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Quantification of Terahertz Scattering in Water-Oil Emulsion

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***Abstract*— Scattering in water-paraffin oil emulsions was studied by terahertz time-domain spectroscopy (THz-TDS) with the aim of estimating droplet size and concentration. The loss coefficients in water-oil emulsion systems were measured at very low concentrations of water (≤ 1% v/v) to establish the sensitivity of THz-TDS. As expected, the scattering losses increased linearly with water concentration. The scattering spectra exhibited a quadratic dependence on frequency, indicating that Mie scattering was the dominant scattering loss mechanism in this system.**

I. INTRODUCTION

Mineral oils act as the primary component of lubrication in engines and suffer from contamination and degradation with use, which can severely impact their efficacy. A major contaminant of lubricating oils is water, which can be present after the refining processes from crude oil and is also generated as a product of fuel combustion. This water is present as free water droplets as well as water-mineral oil emulsions. Both free water and emulsified water are dangerous to engine function, resulting in corrosion of fuel tanks, pipes and engine parts, forming particulates or rust that can accumulate in the system. There are also risks of lubrication loss due to the relatively smaller viscosity of water, and fuel flow obstruction may also be caused by corrosion by-products and sludge resulting from microorganisms whose growth is encouraged by aerobic conditions [1].

This, coupled with the strong absorption by water at THz frequencies [2], serves as strong motivation to employ THz-TDS as a direct, non-contact spectroscopic technique that can be developed into an in-situ sensor for quality assessment of ageing of oils. In addition to water, THz-TDS has been shown to detect other contaminants such as gasoline [3], engine wear and tear particles, soot [4] and other leakage or abrasion products from the engine. Moreover, at THz frequencies, the scattering loss from the contaminants in engine oil can be quantified, which can allow for extrapolation of both their concentration levels and size [5].

II. RESULTS

Spectroscopy grade paraffin oil was chosen for this study due to its high purity and because its mechanical properties are like lubricant oils. Water-oil mixes of 0.125%, 0.5%, and 1% v/v (1% v/v = 10,000 ppm) were prepared by stirring the measured volume of water into paraffin oil at ~350 rpm for 1 minute, thus breaking up the planar interfaces between the oil and water to initiate the emulsification process. The samples were subsequently sonicated in an ultrasound bath (Grant, 150 W, 44 kHz) for 45 minutes until a visually homogenous, cloudy-white emulsion was obtained (Figure 1 inset). The resulting emulsions were measured by a Teraflash Pro Time-Domain Terahertz spectrometer from Toptica Photonics. Two cells were manufactured in house, having a CNC machined stainless steel body and Z-cut quartz windows (Figure 1 inset). The two cells had different beam path lengths, which were 10 mm and 20 mm.

The Fresnel reflection losses at each interface of the cell were accounted for in parameter calculations [6]. The uniformity and reproducibility of the emulsions was confirmed by preparing separate samples having the same water concentration and measuring them in the two cells, showing consistent loss spectra. The mechanical stability of the emulsions during the measurement was confirmed by acquiring 2000 averages every 3 minutes for 36 minutes. Changes in the THz loss spectra became observable every 9 minutes, indicating that the emulsions remained stable during the data acquisition, which took 1.5 minutes.

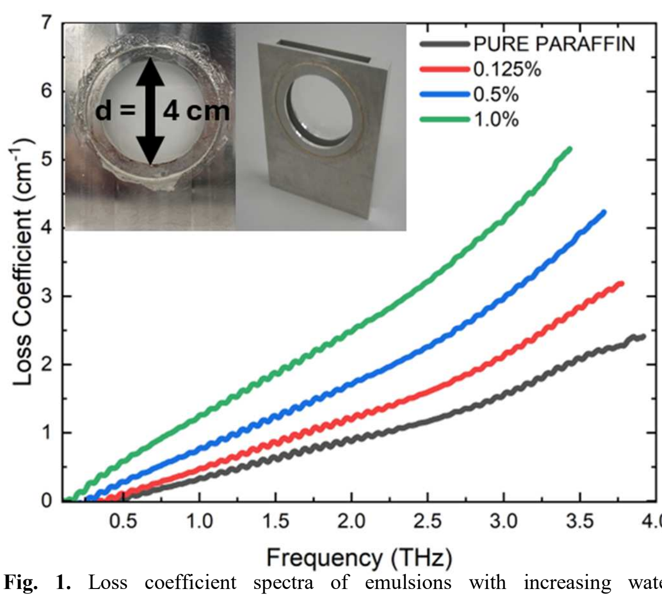

Fig. 1. Loss coefficient spectra of emulsions with increasing water concentration (0.125%, 0.5%, 1.0% v/v); the inset depicts an example of a filled cell and an empty cell (l = 10 mm).

