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# Characterization of food stuffs using Magnetic Resonance Elastography

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## Abstract

Viscoelastic properties of formulated food products are often associated with the textural properties of the material. Plasticity provides an important food quality factor. Unfortunately, viscoelastic properties of food stuffs are normally measured in the bulk phase, prior to packaging. Here we describe the application of a Magnetic Resonance Elastography (MRE) method using a specially designed sample holder for fast, reproducible and non-invasive measurement of spatially averaged viscoelastic constants of packaged samples. MRE experiments provide viscoelastic data as a function of position within samples and can be performed prior and post packaging, on samples including those with an optically opaque container or wrapper.

**Keywords:** Packaged food, Quality control, Non-invasive, MRI

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## 1. Introduction

Rheology plays a very important role in the modern food industry as it defines three major categories of food acceptability, appearance, flavor and touch. Evaluation of food texture, intermediate or final product quality control and shelf-life testing often involve rheological testing (Steffe, 1996). The food industry spends great efforts trying to control the extreme softening and textural degradation that occurs during food preservation, instantaneously or over time (Hui, 2005; Taub & Sing, 1997; Kilcast, 2004). Many different techniques to measure viscoelastic properties of materials exist (Steffe, 1996). It is not the purpose of this paper to review all of these, however, it should be pointed out that most of these techniques are applied either in- or on-line on a production line or require access to the material without its packaging. Results from measurements performed this way are usually only valid for a narrow range of geometries, frequencies, etc.

The most common dynamic method used to measure viscoelastic properties of food stuffs is oscillatory testing (Steffe, 1996). Harmonic variation of stress or strain shows that viscoelastic properties are very sensitive to the chemical and physical composition of the materials. This makes the technique ideal to relate rheological properties to human sensory perception and to study the shelf-life of products (Rao & Skinner, 1986). Unfortunately, none of the techniques frequently used can be used on packaged, finished, products to test their quality during shelf-life. Recently, Magnetic Resonance Imaging (MRI) was used to monitor moisture distribution in multi-component food systems and spatially resolve its re-arrangement

26 during storage (Ramos-Cabrer *et al.*, 2006). It was also shown, using the  
27 NMR MOUSE, that magnetic resonance presents a good option for non-  
28 invasive assessment of micro-structural parameters in manufacturing and  
29 in the supply chain (Haiduc *et al.*, 2007). Although MRI provides informa-  
30 tion on (micro) structural features such as size and spatial distribution by  
31 means of different water relaxation parameters of these structures, it does  
32 not provide direct information on physical properties such as shear.

33 Recently, Dynamic Mechanical Analysis (DMA) (Jones, 1999) was used  
34 to validate Magnetic Resonance Elastography (MRE) measurements of shear  
35 in agarose gels (Chen *et al.*, 2005). DMA analysis was performed on pre-  
36 formed disks of agar gel poured in petri dishes in order to control slab  
37 thickness. The experiments provided values of the shear modulus as a  
38 bulk parameter. Similar experiments have been performed using mechan-  
39 ical compression tests (Hamhaber *et al.*, 2003). MRE on the other hand  
40 is capable of providing data as a function of position within the sample.  
41 This is an extremely valuable asset as this allows the study of complete  
42 end products of a food processing unit, without the need to prepare sam-  
43 ples for study. In many cases MRE measurements can be performed non-  
44 invasively, without having to remove the packaging, allowing for easy  
45 spatial mapping of viscoelastic properties, especially useful for products  
46 with complex compositions. MRE is a phase-contrast magnetic resonance  
47 imaging technique to map spatial displacement patterns corresponding to  
48 harmonic shear waves initiated by the mechanical oscillations of a spe-  
49 cially designed actuator attached to the object (Lewa, 1992; Muthupillai *et*  
50 *al.*, 1995; Plewes *et al.*, 1995). Apart from many medical applications study-

51 ing specific changes in tissue stiffness related to disease, *e.g.* liver fibrosis,  
52 cancer, *etc.* (Fatemi *et al.*, 2003), MRE has also been used to non-invasively  
53 study phase transitions in gels (Sack *et al.*, 2001).

