A Novel Method To Measure Emulsification

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Introduction

The rate of lipolysis of dietary fat depends critically on emulsion particle size, which influences fat absorption and metabolism. In order to study the factors affecting fat emulsification within the gastric lumen it is necessary to measure the degree of emulsification in vivo. The measurement of emulsification is also important in the food industry and in other areas. NMR has been widely used to measure droplet size distributions using PGSE methods1, which are sensitive to motion and require high gradient amplitudes. A technique based on relaxation would be inherently more robust, particularly in vivo. We are not aware of any literature on using NMR relaxometry in this way, although it has been used to characterise emulsion fat fraction2. We have previously observed3 that T2 depends on droplet size, and have hypothesised that this may be due to diffusion in local field gradients between the droplets and water3,4.

This paper presents a technique for measuring emulsification based on EPI relaxometry, and a theoretical model5 to explain the data. Changes in 1/T2 of 5 oil-in-water emulsions in the range 0.4-20.9 μm were measured and modeled in vitro at 37°C.

Theory

The signal changes due to microscopic susceptibility variations can be quite complex and have been modelled using Monte Carlo techniques6. As validation we can use also outer-sphere relaxation theory to obtain analytical solutions in the motionally narrowed regime, valid for δω-τg<1 (δω = equatorial frequency shift and τg = R2/D with R = oil droplet radius and D = water diffusion coefficient at 37°C). For the particle sizes we are investigating, the secular component is dominant and the change in transverse relaxation rate ΔR2 can be expressed as:

$$\Delta R_2 = \frac{1}{T_{2,\text{emulsion}}} - \frac{1}{T_{2,\text{water}}} = \frac{16}{135} f \delta \omega B_0^2 \frac{R^2}{D}$$

where f is the volume fraction of the oil, γ the proton gyromagnetic ratio, Δω the difference in magnetic susceptibility between oil and water, and B0 the static field (all in SI units).

Methods

Oil-in-water emulsions were produced using 8% olive oil and 2% monoesterate emulsifier. Droplet sizes were measured by laser diffraction methods. EPI was performed on a whole-body 0.5 T purpose-built scanner. Actively shielded gradients and a 50 cm diameter bird-cage coil were used to acquire single-shot EPI images with a 128 by 128 matrix and 3.5 mm by 2.5 mm by 1 cm resolution. Δω of olive oil was measured using a phase mapping technique7. T1 data was acquired using an hyperbolic secant IR EPI sequence at 15 inversion times from 60 ms to 12 s. A SE EPI sequence was used to acquire data to measure T2 at 8 echo times from 60 to 700 ms. T1 measurements were carried out on 5 samples for each particle size. Proton random walk simulations (10000 spins with 170 μs temporal sampling out to a time of 100 ms) were performed to model the signal changes due to the oil droplets.

Results

We measured Δω=1.55·10^-6 in SI units. T2 of water was measured to be 2 s. For this Δω, the analytical expression shown above will be valid for droplet diameters ≤ approx. 12 μm under our experimental conditions. The mean droplet diameters produced were 0.4, 2.7, 6.4, 11.6 and 20.9 μm. No significant changes in T1 with changing mean droplet size were found (mean T1=2.86 s). However, it was observed that the transverse relaxation rate 1/T2 increased for larger droplet sizes (Figure 1).

Discussion

T2 measurements were found to provide a robust method of assessing emulsion droplet size. Excellent agreement was found with the Monte Carlo results and the analytical expression in the range of validity (≤ 12 μm), which is the range of interest in the gastric environment. The discrepancy between the Monte Carlo simulation and the experiment for the largest droplet size is probably due to visible creaming of the emulsion. Other systems could be studied in this way, and if necessary the sensitivity of the technique could be increased by doping the water phase of the emulsion to increase Δω, T1 did not vary significantly as surface relaxation is not expected to be a major contribution in T1 relaxation of oil emulsions4. Fat concentration in the stomach will change with time due to meal dilution by secretion, emptying and layering. Therefore it would be necessary to determine simultaneously the fat concentration at each time point and that could be achieved by using fat/water suppressed imaging, localised spectroscopy or measurements of the mean T1 of the non-exchanging fat/water mixture.

Conclusion

This paper shows that it would be possible to measure oil emulsion droplet size in vivo within the gastric lumen using EPI relaxometry by exploiting the effects of Δω on the emulsion 1/T2. The speed of EPI quantitation will allow dynamic changes in emulsification to be studied in vivo. The emulsions used in this work are food-grade, acceptable to volunteers and provide good contrast between the gastric lumen and the surrounding organs. Future work will test this method in vivo against laser diffraction measurements on nasogastric aspirates and will validate the theory further by comparing the results of single echo and multiecho T2 measurements.

References


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