

## Nano-Rheometry for Non-Invasive Monitoring of Texture During Food Oral Processing

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### ABSTRACT

Food oral processing is the first step in the digestive process which prepares food for swallowing and digestion. The process only lasts a few seconds, yet it determines our complete perception of texture, taste and aroma of the product we are eating. This oral processing is an intricate combination of voluntary and involuntary actions, and it involves complex flow geometry, mass transport of fluids and gases and signal processing and feedback from the brain. Any attempt of inserting measuring devices in the mouth will fail because the complete oral processing will be influenced.

We have developed a remote, non-invasive determination technique using magnetic sensing of magnetic nanoparticles iron oxide particles. A small amount of these particles senses their surrounding texture through their rotation and the nano-viscoelasticity can therefore be picked up without disturbing the oral processing.

### INTRODUCTION

Flavour, texture, appearance, sound, and temperature are attributes important for food palatability<sup>1</sup>. Contrary to aroma, texture is associated with large molecules and detected through a physical pathway<sup>2</sup>. Texture during oral processing is difficult to monitor non-invasively.

Texture integrates mechanical and thermal properties as well as perception of particles (grittiness). Rheology is a major factor for texture, and it has been shown e.g., that increased viscosity lowers perceived taste intensity due to reduced transport-rate of taste component<sup>3,4</sup>. However, there are limitations in rheological measurements for identification of food texture and *in vivo* human measurements must be introduced to complement rheometry<sup>2,5,6</sup>. No published methods give direct rheological properties of the food during oral processing and most research has been performed on expectorated boluses<sup>7</sup>.

By measuring and analysing the dynamic magnetic response from magnetic nanoparticles (MNP) in a viscoelastic medium the stochastic Brownian relaxation of the MNPs can be determined. Local nano-viscoelastic properties (complex shear modulus  $G^*$  and dynamic viscosity  $\eta^*$ ) can be calculated from the Brownian relaxation time  $\tau_B$ <sup>8,9</sup>. This makes it possible to determine viscoelastic properties remotely, non-invasively in the oral cavity. It requires addition of e.g. iron oxide particles (E172) to the food. The technique has been used to determine rheological properties of Newtonian fluids, non-Newtonian fluids and to monitor systems from solution to gel<sup>9-12</sup>. A time varying magnetic field is applied over the MNP system and the

dynamic response is measured with a highly sensitive AC Susceptometry (ACS) instruments. The frequency is varied, and the response is measured at each frequency which gives the Brownian relaxation time. For viscoelastic fluids complex models have to be used for dynamic magnetic modelling of the experimental data such as the model by Raikher et al.<sup>11, 13</sup>.

In the current study we have scaled up the ACS system so it can be used for magnetic nanorheology (MNR) during oral processing.

## **MATERIALS AND METHODS**

### **Materials**

Strained tomatoes, sunflower oil and gelatine was obtained from the local grocery store. Sunflower oil was obtained from the local grocery store. Powdered egg white and egg yolk, and modified starch was kindly provided by Findus Special Foods (Malmö, Sweden).

Starch embedded MNPs (100nm in diameter) of iron oxide cores and covered with PEG were obtained from Micromod Partikeltechnologie GmbH (Rostock, Germany) as a solution of 25 mg/ml MNP.

### **Preparation of foods**

Tomato gels were prepared as previously described from strained tomatoes heated above 80°C and gelatine added and stirred into the solution<sup>14</sup>. The proportion of ingredients was 65% (w/w) strained tomato, 33% MNP(w/w) solution and 1.8% (w/w) gelatine. The solution was directly transferred to the rheometer for conventional rheometry or cooled and kept at 8°C overnight before magnetic nanorheometry.

A solid timbale was mixed from strained tomato, egg white, egg yolk, starch, NaCl, oil and MNP solution (33%). The dispersion was mixed and heated at 80°C for 60 minutes in pieces. For mechanical testing and rheometry the MNP-solution was exchanged for water.

### **Rheometry**

An HR 30 rheometer (TA Instruments, New Castle, DE, USA) was used for Small Amplitude Oscillatory Shear (SAOS) analysis, equipped with a 25 mm-diameter parallel plate system and 2 mm gap for timbale characterization, and 40 mm-diameter parallel plate system and 1 mm gap for gel characterization. The bottom plate was temperature controlled and the measuring system was enclosed in a solvent-trap enclosure. Slices 25 mm in diameter and 2 mm thick were cut using a vacuum holder<sup>15</sup>. The gap was actively adopting to changes in samples volume by temperature.