54 We report the design and use of a simple but effective pneumatic ac-  
55 tuator for the study of shear stress in food products and show its use in  
56 the determination of shear stress in pre-packaged fruit puddings as an ex-  
57 ample. MRE experiments enabled us to determine the shear modulus as a  
58 function of position in the samples and to monitor the distribution of fruit  
59 pieces. At present this technique is not useful for factory in-line quality  
60 control tests, however, it will be useful in the product development phase  
61 in the laboratory.

## 62 2. Materials and Methods

### 63 2.1. Magnetic Resonance

64 All MRE experiments were performed at room temperature using a 3T  
65 TrioTim medical MRI system (Siemens, Germany). Samples were mounted  
66 on a sample holder (RF coil insert) equipped with rollers as shown in Fig-  
67 ure 1. The roller axes were positioned parallel to the main magnetic field,  
68 to allow an oscillatory motion in the direction perpendicular to the field  
69 as shown in Figures 1 C and D. The sample holder was positioned in be-  
70 tween two flexible, pneumatically driven actuators positioned on the side  
71 of the commercial RF head coil (Fig. 1B). Two speakers, delivering sound  
72 pressure through separate hard-walled flexible tubes, were used to drive  
73 the actuators in an out-of phase sense (180° phase shift). The direction  
74 of the main magnetic field  $B_0$ , the actuator-induced motion of the sample

75 and the induced shear waves are shown in Figure 1 D. A programmable  
76 waveform generator, synchronized to the MRE pulse sequence, was used  
77 to deliver an acoustic frequency in the range of 20-150 Hz with actuator  
78 amplitudes up to 500  $\mu\text{m}$ .

79 MRE experiments were performed with a modified Gradient Echo se-  
80 quence using a 40° RF-flip angle, an echo time (TE) of 18 ms, a repetition  
81 time (TR) of 500 ms, a 10×10 cm<sup>2</sup> field of view (FOV), a 64×64 data matrix  
82 and sinusoidally shaped motion encoding gradients with strengths vary-  
83 ing from 5 to 25 mT·m<sup>-1</sup>. Motion encoding, using the oscillating sinus-  
84 shaped gradients, was performed at 75-150 Hz, to match the oscillation  
85 frequency of the sample. The number of motion encoding gradient pairs,  
86 using one cycle per acquisition, was set to 8, *i.e.* 8 images were acquired  
87 at a different phase of the oscillation. Prior to motion encoding the ampli-  
88 tude of the acoustic signal was ramped over a period of 200 ms to avoid  
89 transient signals (Sack *et al.*, 2008).

90 The designed dual-actuator unit, shown in Figure 1B and C, can be  
91 easily mounted into a commercially available head coil (Siemens, 3T), by  
92 replacing the original head restrainers and adjusting the fit to the sample  
93 holder, providing good contact of the holder with the two actuators. It  
94 should be noted that, although the setup described in this work used a  
95 medical MRI scanner, the concept can easily be adapted to non-medical  
96 MRI equipment. An adaptation for micro-imaging of agar gels and frog  
97 eggs using a typical vertical bore magnet (56 mm, narrow bore, Oxford  
98 Instruments, Oxford, UK) using a home build piezo-electric actuator has  
99 been reported (Othman *et al.*, 2005).

100 Data processing was performed using MREwave, software obtained  
101 from Dr. Ehman's laboratory (Mayo Clinic, Rochester, MN). MREwave  
102 uses a local frequency estimation algorithm which provides an estimate of  
103 the local spatial frequency of shear wave propagation in the sample (Man-  
104 duca *et al.*, 2001; Knutson *et al.*, 1994). The algorithm is relatively insensi-  
105 tive to phase noise and yields an accurate isotropic frequency estimation.

106 As an example of a packaged food product we used a small container  
107 of fruit pudding (98 g) containing water, sugar, fructose, coconut, citric  
108 acid, seaweed extract and a natural flavor (ABC Fruit Pudding, packaged  
109 for Loblaw's Inc., Canada). The pudding was packaged in a hard plastic  
110 conical container with a bottom and top diameter of 5.5 and 6.7 cm, re-  
111 spectively, and a height of 3 cm. Coconut was added as small cubes with  
112 an approximate dimension of  $1 \times 1 \times 1 \text{ cm}^3$ .

