Optimisation of applied harmonics in Fourier Transform Rheology to enable rapid acquisition of mechanical spectra of strain-sensitive, time dependent materials

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ABSTRACT

Biological fluids such as food boluses are complex fluids which often are inhomogeneous, change over time and have a limited linear region. The rheological properties of a food bolus determine how easy it is to swallow which is crucial for those suffering from swallowing disorders.

It is advantageous to use Fourier transform rheology to quickly obtain the mechanical spectrum of a bolus as it changes over time. Several harmonic strains are superimposed, and the resulting stress response is transformed into a mechanical spectrum. A novel optimisation algorithm was applied to minimise the maximal strain and strain rate applied to the sensitive bolus sample. The time to obtain a mechanical spectrum was reduced from 10 to 3.5 minutes.

INTRODUCTION

The global population is getting older and the fraction of individuals over 65 years is already 20 % in Sweden and as high as 30 % in Japan (global average is 9 %). Apart from the obvious benefits it also means a higher prevalence of age-related issues such as swallowing disorders, or dysphagia, which affects 40 % of the ones older than 70 due to e.g. dementia or side effects of medication. Many of these need texture adjusted foods which are easy to chew and swallow without causing aspiration or leaving residues in the throat. The food oral processing results in a bolus with rheological properties ready to be swallowed. By adjusting the composition and particle size of the food the rheological properties of the bolus can be tuned for easy swallowing. For fluid foods, increased shear viscosity is adopted to slow down the flow thus giving time for an age-impaired system to react. We have previously shown that fluid elasticity improves bolus cohesivity and leads to easy and safe swallowing^{1,2}.

On signs of dysphagia Swedish patients are given softer solid food with decreased particle size. In the national scale the "timbale" consistency class is the most commonly served to dysphagia patients still able to eat solid food^{3,4}. The following classes are "gel food", "high viscosity" soups and "low viscosity" soups. Other common systems for consistency classification are the American Dietetic Association guidelines and IDDSI, The International Dysphagia Diet Standardisation Initiative⁵, but these place less emphasis on solid foods.

After ingesting a piece of a solid food, we chew it and mix it with saliva until the particle size is reduced and it forms a cohesive bolus ready to be swallowed. This is an unconscious process even if we actively can influence e.g. how long we chew the food. The bolus is a viscoelastic fluid and the rheological properties can be determined in small-amplitude oscillatory shear (SAOS), as well as in non-linear shear and extensional flow. Bolus experiments are typically

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performed by the subject chewing the food until ready-to-swallow and then spits out the bolus. The rheological properties can then be determined, and the method has to be carefully designed as the properties change with time and linear region for SAOS is limited. A mechanical spectrum from 0.01-10 Hz typically takes 10 minutes to perform, but using Fourier Transform, or multiwave rheology this can be shortened to a few minutes. A broad range of frequencies are applied simultaneously instead of in series, and the resulting stress is Fourier transformed to give the mechanical spectrum. However, superimposed strain harmonics add up and may impose strains outside the linear region. Another approach to shorten the acquisition time of a mechanical spectrum is to use "chirps"⁶.

The present paper presents a method to optimise the phase of the applied harmonics to minimise both strain and maximum strain rate.

THEORY

"Fourier Transform Mechanical Spectroscopy" was first introduced by Holly and co-workers in 1988 for 3 simultaneous frequencies⁷. Fourier Transform Rheology (FTR) has then been developed and refined⁸, and recently used much in large-amplitude oscillatory shear (LAOS)^{9, 10}.

Individual sinusoidal functions are superimposed to give a complex harmonic shear deformation as shown in the example in Figure 1. The 2nd harmonic has twice the fundamental frequency, the 3rd three times the fundamental frequency etc. The maximum amplitude in Figure 1 is more than three times the strain of the individual sinusoidal functions and depends on the individual amplitudes as well as on the phase.

The stress resulting from the superimposed harmonics is similarly complex and Fourier analysis is used to obtain the mechanical spectrum, $G^{*}_{(f)}$.