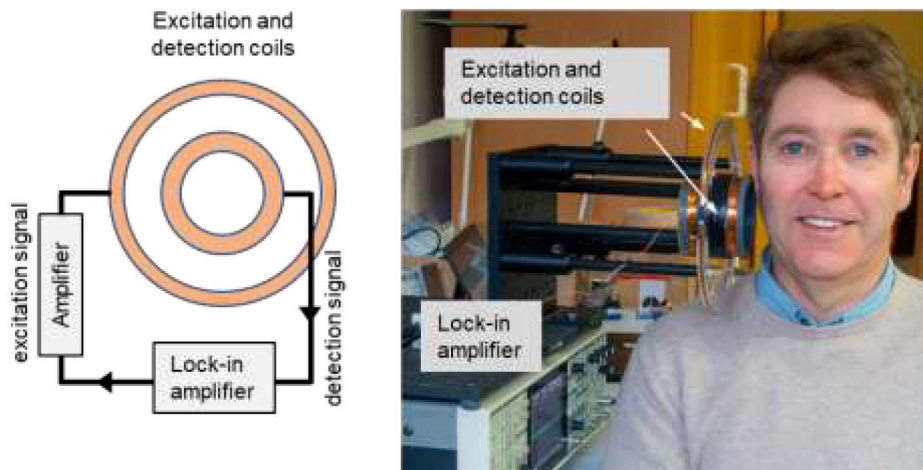
The complex shear modulus was monitored during heating from 8-45°C min to cover the effect of temperature while ingesting a piece of the food and high heating rate (15°C/min) was therefore used.

### **AC Suceptomety**

The ACS system consist of an excitation coil to create an alternating magnetic field and two detection coils which are differentially connected for measuring the response of the magnetic sample. The differentially connected pickup coils work as first order gradiometer and screens the environmental magnetic noise and the applied magnetic field. Using lock-in measurement technique, the in-phase and out-of-phase components of the voltage induced in the pickup coils from the magnetic sample is measured. The sample response is measured with great sensitivity at different excitation frequencies and can be converted to the magnetic susceptibility of the material<sup>16</sup>.

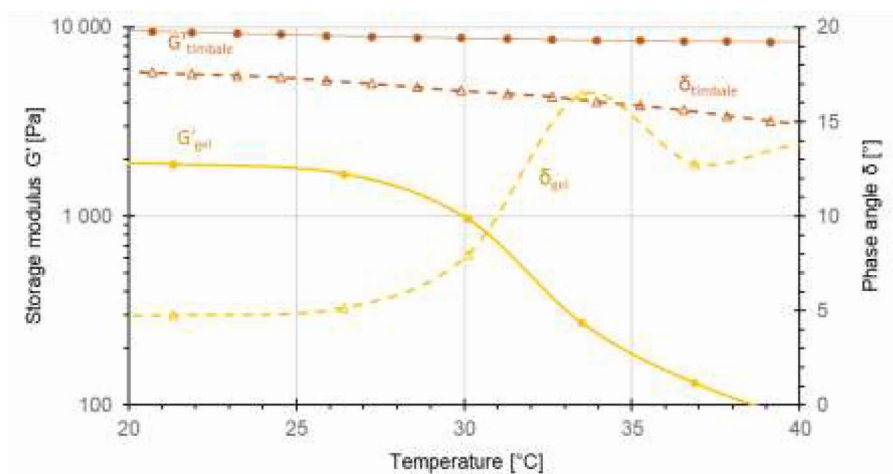
## RESULTS AND DISCUSSION

The AC Susceptometry system is shown in **Fig. 1**. The subject placed himself so that the magnetic coils almost touched the cheek in order to catch the signal from the iron oxide particles in the food, as the maximum monitoring range was about two centimetres. The signal is stored in the memory of the lock-in amplifier and converted to the two components of the magnetic susceptibility  $\chi$ . A similar complex notation as in SAOS is used to render the in-phase component  $\chi'$  and the out-of-phase component  $\chi''$ .



