DIAMAXATR ACESSORY **NO. 21161**

Investigating a Saponification Reaction Using the DiaMaxATR™

Figure 1. The DiaMaxATR.

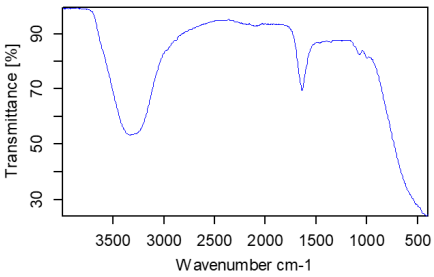

Figure 2. ATR spectrum of oven cleaner.

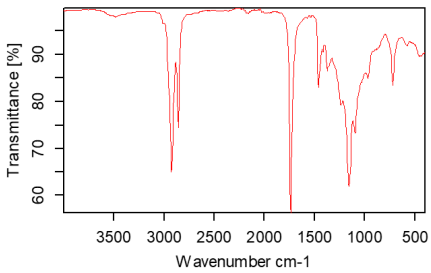

Figure 3. ATR spectrum of burned sun flower oil.

INTRODUCTION

Diamond ATR is a useful tool in analyzing reactions with certain types of corrosive samples, such as harsh cleaning agents. Manufacturers in this industry can study potential unwanted by-products as well as the effectiveness of the cleaning agent.

In this note, a saponification reaction was examined using oven cleaner and burned sunflower by diamond ATR spectroscopy.

EXPERIMENTAL

Infrared spectra were collected on an FT-IR spectrometer equipped with the Harrick DiaMaxATR™ single-reflection high throughput diamond ATR accessory. Spectra were collected at an 8 cm^{-1} resolution, a gain of 1, and signal averaged over 32 scans. The aperture was set to 100%. Spectra were collected in the range 4000-400 cm^{-1}. The instrument was purged to reduce water vapor and CO_2 interferences.

The samples used were EasyOff Heavy Duty Oven Cleaner and Sunflower oil (H&B Oils Center Co.). Approximately 14 ml of sunflower oil was placed in a glass container and was heated in an oven at 260 °C for 2 hours. A small piece of the burned sunflower oil film was placed on the diamond. The film was pressed against the diamond ATR crystal using the pressure applicator. After a spectrum was taken of the burned sunflower oil film, the pressure applicator was raised, and two drops of oven cleaner was deposited on the film. The pressure applicator was used to keep the film firmly pressed against the crystal. Spectra were collected every 30 seconds for a total of 45 minutes using Harrick's TempLink software.

RESULTS AND DISCUSSION

Although the active ingredient in oven cleaner is sodium hydroxide, which contains an –OH functional group, it is clear from the characteristic broad band at 3333 cm^{-1} and the band at 1638 cm^{-1} (Figure 2) that the active ingredient in the oven cleaner is diluted with water.

Figure 3 is the burned sunflower oil film sample, which shows a weak broad band present in the 3400-3500 cm^{-1} spectral range, which indicates O-H stretching. There are two peaks present in the 3000-2850 cm^{-1}

spectral range, 2922 cm^{-1} and 2853 cm^{-1}, which are due to C-H stretch vibrations. A prominent peak found at 1740 cm^{-1} is likely attributed to the ester carbonyl group found in triglycerides in the sunflower oil.

The active ingredient in oven cleaner, sodium hydroxide, hydrolyzes the triglycerides, producing carboxylate salt and an alcohol, known as a saponification reaction. Figure 4 illustrates the changes upon the introduction of oven cleaner to burned sunflower oil at the beginning of the reaction and at the final stage, 45 minutes later, in addition to burned sunflower oil before oven cleaner was introduced. As time passes, the O-H band in the 3400-3500 cm^{-1} spectral range shifts from 3377 cm^{-1} to 3384 cm^{-1} and broadens initially before decreasing in intensity, which is likely due to water evaporating from the oven cleaner. The presence of an alcohol is at 45 minutes as a result of sodium hydroxide hydrolyzing the triglycerides. There are no significant changes to the C-H stretch peaks in the 3000-2850 cm^{-1} spectral range. The ester carbonyl group at 1740 cm^{-1} decreased upon the introduction of oven cleaner, and a new peak appeared at 1558 cm^{-1}, which is likely the presence of carboxylate salt of fatty acids. The peak at 1558 cm^{-1} increased in intensity as the reaction progressed, which indicates a saponification reaction likely occurred either partially or fully.

In Figure 5, a spectrum of burned sunflower oil and oven cleaner mixture at 45 minutes and a spectrum of oven cleaner drying at 45 minutes was compared to confirm whether a saponification reaction occurred or if the oven cleaner drying concentrated the ingredients previously obscured by water. As the oven cleaner dries, the broad –OH band shifts to a lower wavenumber and a few peaks appear in the C-H stretch region, indicating an increase in the concentration of the ingredients as the water evaporated. Note that there is no peak at 1558 cm^{-1} after the oven cleaner dried, indicating that this new peak that appeared after oven cleaner was deposited on burned sun flower oil is likely carboxylate salt of fatty acids, a product of the reaction between oven cleaner and burned sunflower oil.

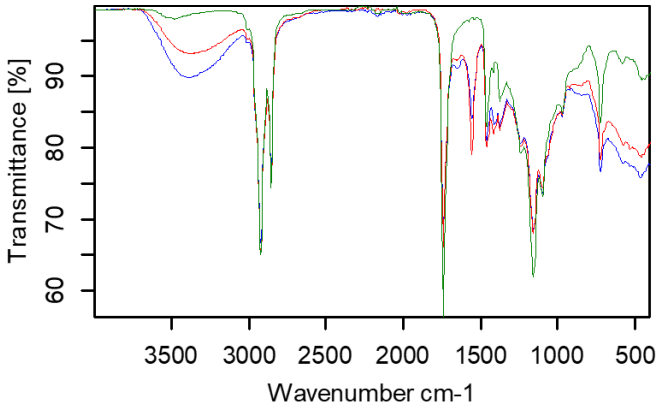

Figure 4. ATR spectra of burned sunflower oil (green), burned sunflower oil and oven cleaner mixture at 0 minutes (blue), burned sunflower oil and oven cleaner mixture at 45 minutes (red).

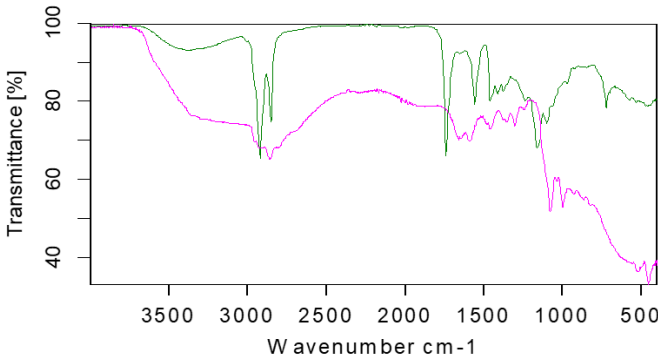

Figure 5. A comparison of the ATR spectra of burned sunflower oil and oven cleaner mixture at 45 minutes (green) and oven cleaner drying at 45 minutes (pink).

CONCLUSION

The DiaMaxATR accessory is extremely useful in analyzing samples such as harsh and corrosive cleaning agents, such as oven cleaner and its reaction with potential contact materials, such as oils, grease, and other burned food products found on oven surfaces.