Figure 1. Superposition of fundamental frequency, 2nd, 3rd and 4th harmonic of equal amplitude and phase.

The strain amplitude of the individual harmonics and their respective phases are set to give a resulting waveform as the example in Figure 1. When dealing with strain sensitive samples it is desirable to keep the maximum strain amplitude low not to break any structure of the sample. It is also desirable to keep the strain rate low to minimize effects of sample and measuring system inertia.

The strain amplitude in the rheometer software used herein can be written

$$f(t) = \sum_{n=1}^{N} (-1)^{\omega_n} \alpha_n \sin(\omega_n t + \varphi_n)$$

for (integer) frequencies $\omega_1, ..., \omega_N$, amplitudes $\alpha_1, ..., \alpha_N$, and phases $\varphi_1, ..., \varphi_N$. The strain rate is the derivative of the strain amplitude i.e.

$$f'(t) = \sum_{n=1}^{N} (-1)^{\omega_n} \alpha_n \omega_n \cos(\omega_n t + \varphi_n)$$

The aim is to minimize both the maximum absolute strain amplitude and the maximum absolute strain rate. The frequencies ω_n are fixed by the experiment (relative to the fundamental frequency ω_1 of the experiment, the actual value of which does not affect the optimization). Further, it is not meaningful to optimize with respect to the amplitudes α_n because the optimum is found for $\alpha_n = 0$ for all *n*. Hence, we wish to optimize with respect to the phases φ_n for fixed frequency and amplitude parameters. Minimizing the two quantities is a multi-objective optimization problem; we wish to minimize both $\max_t |f(t)|$ and $\max_t |f'(t)|$ and the minimum value of both will not occur for the same of phase parameters φ_n . We perform a so-called scalarization of the multi-objective optimization problem, minimizing the weighted sum

$$C = \gamma \max_t |f(t)| + (1 - \gamma) \max_t |f'(t)|$$

with respect to φ_n . Here, γ is an arbitrary weight parameter that specifies the trade-off between minimizing maximum absolute strain amplitude and the maximum absolute strain rate. Generally, the maximum of sums of sines and cosines cannot be found analytically; therefore, the maximum is found by grid search on 1,000 points in the interval from 0 to 2π . The target C is a continuous but not differentiable function of Hence, we use derivative-free φ_n . optimization, implemented in the 'patternsearch' algorithm Matlab in (Mathworks, Natick, MA, US), initiated with a large number of random initial parameter guesses. We choose γ in the following fashion. For the frequency and amplitude parameters used herein, we evaluate the average value of the ratio of the two maxima,

$$r = \left\langle \frac{\max_t |f'(t)|}{\max_t |f(t)|} \right\rangle,$$

for uniformly distributed, independent phase parameters. To compensate for the different scales of the two values, one reasonable choice is γ such that $\gamma/(1 - \gamma) = r$ or $\gamma = r/(1 + r) \approx 0.95$, which is the value we use.

MATERIALS AND METHODS

Bread timbales (96008767) from Fundus Special Foods, Malmö, Sweden were used and 4g was served to a healthy subject who was instructed to chew until ready to swallow, and then spit out the bolus. About 0.5 cm³ was loaded on a 20 mm bottom plate at 37°C of 20 mm parallel plate system in an ARES G2 (TA Instruments, New Castle, USA). The upper plate was lowered, and the sample trimmed to give a final gap of 1.5 mm. A solvent trap system was used to ensure as homogenous temperature and humidity surrounding the sample as possible.

Two "multiwave" experiments were performed in series with N = 10 components covering the following frequencies: Fundamental ($\omega_1 = 1$), 2nd, 3rd, 4th, 6th, 10th, 16th, 32nd, 64th and 100 ($\omega_{10} = 100$) harmonic. The fundamental of the first multiwave experiment was 0.01 Hz and in the 2nd experiment 0.33 Hz. The amplitudes were chosen in an exponentially decaying manner such that $\alpha_1 = 0.4$ % strain and $\alpha_{10} = 0.1$ % strain.

