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Detection of Organic Crystallites in Ice Using Terahertz Time-Domain Spectroscopy

Sergey Mitryukovskiy, Jean-François Lampin and Romain Peretti
IEMN, CNRS, Université Lille 1, Villeneuve d'Ascq, 59652 France

Abstract— We report on the detection of a narrow absorption signature of organic crystallites (alpha-lactose monohydrate) in ice using terahertz time-domain spectroscopy.

I. INTRODUCTION

TERAHERTZ time-domain spectroscopy (THz-TDS) is a powerful tool to characterize organic samples and many compounds, like explosives and pharmaceuticals [1-4]. Indeed, THz-TDS study of many samples in their native aqueous environment is limited due to the strong absorption by liquid water or its vapor in the THz range. However, in aqueous ice there are fewer molecular rotational modes and THz pulses propagate much further [5, 6]. Thus, it enables the possibility to probe frozen sample such as a frozen product from the Agribusiness with the goal to probe their quality and even more to check if the cold chain had been followed. Still, one needs to go a step further in analyzing iced samples to better benchmark what information will be obtained from such experiments. As a beginning, the detection of sodium chloride ion pairs in ice was reported [5], dielectric constants of H₂O, D₂O [6] and Ih ice [7] in THz range was measured over temperature.

We present a THz-TDS study of iced samples prepared from lactose aqueous solutions, and then of ice with undissolved lactose crystallites. Those samples consist in a step further toward real frozen sample experiments. The observed strong absorption signature of the crystallites allows its identification and demonstrates the perspective of the approach.

II. RESULTS

THz absorbance spectra were recorded using a TeraSmart THz-TDS setup by Menlo systems GmbH [8] placed into a glove box with a nitrogen gas flow to reduce water vapor absorption during the measurement.

An alpha-lactose monohydrate powder obtained from Sigma-Aldrich was used for sample preparation. The lactose powder was dissolved in distilled water to obtain a saturated solution, which was then diluted by distilled water to get several sample solutions with different lactose concentration (namely 0.569 mol/L, 0.285 mol/L, 0.142 mol/L, and 0.071 mol/L). We also prepared a sample with the lactose concentration (~ 1 mol/L) higher than the saturation concentration to assure the presence of undissolved lactose crystallites. The sample solutions were contained in 4-mL plastic cuvettes to form rectangular cuboid samples of ice with the base dimensions of 10 mm × 10 mm (fixed) and the height around 40 mm (which is approximate due to the nonuniform volume increase during crystallization). The cooling of the samples was performed by placing the cuvettes into a cryogenic vessel filled with liquid nitrogen (the sample with undissolved lactose was shaken and rapidly frozen to keep the

uniform distribution of lactose crystallites in ice). The iced samples were then inserted into the path of the THz pulses.

The retrieved absorbance spectra are presented in the Fig. 1. The absorbance intensity increases with the increase of the lactose concentration. This is due to modification of the ice structure. For the sample with lactose concentration higher than the saturation one, a narrow peak of absorption is observed around 0.53 THz, a clear signature of alpha-lactose monohydrate crystallites in ice, which is consistent with the previously reported absorbance spectrum of lactose powder pressed into pallets [1, 9, 10].

III. SUMMARY

The resonant attenuation signature of alpha-lactose

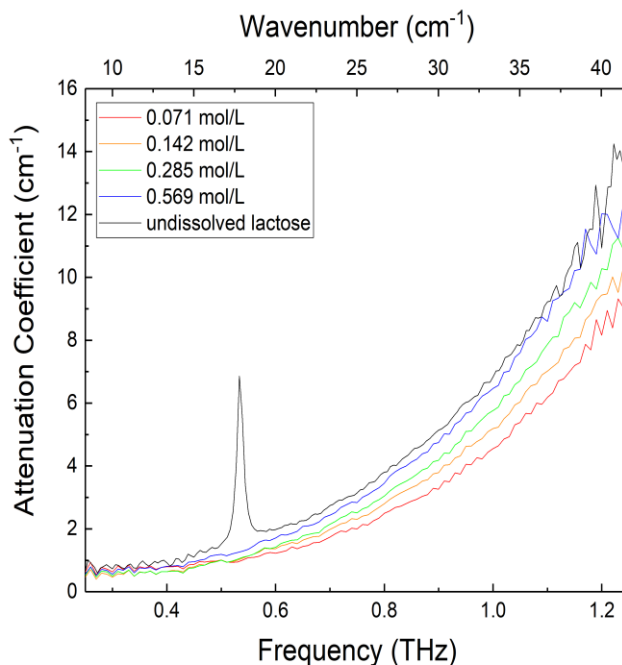


Fig. 1. Attenuation coefficient spectrum of ice prepared from lactose aqueous water solution at different concentrations (color curves) and from the saturated solution with undissolved lactose crystallites (black curve).

monohydrate crystallites in ice was observed by THz-TDS. The use of ice is advantageous for characterization of organic samples and fully compatible with agribusiness industry. At the conference, we will present our further study of various organic structures (carbohydrates)

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