## 113 2.2. *Dynamic Compression*

114 Dynamic compression tests were performed at ambient temperature  
115 on three samples using an Enduratec ElectroForce 3200 test instrument  
116 (Bose Corporation, MN, USA). Specimens of pudding without coconut in-  
117 clusions, having a diameter of 18 mm and a height of 5 mm, were submit-  
118 ted to oscillating deformation in unconfined compression. The resulting  
119 forces were measured using a 500 g range load cell. To maintain a linear  
120 viscoelastic response, the oscillation amplitude was kept small (3% strain).  
121 The frequency was varied from 1 Hz to 20 Hz. Viscoelastic parameters  
122 were determined for each frequency from which the shear modulus was  
123 calculated, using a Poisson ratio of 0.49.

### 124 3. Statistics

125 Data in this study are represented as means  $\pm$  standard deviation. Mea-  
126 surements were performed in triplicate. Data fitting was performed with  
127 QtiPlot (GPL) using the Marquardt-Levenberg algorithm.

### 128 4. Theory

129 In general, linear viscoelasticity of soft materials can be described by  
130 appropriate mechanical models composed of dashpots and springs (Steffe,  
131 1996). Springs represent the solid element obeying Hooke's law while the  
132 dashpot introduces the Newtonian fluid characteristics. The two simple  
133 models combining one component of each are the Maxwell and the Voigt  
134 model (Catheline *et al.*, 2004). In the Maxwell model the elements are ar-  
135 ranged in series, while in the Voigt model the components are connected  
136 in parallel. Both models provide a similar and good description of dis-  
137 persion, however, the Voigt model was able to predict the frequency de-  
138 pendent shear attenuation much more accurately (Catheline *et al.*, 2004).  
139 Catheline *et al.* showed that the Voigt model describes agar gels and bio-  
140 logical soft tissues best, especially in the frequency range of  $f \geq 100$  Hz.  
141 At relatively high shear elasticity in the range of 5 kPa, the Voigt model  
142 provides an excellent approximation. However, when both elasticity and  
143 viscosity are relatively low, the Maxwell model does provide a good pre-  
144 diction of the speed of shear wave propagation, especially at lower fre-  
145 quencies (0-150 Hz).

146 Application of a sinusoidally modulated stress is known to result in  
147 a sinusoidally varying strain at the same frequency. This allows for the

148 following definition of stress and strain:

$$\sigma = \sigma_0 \cdot \exp(i\omega t) \quad (1)$$

$$\varepsilon = \varepsilon_0 \cdot \exp(i(\omega t - \Delta\phi)) \quad (2)$$

149 Here  $\sigma$  represents stress while  $\varepsilon$  describes strain and  $\Delta\phi$  the phase differ-  
 150 ence. The complex modulus describing their relation can thus be written  
 151 as:

$$M(\omega) = \frac{\sigma_0}{\varepsilon_0} \cdot \exp(i\Delta\phi) = M_1(\omega) + iM_2(\omega). \quad (3)$$

152  $M_1(\omega)$  is called the elastic (or storage) modulus, which is in phase with  
 153 the applied oscillating strain, while  $M_2(\omega)$  represents the viscous (or loss)  
 154 modulus which is out of phase ( $90^\circ$ ) with the applied strain. For the Voigt  
 155 model, these complex moduli are related to the elasticity modulus  $E$ , and  
 156 the viscosity coefficient  $\eta$ , by:

$$M_1^V(\omega) = E \quad (4)$$

$$M_2^V(\omega) = \omega \cdot \eta \quad (5)$$

157 For the Maxwell model the moduli are defined as:

$$M_1^M(\omega) = \frac{E \cdot \omega^2 \eta^2}{E^2 + \omega^2 \eta^2} \quad (6)$$

$$M_2^M(\omega) = \frac{E^2 \cdot \omega \eta}{E^2 + \omega^2 \eta^2} \quad (7)$$