RESULTS AND DISCUSSION

The storage and loss moduli of a bolus of bread timbale are shown in Figure 2. Judging from the logarithmic scales the linear region seems to extend up to 1% strain, but by careful inspection of G' (inset in Figure 2) the linear response is reasonably linear below 0.5 % strain. This is confirmed by the Fourier transform of the stress signal. When plotting the ratio of absolute magnitude of the 3rd harmonic and that of the fundamental frequency (right y-axis in Figure 2) it starts increasing well below 1 % strain. This ratio is a measure of the non-linearity of the sinusoidal stress signal and should be kept low. At the same time a high strain minimises noise in the measured torque. For this sample the upper strain limit was set to 0.5 % as a balance between the two criteria.

The mechanical spectrum for the food bolus for frequencies 0.01-10 Hz takes about 10 minutes to obtain when applying the frequencies in sequence. The exact time depends on how long the start of data acquisition is delayed at each frequency to alleviate inertia effects. The development of storage modulus and phase angle of the food bolus sample over time after loading it in the instrument is shown in Figure 3.



Figure 2. Complex shear moduli and the ratio of absolute magnitude of the 3rd harmonic and that of the fundamental frequency (1 Hz) as function of applied strain for a food bolus. The inset shows a linear close-up of G'_(strain).

Time 0 is set to 60 s after the sample touches the heated lower plate. G' increase with time due to enzyme action and structural rearrangements and it is therefore necessary to obtain the mechanical spectrum as quickly as possible. It is equally important to allow a soak time for temperature equilibration in the sample. A solvent trap enclosure is placed around the sample, which on the pre-heated bottom plate, but a temperature difference between the plates is nevertheless unavoidable.

By using FTR a mechanical spectrum for frequencies 0.01-33 Hz can be obtained in 3.5 minutes. Figure 4a shows this spectrum as symbols, where the separate spectra 0.01-1 Hz and 0.33-33 Hz overlap perfectly. The "classically obtained" mechanical spectrum is shown as lines in good agreement with the FTR spectra. The maximum strain amplitude applied in the FTR spectra was 0.47 %.

However, it is important to use an optimised superposition of applied harmonic amplitudes and phase as described in the Theory section. If only the strain amplitude is adjusted but all phases are set to 0° the two FTR mechanical spectra do not perfectly overlap, as shown in Figure 4b.



Figure 3. Evolution of the storage modulus and temperature after loading a bolus in the measuring system.

If a superposition of harmonics of equal amplitude (0.5 % strain) and phase (0°) the resulting mechanical spectrum is shown in Figure 4c. The discrepancy between the classical and FTR spectra depends on the individual strain amplitudes adding up to a maximum of 2.7 % which is outside the linear region breaking the internal bolus structure.

The boundary conditions for the optimised superposition was an exponentially decreasing amplitude since the stress signal is lower at low frequency thus more sensitive to noise. The optimisation then gave the strain amplitudes and phases in Table 1.

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Harmonic	Strain	Phase
	amplitude	[°]
1 (fundamental)	0.200	47.2
2	0.173	168
3	0.151	125
4	0.133	359
6	0.115	343
10	0.075	169
16	0.056	2.83
32	0.050	300
64	0.050	195
100	0.050	22.2

Table 1. Strain amplitude and phase of theapplied optimised harmonics.



Figure 4. Mechanical spectrum of a food bolus. Symbols show the spectrum obtained using Fourier Transform rheology and the "classical" spectrum by lines. a) Spectra obtained by optimised phase and amplitude of the individual harmonics, b) Adjusted strain amplitude, constant phase and c) strain amplitude 0.5 % and phase 0 for all harmonics.

CONCLUSION

The optimisation of amplitude and phase for the harmonics used in a Fourier Transform experiment enables fast and accurate acquisition of the mechanical spectrum also for strain sensitive samples.

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