF Analyzer with a sample loaded in test stand; (b) Live readout of stainless steel 316 and its elemental composition on the Niton XL5 Plus Analyzer.

When the same type of material, for example stainless steel 316, is used at multiple places in the production line, identification of foreign bodies made of this material via its composition or grade may not be specific enough to locate the source of contamination. Then, another approach using identification via spectral fingerprint is the preferable approach. In this case the user builds a library of spectra with the measurements of metallic and non-metallic materials acquired during the mapping process. Once the user has built out the custom library, the spectral fingerprint of contaminant samples can be compared to the library entries or spectra of the suspected source to more easily find and address the contamination source. If there is no match, then there is a probability that the contaminants were in the raw materials, so supplies should be thoroughly checked before entering the production process.

The use of custom libraries to help identify contamination sources proved useful in a case where a single piece of glass was discovered in a processed food product. The discovery prompted an investigation to identify the source of contamination. The production line was quickly ruled out as a potential cause. Subsequently, a thorough inspection of the vegetable raw materials revealed the presence of additional pieces of glass debris. Figure 5 illustrates that one of these pieces (represented by the grey curve) had a spectral fingerprint similar to the foreign body found in the processed food product (represented by the blue curve). However, the spectrum of the second glass fragment (represented by the orange curve) differed noticeably from that of the foreign body. This suggests that the contamination in the vegetable raw materials likely originated from multiple sources.

In addition to identifying contaminants, HHXRF enables maintenance teams at food processing plants to easily verify specifications for critical materials used in processing equipment such as auger conveyors, chopping blades, frozen food block flaker blades, mixing paddles, etc. to prevent failure of non-compliant equipment. A good illustration of this is the use of grade 304 instead of grade 316 stainless steel in a sieve's mesh. Grade 304 has a lower resistance to corrosion and, over time, presents a higher risk of failure and of contaminating foods containing salt such as sauces or soups.

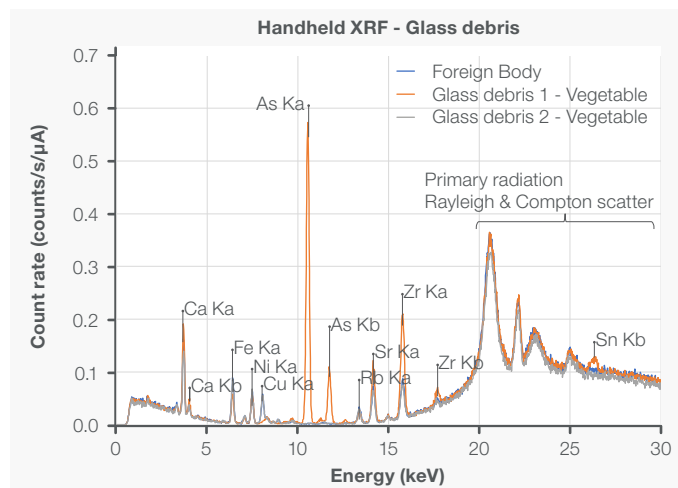


Figure 5. XRF spectra of a foreign body found in processed food and two pieces of glass debris subsequently detected in the vegetable used as raw materials. The foreign body (blue curve) has a similar spectral fingerprint to glass debris 2 (grey curve) but presents a noticeably different spectrum than glass debris 1 (orange curve) when looking at trace metals such as copper (Cu), arsenic (As), strontium (Sr), zirconium (Zr) or tin (Sn).

Conclusion

FTIR spectrometry and handheld XRF spectrometry are rapid, easy-to-deploy analytical techniques that provide great value for food processing plants. In enhanced quality assurance programs, metal detectors and X-ray inspection systems detect foreign materials, after which these analytical techniques maximize the user's ability to identify the origin of foreign body materials so corrective measures can be rapidly implemented. FTIR is readily suited to differentiate between different types of organic and polymer-based materials, while handheld XRF serves as a complementary analytical partner for the analysis of inorganic materials such as metal, glass, or ceramics. In addition to FBI, both techniques can provide food manufacturers with data to support assertions of quality or dispute false claims of contamination.

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