158 Assuming we can describe the objects as isotropic homogeneous and in-  
 159 compressible systems, the propagation of shear waves can be modeled  
 160 by the Helmholtz equation (Catheline *et al.*, 2004). The Helmholtz equa-  
 161 tion allows us to calculate the shear speed attenuation for the Voigt and

162 Maxwell model in terms of the Lamé coefficients  $\mu_l$  and  $\eta_s$  for shear elas-  
 163 ticity and viscosity, respectively, as:

$$c_s^V(\omega) = \sqrt{\frac{2(\mu_l^2 + \omega^2\eta_s^2)}{\rho(\mu_l + \sqrt{\mu_l^2 + \omega^2\eta_s^2})}}, \quad (8)$$

$$c_s^M(\omega) = \sqrt{\frac{2\mu_l}{\rho \left(1 + \sqrt{1 + \frac{\mu_l^2}{\omega^2\eta_s^2}}\right)}} \quad (9)$$

164 Usually, for biological tissues and gels, the density,  $\rho$ , is assumed to be  
 165  $1000 \text{ kg}\cdot\text{m}^{-3}$  as a very good approximation. For a pure elastic medium  
 166 ( $\eta = 0$ ) and according to the Voigt model, the speed of the shear waves be-  
 167 comes frequency independent. This can be considered the low-viscosity  
 168 approximation in which  $c_s^V(\omega) \approx \sqrt{\mu_l \cdot \rho^{-1}}$ . In the limit of low viscosity,  
 169 the Maxwell model provides  $c_s^M(\omega) \approx \sqrt{2\mu_l \cdot \rho^{-1} \cdot \omega\eta_s}$  which indicates a  
 170 frequency dependence. For viscoelastic media the shear wave speed is  
 171 predicted to increase monotonically with frequency for either model out-  
 172 side the low viscosity limit.

## 173 5. Results and discussion

174 Figure 2 shows a scout image of the fruit pudding. The scout echo was  
 175 obtained with a Fast Spin Echo sequence in order to obtain the contrast  
 176 between the pudding and the coconut chunks. The bottom of the container  
 177 is shown on the right hand side of the image where the pieces of coconut  
 178 are indicated with arrows. Other slice orientations, not including pieces of  
 179 coconut, could also be selected. However, for the purpose of showing the  
 180 capabilities of the MRE technique and the data processing, we opted for  
 181 an orientation containing a few stiff objects.

182 The exact slice orientation as used for the scout image in Figure 2 was  
183 applied in the MRE experiments. MRE was performed at four different  
184 actuator frequencies; 75, 100, 125 and 150 Hz, to study the frequency de-  
185 pendence of the shear modulus. A typical data set for the 100 Hz actuator  
186 frequency is shown in Figure 3. The oscillatory motion from the actuator  
187 was applied perpendicular to the dotted line in Figure 3A in a top-bottom  
188 fashion for convenience, as shown in Figure 1D with respect to the exter-  
189 nal magnetic field  $B_0$ . This makes the shear wave propagate horizontally  
190 from right to left in all the images shown. Knowing the pixel resolution  
191 in the MR images, the wavelength of the shear waves could be calculated  
192 from phase images, shown in Figure 3A,B. The speed of the transverse  
193 waves could thus be obtained at each frequency. Figure 4A shows the  
194 slight frequency dependence of the wave propagation speed, indicating  
195 that the puddings are not a pure elastic medium. For viscoelastic media  
196 such as biological tissue and gels, the shear wave speed increases mono-  
197 tonically with the frequency,  $\omega$ , due to dispersive coupling with viscosity.  
198 The higher the slope in Figure 4A, the more viscous the material. This  
199 relation can be used to check the shelf-life, for instance, of food products  
200 against changes in viscosity without having to remove the materials from  
201 its wrapping or container.