Figure 1 shows the loss spectra of the water-in-oil emulsion samples with 0.125%, 0.50% and 1.0% v/v water content. As expected, the spectra of pure paraffin as well as the emulsions are featureless with a rising absorption edge. The measured loss coefficient increases with increasing water concentration with the dispersion profile becoming steeper as water content increases. This indicates that THz-TDS can be used to quantify water contamination levels in oil as low as 1250 ppm. Figure 2 depicts the calculated scattering spectra of the emulsions, obtained by subtracting the absorption contributions of paraffin and water from the total measured loss [7]. A quadratic fit has been applied to the scattering loss, confirming Mie scattering in these emulsions and indicating droplet size of the order of the incident THz wavelength [5,7]. Similar behavior was observed in solid compacts [7], indicating that THz scattering in water-in-oil emulsions behaves similarly to solid mixed-media compacts and can be used to quantify the size and concentration of water droplets.

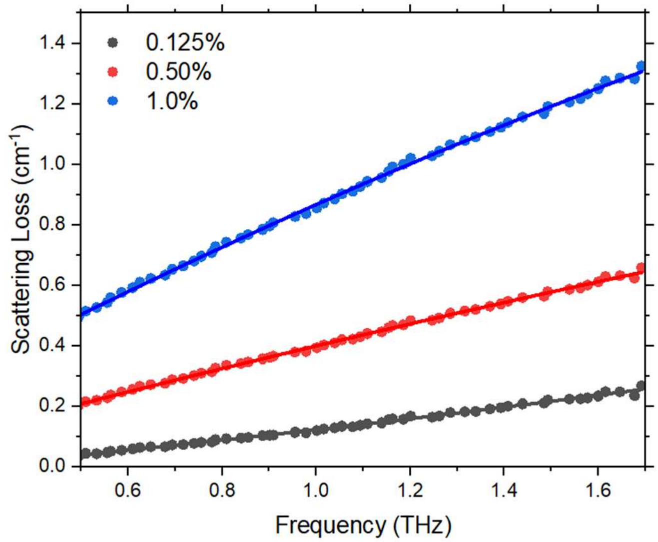

Fig. 2. Scattering loss spectra of the measured emulsions together with a quadratic fit ($R^2 > 0.98$)

The scattering loss for each of the three concentrations is extracted at 0.5 THz intervals across 2-3.5 THz and plotted as a function of water concentration, as shown in Figure 3, exhibiting a linear increase. Such linear dependence of scattering on concentration at low concentrations has similarly been reported for borosilicate microspheres suspended in a PTFE matrix [7].

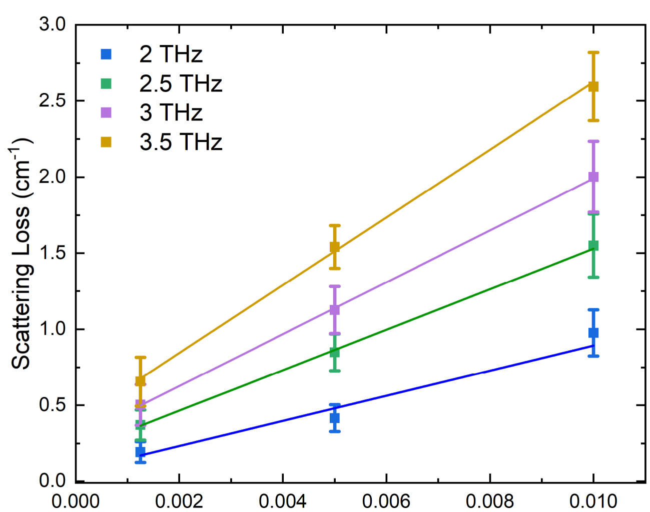

Fig. 3. Scattering loss estimated from total loss coefficient and fitted linearly ($R^2 > 0.99$) at 2, 2.5, 3 and 3.5 THz, as a function of volume ratio of distilled water.

III. Conclusions

Our results have demonstrated that THz-TDS is a sensitive tool to detect and quantify water concentration in water-in-oil emulsions down to 1250 ppm, possibly even lower. The calculated scattering loss was shown to increase linearly with water concentration, a phenomenon also reported for solid mixed-media compacts. This makes quantification of scattering a feasible methodology to investigate dispersed phases in a liquid medium, thus allowing the extraction of its domain size and concentration. This could potentially be useful for a variety of industrial applications such as in-situ quality assessments of of liquid suspensions in the pharmaceutical industry, formulations of cosmetics phases in the beauty industry, understanding emulsification behavior of food products, and obtaining contaminant information from aerospace fluids in the aviation industry.

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