Specac

Application Note

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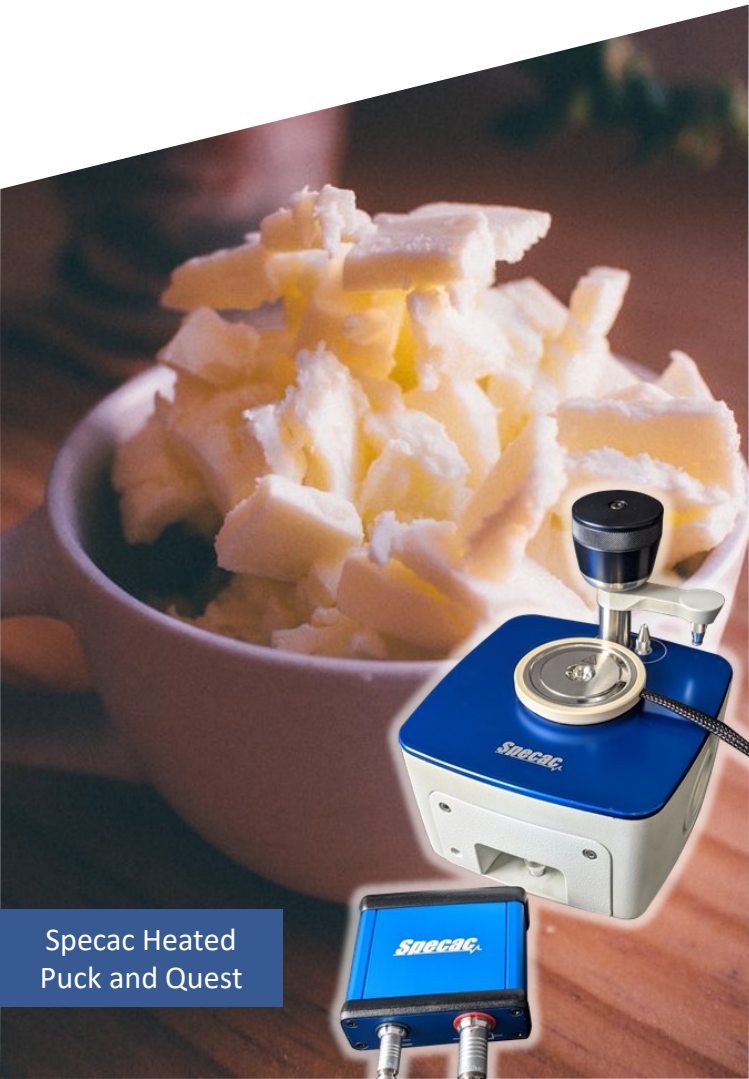
Separation of butter into phases with the Quest™ heated puck



Specac Heated Puck and Quest

Inside: Find out how the heated puck can be used to investigate the separation of butter when heated.

Introduction

Milk is a colloidal mixture of water and fat particles surrounded by a shell of proteins, which stops them from congealing into larger particles. These proteins include lactose and casein and can be quantified by FTIR [1]. Butter is produced by churning milk to break down the protein shell, allowing the fat to coagulate into large butter particles suspended in a buttermilk liquid phase containing the lactose proteins. The butter phase, (containing other proteins, including casein) is extracted by washing with water and then worked into a solid mass. This solid mass is an emulsion comprised of fat (~80%), water and milk solids.

On heating butter to its melting point the emulsion separates into a fat phase on top and a water phase containing the milk solids on the bottom. This process is known as the clarification of butter.

Here we investigate this process by heating butter stepwise 5 °C at a time on a Quest™ ATR with a 30 min hold at each temperature.

The heated puck enables the ATR study of the temperature induced behavior of liquids, small solids and powders.

- Heat liquid and solid samples up to 110 °C.
- Accurate, stable temperature measurement via 4-wire RTD sensor.
- Superior physical and chemical robustness provided by monolithic diamond ATR crystals.
- Setting and logging temperature made simple by PC software and compact USB interface.
- Retrofits to all Quest family accessories.

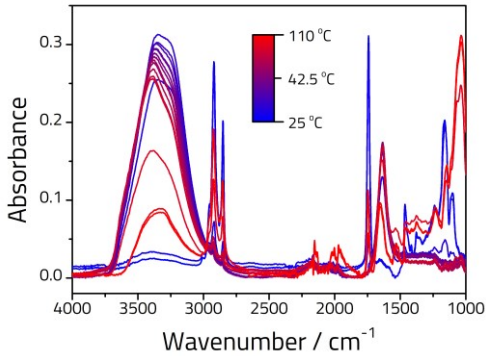


Figure 1: FTIR spectra showing the effect of heating a sample of uncovered butter from room temperature to 110 °C.

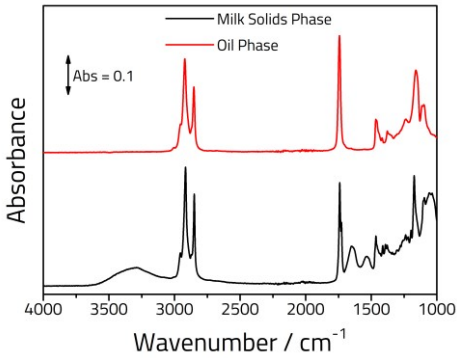


Figure 2: ATR spectra recorded at room temperature from the two separate phases formed by heating butter to 110 °C.