202 To calculate the shear modulus,  $\mu_l$ , for the fruit pudding as a function of  
203 position in the sample, a Local Frequency Estimation (LFE) algorithm was  
204 used (Manduca *et al.*, 2001). LFE is a robust algorithm that yields accurate  
205 and isotropic estimates for the local frequency. For the area in between the  
206 two coconut pieces (see Figure 3D), an averaged local frequency estimate

207 of  $f_{LFE} = 96.2 \pm 6.5$  Hz which is a very good approximation of the actu-  
 208 ator frequency of 100 Hz. The LFE algorithm is relatively insensitive to  
 209 noise, making it a perfect tool in combination with MR which sometimes  
 210 can suffer from low signal to noise ratios. The LFE algorithm has limited  
 211 resolution, causing blurring of the frequency estimate near sharp bound-  
 212 aries. As a result the estimation of the shear modulus suffers from errors  
 213 at the edge of sample and, for the same reason, near inclusions when the  
 214 local stiffness changes rapidly. However, if the actual value of the shear  
 215 modulus of inclusions is not of interest, the LFE is an excellent algorithm  
 216 to detect these inclusions as they will be detectable as a change in local fre-  
 217 quency (see Figure 3D). Using the LFE algorithm thus allows for the accu-  
 218 rate spatial detection of inclusions, however, not for the determination of  
 219 their stiffness (in most cases, depending on the frequency used). Another  
 220 algorithm, AIDE, based on algebraic inversion of the equations of motion  
 221 (Helmholtz equation), requires the calculation of second derivatives which  
 222 makes this algorithm very sensitive to noise and not so suitable for MR.  
 223 In Figure 3C a map of the calculated shear modulus is shown. The area in  
 224 between the inclusions is characterized by a shear modulus,  $\mu_l = 0.75 \pm 0.15$   
 225 kPa. This value compares well with previously reported shear moduli  
 226 for alginate gels (LeRoux *et al.*, 1999). Seaweed (alginate) is an important  
 227 component of the fruit pudding. It should be mentioned that the shear and  
 228 viscous properties of alginate gels strongly depend on the amount of cross-  
 229 linking which is controlled by very small amounts of bi-valent cations. For  
 230 the measured  $c_s(\omega) < 1 \text{ m} \cdot \text{s}^{-1}$  in the excitation frequency range of  $75 \leq f \leq$   
 231 150 Hz, we observe the Voigt model to provide a good description of the

visco-elastic properties of the material. According to eqn.8, using a two parameter fit for viscosity and the shear modulus, the viscosity of the fruit pudding measured at 100 Hz is approximately  $0.49 \pm 0.03$  Pa·s (see Figure 4A). The same fit resulted in a value of  $0.61 \pm 0.02$  kPa for the shear modulus at 100 Hz. The density of the fruit pudding was kept fixed at  $1000 \text{ kg}\cdot\text{m}^{-3}$  throughout the data fitting. The observed frequency dependence of the shear wave velocity shown in figure 4A could not be fitted to the Maxwell model as described by eqn. 9.

Using dynamic compression, a separate measurement of the shear modulus in samples of the fruit pudding was performed in parallel. The relative softness of the material prevented measurements above 20 Hz. Figure 4B shows the obtained frequency dependence. This data was fitted to a linear relationship between shear stress and frequency in order to extrapolate the measurements to 100 Hz. The obtained fit predicted a shear modulus of 0.69 kPa (see Fig. 4B), a value that corresponds well with that obtained from MR elastography measurements. The frequency dependence of the shear modulus in the fruit pudding (Fig. 4B) is small in the frequency range of 1-100 Hz. A Maxwell model would predict a much larger frequency dependence in this range. Together with the MRE results, the dynamic compression measurements indicate that the Voigt model provides the best description of the visco-elastic properties of these fruit puddings.

In conclusion, we have shown that MRE can be used to non-invasively determine the spatial distribution of viscoelastic properties from food stuffs. MRE experiments could be a helpful addition to the development of multi-component food products and could be used for highly automated quality

control checks in the laboratory. Although a medical 3T scanner was used in the experiments described above, these experiments can also be performed at standard laboratory MR systems, including microimagers (Othman *et al.*, 2005), for samples of different shape and size, depending on the magnet bore width. Of course, with a decrease in sample size, an increase in oscillatory frequency is needed. This can be accommodated switching to piezo-electrical or electro-mechanical actuators. The use of MRE also allowed for an extension of the dynamic range of frequency measurements. Dynamic compression measurements could only be performed up to 20 Hz due to the softness of the material, however, using MRE we were able to measure the shear modulus up to 150 Hz. The relation  $c_s = \lambda \frac{\omega}{2\pi}$  and the field of view (FOV) of the MR image set a lower excitation frequency limit for the experiments while an upper limit is essentially set by the strength of the used encoding gradients.