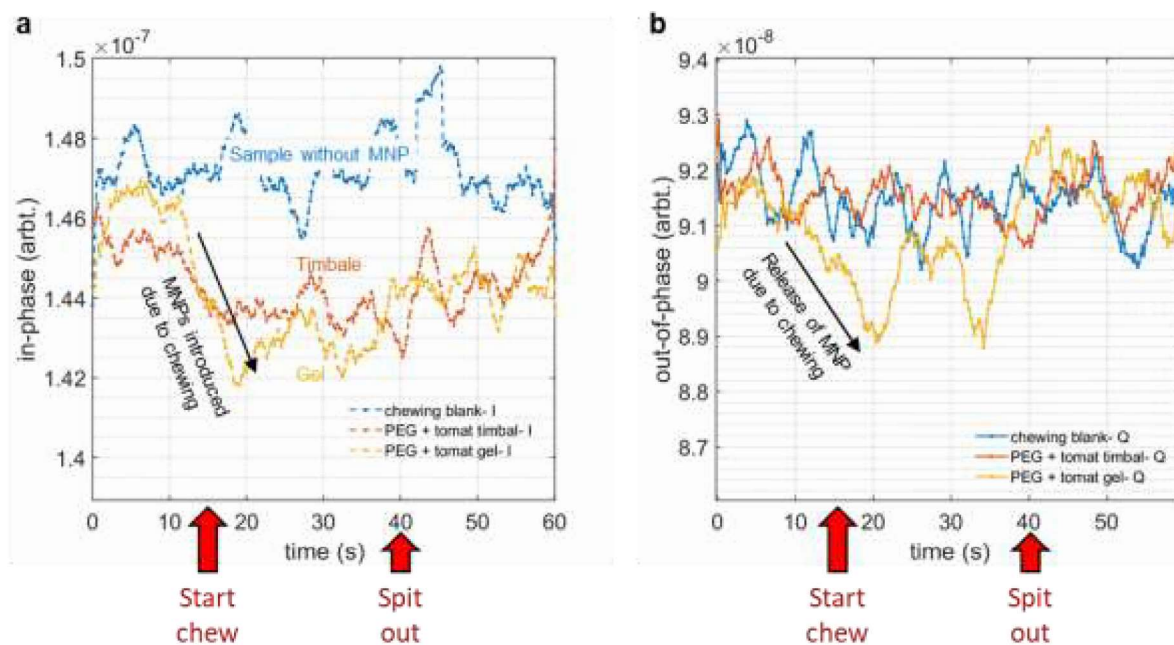
**FIGURE 1:** Present setup of MNR for non-invasive determination oral processing showing the transmission and pick-up coil system and electronics (lock-in amplifier), and one healthy subject.

Two model food products with distinctly different texture properties were used with timbale and gel consistency<sup>14, 17</sup>. Both were made from strained tomatoes. The timbale structure is formed from egg protein and starch, whereas the tomato gel is formed by adding gelatine to the strained tomatoes and let them gel<sup>14</sup>. When ingested both model foods are heated from room temperature to 37°C, then chewed to smaller pieces and mixed with saliva. **Fig. 2** shows that the storage modulus of the timbales was almost one order of magnitude larger than the gels at 20°C and due to melting of the gelatine gel even larger at 37°C. This is important for those suffering from dysphagia who needs a gel diet. The gels melt in the mouth thus requiring minimal oral processing.



**FIGURE 2:** Effect of heating on a tomato timbale (orange) and tomato gel (yellow).

**Fig. 3** shows that the magnetic response of foods with magnetic iron oxide particles can indeed be detected using the developed setup. The food was ingested at 10s, chewed on the side of the mouth closest to the coils and spat out at 40s. The presence of the particles was shown as a change in the in-phase component,  $\chi'$ , of the magnetic susceptibility as shown in **Fig. 3a** for both the gel and timbale foods, as opposed to a blank without particles (blue line). The mobility of the particles was shown by the out-of-phase signal  $\chi''$  in **Fig. 3b** for the gel food but was difficult to resolve for the timbale due to less mobility in the food matrix.



**FIGURE 3:** Proof-of-concept of chewing test. Timbale and gel samples containing magnetic iron oxide particles were ingested at 15s, chewed and spat out at 40s. The in-phase signal (a) of the magnetic susceptibility reflects the amount of detected particles in the mouth and the out-of-phase signal the rheological response.

## CONCLUSIONS

The nano-rheological response of foods with magnetic iron oxide particles can be detected through the magnetic susceptibility, non-invasively during food oral processing. The monitoring distance covers texture monitoring during one-sided chewing and the working distance therefore needs to be expanded. The sensitivity of the system thus needs to be increased.

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