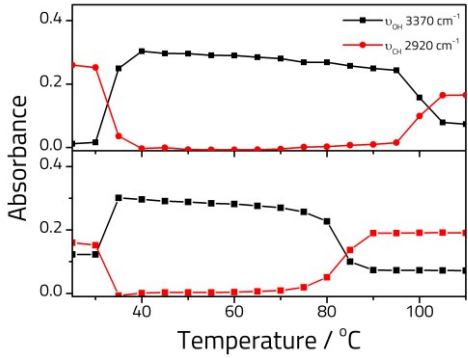


Figure 3: Changes to the OH and CH stretching bands observed for the sample of butter as the temperature is increased from room temperature to 110 °C. Top Panel: ~0.6 g butter placed on puck. Bottom Panel: ~0.2 g butter placed on puck.

Experimental

Unsalted butter made from 100% British milk was used in this work. Spectra were recorded on a commercially available spectrometer with 32 scans per spectrum at 4 cm^{-1} resolution using a Quest ATR accessory fitted with a heated diamond puck. Temperature control was achieved using the Specac Temperature Controller software. Two experiments were run with different masses of butter place on the Quest in each case.

Background/sample spectra were recorded by heating the puck in 5 °C increments, with a 30-minute hold at each step to allow the puck and sample to stabilise before recording a background spectrum. The experiment was run overnight by synchronizing the spectrometer program to the temperature controller programming of the heated puck. Each sample spectrum was reprocessed with the correct temperature background to minimise diamond phonon interference.

Results and Discussion

Figure 1 shows the impact of heating butter from room temperature to 110 °C on the FTIR spectra. At low temperature (darkest blue line) peaks associated with the oil component of the emulsion are clearly visible (saturated CH stretches, 3000-2800 cm^{-1}, C=O stretch at 1740 cm^{-1} and fingerprint region, 1500 -1000 cm^{-1}), as well as small peaks associated with the water component (3390 and 1630 cm^{-1}). The evolution of the OH and CH stretching peaks is shown in Figure 3 (top panel). As the sample is heated the emulsion separates into two phases: an oil phase which floats to the top and disappears from the spectrum due to limited depth of penetration [2]; and a water phase which sits on the bottom as evidenced by the increase in peaks assigned to water. As the sample is further heated to the boiling point of water, the water peaks decrease in intensity and new peaks are observed (bright red line) including CH and C=O stretching peaks with additional peaks at 1530 and 1035 cm^{-1}. The peak at 1530 cm^{-1} is assigned to an Amide II protein band from proteins in the milk solids (tentatively to casein). These peaks were potentially always present in the water phase, but until they were concentrated by boiling the water, they were below the limits of detection. A residual peak in the OH region is potentially due to carboxylic functional groups within these proteins. On cooling a translucent oil phase was observed at the top of the puck, whilst a white phase

containing the milk solids was observed at the bottom.

The two phases were extracted and ATR spectra of both were obtained (Figure 2). In the case of the oil phase a good spectrum was obtained by smearing it onto the ATR crystal without using a load to bring it into contact with the diamond. With the milk solids phase a 40 lb load was used to ensure good contact to obtain an ATR spectrum. As expected, the milk solids phase matches the spectra recorded at high temperature after the water phase has evaporated. The oil phase closely matches peaks that were initially present in the butter, which diminished on heating.

Finally, the experiment was repeated with a reduced quantity of butter to investigate what changes this would have on the recorded spectra. This is shown in Figure 3 (bottom panel). The initial concentration of water is significantly higher in the room temperature spectra than those recorded in the first experiment. Upon investigation it was discovered that the butter was not very homogenous, with large variations in the ratio of peaks assigned to water and oil being observed. This is potentially due to the age of the butter and the length of time it had been stored in the fridge. Nonetheless similar melting and phase separation is observed at the same temperature. One critical difference is the temperature where the water disappears from the spectra and the milk solids appear. Reducing the quantity of water reduces the time for the water to evaporate resulting in these peaks appearing at a lower temperature. This may also be impacted by the thickness of the oil layer, which will be higher with increased quantity of butter. If the experiment dwell time at each temperature was increased this would also impact on when the water content was observed to evaporate in figure 3 (as temperature is also a proxy for time).

Conclusions

The Quest ATR with heated puck provides an excellent solution for the analysis of butter and its transition from solid to liquid phase. The use of ATR enables fast and simple analysis of the sample without the need for any preparation, and following analysis it is simple to wipe clean. The software is simple to programme and monitor temperature steps. The experimental method outlined here is simple to perform and can be easily reproduced.

References

[1] Specac Application note: AN16-02 Analyzing Milk with the Pearl
[2] Specac Technical note: TN21-02 ATR Penetration Depth

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Appendix Available Online

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