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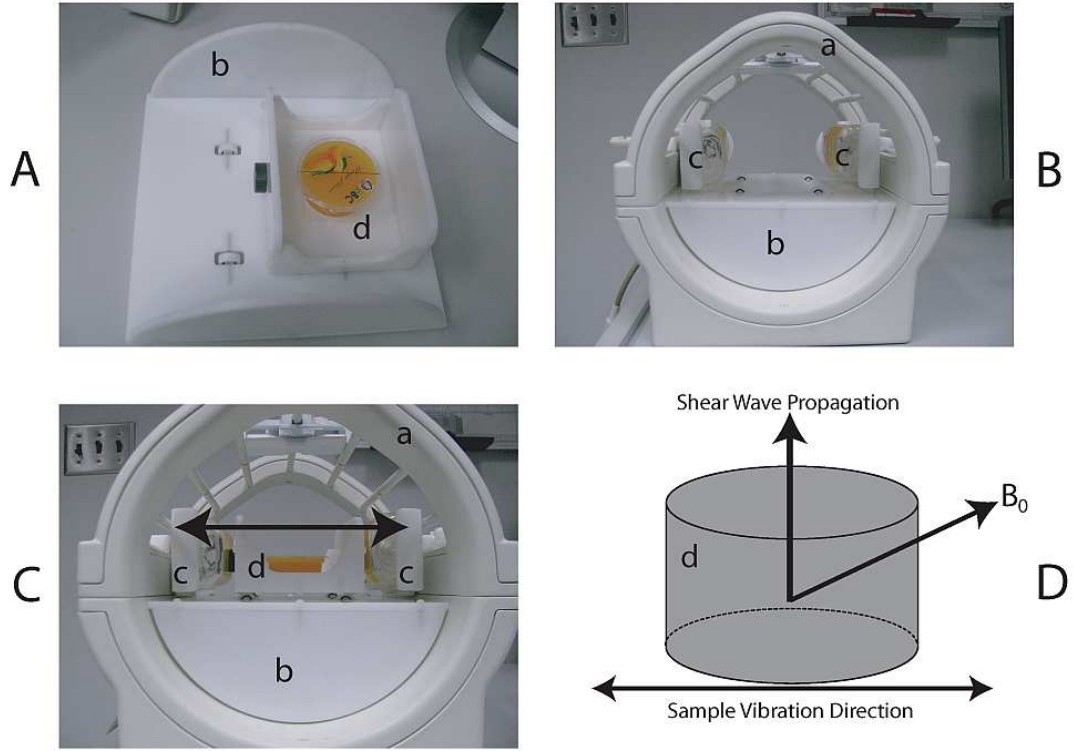


Figure 1: The pneumatic dual actuator set up. A) Removable sample holder with sample(d) shown on top of the coil-insert(b) with rollers to accommodate a left-right motion caused by the actuators. B) Position of the actuators(c) with respect to the coil. C) Complete set up of coil(a), coil-insert(b), actuators(c) and sample bed with sample(d) in the commercial head coil. D) Motion is induced by the actuators(c) from left to right in Fig. C, the orientation of the main magnetic field  $B_0$  is indicated in Fig. D with respect to the orientation of the shear waves and the actuator motion.

