Identification of Ice Machine Filter Contaminants Using Attenuated Total Reflection - Fourier Transform Infrared Spectroscopy (ATR-FTIR)

Lauren Ostopowicz[1], Dominika Gruszecka[1],
[1] Shimadzu Scientific Instruments, Columbia, MD.

1. Introduction

Contamination of foods can occur at many points in production and processing. Sources can come from the growth cycle due to environmental factors, through harvest and storage with contaminated machinery or containers. Even when non-food-contact surfaces are affected, leachables and debris can cause other blockages resulting in maintenance needs, or food spoilage due to improper machine function. In this work, we used Attenuated Total Reflection (ATR), an FTIR technique, to identify the chemical composition of an unknown contamination found in food processing machinery resulting in such blockages. FTIR spectroscopy is widely used to reveal chemical information of various forms of samples. Vibrational modes characteristic of functional groups manifest as distinctive peaks in the infrared spectra, allowing the chemical identification of compounds.

Attenuated Total Reflection (ATR) is an FTIR technique that conveniently introduces the IR beam into solid, opaque, samples with little to no sample preparation required. Library searches of reference spectra can also be used to easily identify the unknown contamination without prior knowledge of infrared spectroscopy data.

2. Methods

Two filter samples were submitted for analysis by the IRXross spectrometer. Figure 1 shows an image of the samples with their corresponding sample numbers used throughout this work. On visual inspection, it is evident there is a film formed over the filter which contributed to machine malfunction.

Fig. 1 Picture of the contaminated filter samples. Samples are referred to as "sample 1" (left) and "sample 2" (right) in this report.

Each sample is placed and secured on the ATR crystal with the constant pressure anvil from above as shown in Figure 2. A closer image is representative of the sample during analysis. Experimental parameters for the FTIR spectrometer are listed in Table 1. The ATR and anvil were cleaned with methanol between measurements to ensure residue did not transfer to the next measurement. After spectral collection, comparison searches are completed within Wiley's KnowItAll™ software, containing about 300,000 FTIR spectra.

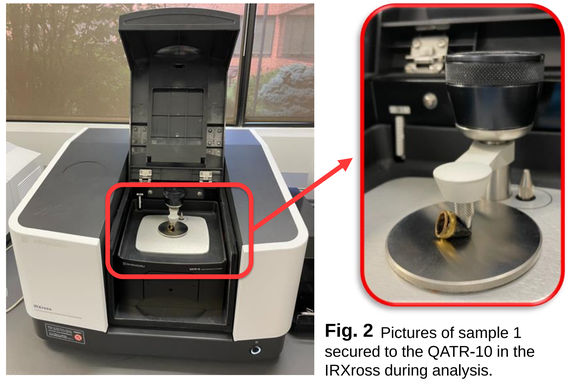

Fig. 2 Pictures of sample 1 secured to the QATR-10 in the IRXross during analysis.

Table 1 IRXross experimental parameter settings

Parameter	Value
Spectral Range	4000-400 cm^{-1}
Resolution	4 cm^{-1}
Number of Scans	56
Measuring Mode	Absorbance
Optical Mode	ATR
Detector	DLATGS
Model	IRXross (QATR-10)

3. Results

All spectra were compared against Wiley's extensive FTIR libraries using KnowItAll™ software to identify each contaminant. To improve the searching algorithm, an automatic ATR correction was performed on the collected data in KnowItAll™ software to correct for known but minor spectral artifacts introduced when using ATR sampling. Sample 1 matches FORLAN LM with 92% statistical confidence and great visual spectral overlap. FORLAN LM is a proprietary formulation made by R.I.T.A. Corporation consisting of a mixture of hydrogenated oils and alcohols. It is a lubricant, emulsifier and emollient used in cosmetic formulations.

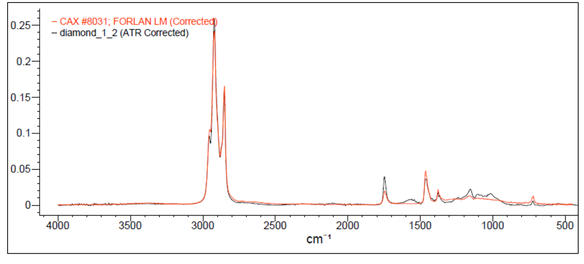

Fig. 3 ATR Corrected FTIR spectrum of sample 1 (black trace) overlaid with the top library search result, Forlan LM (orange trace).

Table 2 Sample 1 library search result summary table

Name	Value
Spectral Match Confidence	92.1 %
Search Result Name	FORLAN LM
Chemical Description of Search Result	A mixture of hydrogenated lanolin, hydrogenated coconut oil, sorbitan, sesquioleate, strearyl alcohol, cetyl alcohol
Source of Result Spectrum	R.I.T.A. Corp.
Spectral Database Name	IR- Sadtler Surfactants (Wiley)

The searching algorithm tends to fit noise fluctuations as real peaks, leading to incorrectly assigned spectral matches. To properly guide the searching algorithm and avoid misrepresenting noise as data of sample 2, the grey areas shown on Figure 4 were excluded from the search. The major peaks in the spectrum result in a match with 85% confidence to a corrosive-protection agent. The small peak at about 800 cm^{-1} circled in blue was not accounted for in the match, and it is a characteristic peak of siloxane group. Based on the size of the peak, there is small concentration of that functional group present in the sample. Interestingly, the lubricant used in the machine was confirmed to be dimethylpolysiloxane; it is probable that a small amount of this lubricant is present in sample 2 in addition to the ones reported in the match.

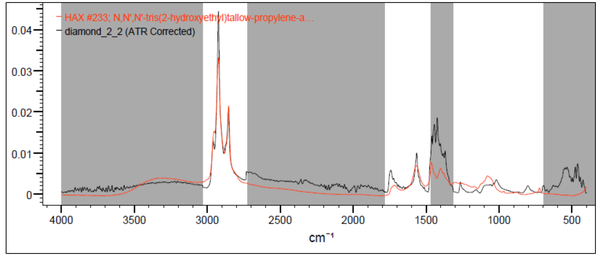

Fig. 4 ATR Corrected FTIR spectrum of sample 2 (black trace) overlaid with the top library search result, N,N',N'-tris(-hydroxylethyl) tallow-propylene-ammonium caprylate (orange trace).

Table 3 Sample 1 library search result summary table

Name	Value
Spectral Match Confidence	85.4 %
Search Result Name	N,N',N'-tris(-hydroxylethyl)tallow-propylene-ammonium caprylate
Chemical Description of Search Result	A corrosive-protection agent for mineral oils and boiler feeder water installations
Source of Result Spectrum	Rewo Chemische Werke
Spectral Database Name	IR- Surfactants, Hummel (Wiley)

4. Conclusion

The search results revealed that sample 1 is likely a hydrogenated oil and alcohol formulation, and sample 2 is most likely a corrosion protection agent for mineral oils and boiler feeder water installations. Shimadzu's IRXross spectrometer and QATR-10 accessory with Wiley's KnowItAll™ library searching software quickly determined the composition of the filter contaminants without the need for sample preparation. This allowed the customer to determine the source of the contamination and solve the problem before impacting future shipments of ice machines.