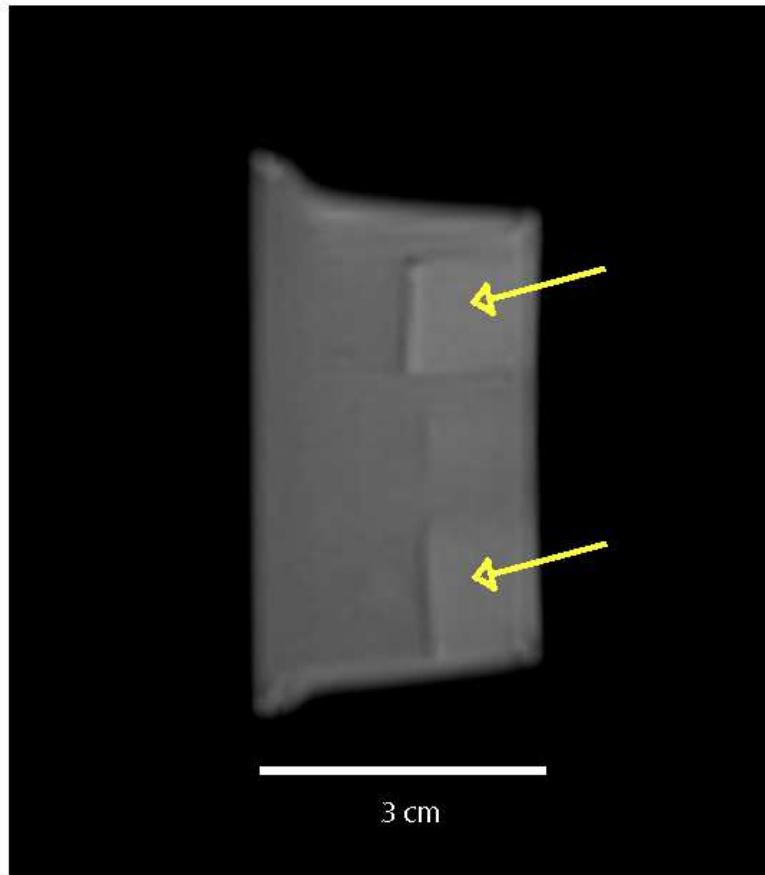


Figure 2: An MRI scout image clearly showing the chunks of coconut as indicated by the arrows. This slice orientation (5mm thick) was used for the MRE experiments.

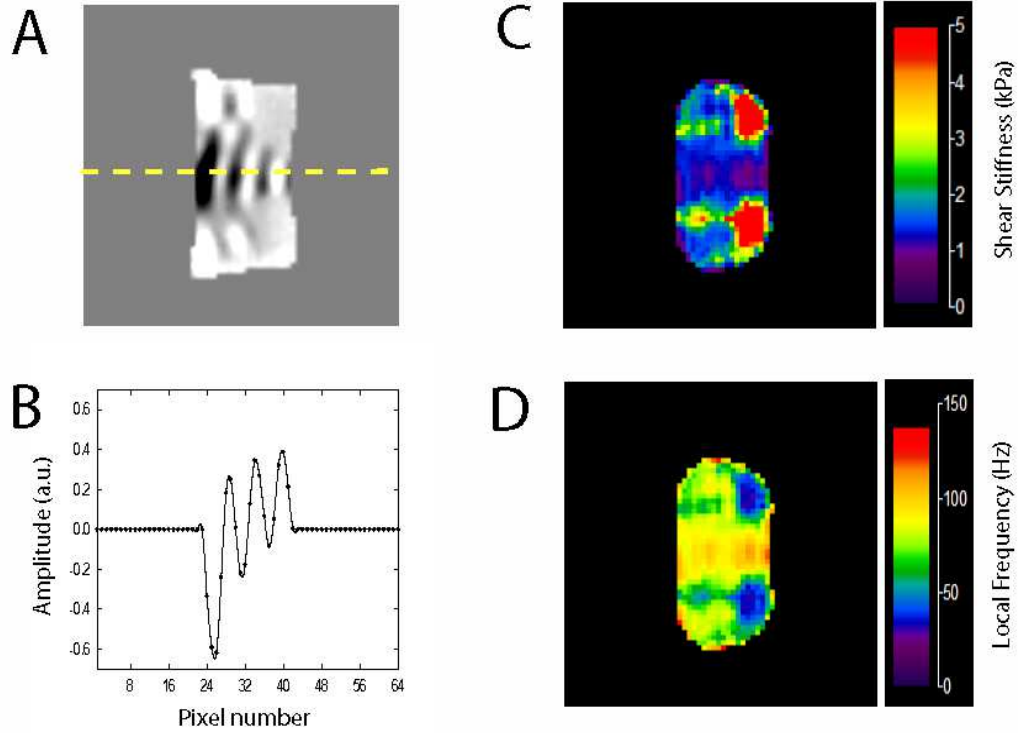


Figure 3: A) Wave image obtained at 100 Hz of mechanical oscillation. In the locations of the coconut pieces, the shear waves were strongly attenuated. Wave propagation through the center of the sample can clearly be seen. B) Amplitude oscillations obtained at the dotted line from the phase image in A. C) The estimated shear stiffness, using the Local Frequency Estimation algorithm. The coconut pieces can now clearly be distinguished due to their different composition from the rest of the pudding. Note that the shear modulus between the two stiffer coconut inclusions appear rather homogeneous. D) The local frequency as calculated by the LFE algorithm as a function of position. As in C for the shear modulus, the local frequency is well defined in the area between the coconut inclusions.

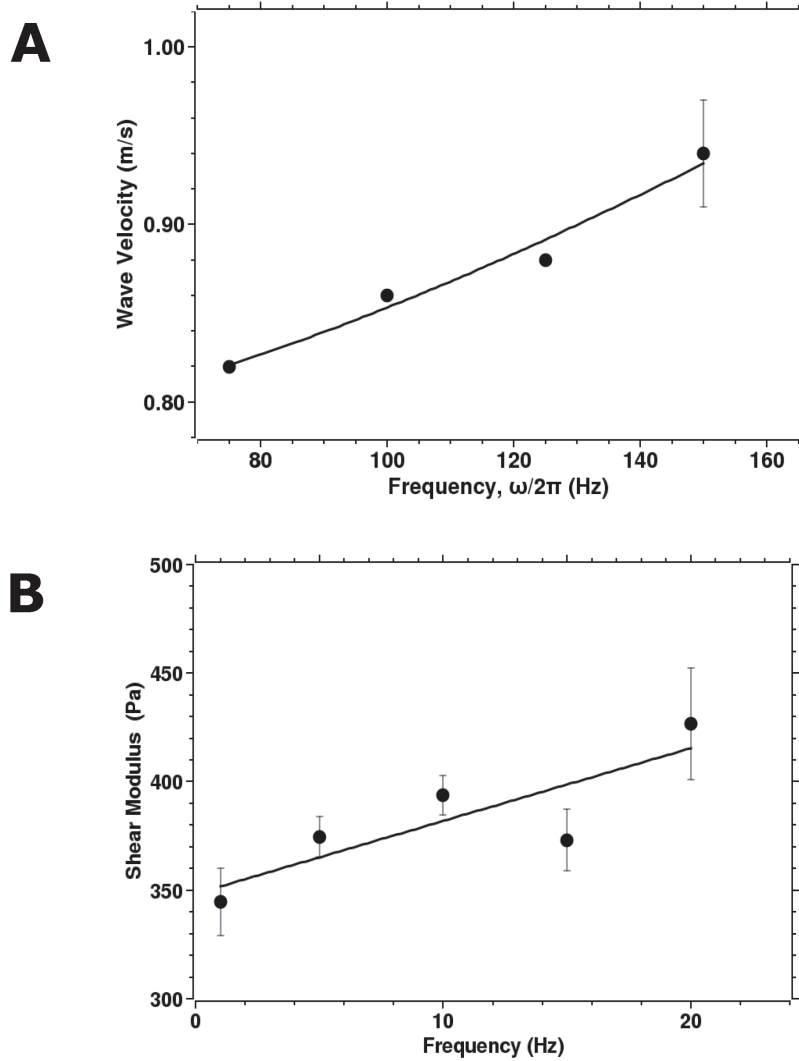


Figure 4: A) Frequency dependence of the shear wave velocity in fruit pudding. The solid curve represents a fit of the data to eqn. 8, using  $\mu_l = 0.61 \pm 0.02$  kPa and  $\eta_s = 0.49 \pm 0.03$  Pa·s. The density of the pudding was fixed at  $1000 \text{ kg}\cdot\text{m}^{-3}$ . B) Frequency dependence of the shear modulus, measured by dynamic compression. The shear modulus was fitted to a linear frequency dependence;  $\mu_l(\frac{\omega}{2\pi}) = (348 \pm 15) + (3.4 \pm 1.2)\frac{\omega}{2\pi}$ .