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ORIGINAL PAPER

Structural and Mechanical Properties of Fats Quantified by Ultrasonics

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Abstract Since the velocity of an ultrasonic wave through a material depends on its density, bulk modulus (*K*), and shear modulus (*G*), a new approach to determine the shear elastic modulus and the mass fractal dimension (*D*) in a fat crystal network was developed. An ultrasonic chirp wave containing a range of frequencies and amplitudes, was used to estimate the structural and mechanical properties of palm oil based fats, crystallized under shear at three different temperatures (20, 25, and 30 °C). Considering the fat crystal network as a two-phase system (i.e. liquid and solid fat) the velocity of sound in both phases was obtained separately, assuming that the speed of sound in the oil phase was inversely dependent on the temperature. A constant shear modulus for the solid fraction was obtained experimentally by rheology, which was independent of the sample's nature. These parameters were used for the determination of sample compressibility and its corresponding shear modulus by ultrasonic velocimetry. In addition fractal dimensions (*D*) were determined by using the relationship of the shear elastic modulus (*G*) to the mass fraction of the solid fat (ϕ) in a weak-link regime. The obtained results are comparable and consistent with previously reported fractal dimension values. This method allows online determination of the shear modulus of fats and could be potentially applied for quality control purposes in manufacturing.

Keywords Ultrasonic · Fat crystal network · Shear modulus · Bulk modulus · Mass fractal dimension

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Introduction

Some of the most important quality characteristics of fat-based food products such as the spreadability of margarine and butter, and the snap of chocolate depend on the crystal network formed within the finished product [1]. Hardness is one of the macroscopic properties that contribute to the sensory perception of a fat containing product [1, 2]. Although it is difficult to relate hardness to structure, a relationship between the elastic modulus of a fat and its hardness index has been proposed [1–4]. In fatty materials, the elastic modulus is related to the sensory impression of hardness, since it is the first level of structural resistance that is felt and overcome in the mouth as the fat is consumed. Therefore it can be used both as an indicator of macroscopic hardness and structure [4]. As a consequence, the problem of predicting the macroscopic sensory attributes of a food product has been simplified to determining the elastic modulus of the fat crystal network formed within such a product [1, 4].

Small and large deformation rheological methods have been developed by researchers to measure the elastic modulus of fats. However, ultrasonic spectrometry has not been widely adopted as one of these rheological methods. Ultrasonic techniques have advantages over others as they can be applied to systems which are optically opaque, concentrated and electrically non-conducting [5]. In addition, ultrasonic measurements are rapid, precise, non-invasive and non-destructive; they can be fully automated and are not hazardous [6].

The objective of this work was to develop a chirp wave ultrasonic velocimetry technique to estimate the shear modulus of different palm based fat systems in order to predict structural characteristics of their fat crystal networks.

Materials and Methods

Materials

Four Palm based fats, labeled as: 1, 2, 3, and 4 were diluted with canola oil in 10% increments from 50 to 100% (w/w). Tristearin (SSS) was used as a reference in measuring the shear modulus of the solid phase.

Crystallization Procedure

Samples 1, 2, 3, 4 with peak melting temperatures of 46.1, 43.5, 40.7, and 41.2 °C, respectively, were crystallized at 20, 25 and 30 °C under a constant agitation rate (400 rpm) during 90 min. The crystallization procedure was performed in an 8.11 cm internal diameter water-jacketed cell. The temperature of the cell was controlled by means of a water bath. The sample was stirring continuously by using a Lightnin mixture (Wytheville, VA, USA). Samples were heated to 120 °C in an oven, held at this temperature for 10 min and then introduced into the cell. The temperature profile was monitored by means of thermocouple dipped into the sample and the crystallization process was followed by means of ultrasonics and pulse nuclear magnetic resonance for 90 min. The crystallization cell was designed with two polycarbonate windows where the ultrasonic transducers were placed [7].

Ultrasonic Measurement

Ultrasonic measurements were performed by means of a SIA-7 ultrasonic spectrometer (VN Instruments Ltd., Elizabethtown, ON, Canada). Transducers of 1 MHz center frequency (GE Panametrics, MA, USA) were used to generate a chirp wave over a range of frequencies and amplitudes. The center frequency of the chirp corresponds to the transducer center frequency (1 MHz) and the range of frequencies these transducers operated in, is called the bandwidth. A synthetic impulse (SI) was generated from the chirp by means of a digital receiver processor. The time of flight (TF) is the time it takes for the wave to travel from one transducer to the other, which can be measured from the SI image. The distance traveled (D) is a constant (81.1 mm) and it depends on the cell geometry. These parameters can give the ultrasonic velocity by means of $v = \frac{D}{\mathrm{TF}}$. Data reported are averages of two replications. Details for the Ultrasonic measurements have previously been reported by Martini et al. [7].

X-ray Analysis

A Rigaku Multiplex Powder X-ray diffractometer (Rigaku, Japan) with a 1/2° divergence slit, 1/2° scatter slit, and a 0.3 mm receiving slit, was set at 40 kV and 44 mA and used to analyze the samples' polymorphism. Scans were performed from 1 to 30° at 1 °C/min. Results were analyzed by using MDI's Jade 6.5 software (Rigaku, Japan).

Rheology Measurements

A TA Instruments AR2000 controlled-stress dynamic Rheometer (TA Instruments, Mississauga, ON, Canada) was used to perform the rheological measurements. Oscillatory tests (small deformation) were performed by means of a strain sweep experiment. The experiments were carried out using a stainless steel parallel plate (20 mm diameter); the normal force was set approximately at 10 N for all samples. A constant frequency of 1 Hz was used and strain values were controlled from 0.003 to 0.8%.

Density Measurement

A standard 50 ml pycnometer was used to determine the sample density. The empty dried pycnometer was weighed, and filled with the melted sample. After 24 h storage at 20, 25, 30 °C the mass was recovered. The difference between the mass of the empty pycnometer and the pycnometer when it is full yields the mass of the fat. No significant differences were found between the density of the liquid and crystallized samples. For this reason despite of the fat sample, density was set at 0.92 g/cm^3.

SFC Measurements

Solid fat content (SFC) was measured by means of pulse nuclear magnetic resonance (p-NMR) using a Bruker Minispec spectrometer (Bruker Optics, Milton, ON, Canada), Samples were taken from the crystallization cell and poured into NMR glass tubes to perform the SFC measurements. The crystallization process was monitored by taking samples every minute for the first 10 min and then every 5 min until the end of the process (90 min). The reported data corresponds to the average of two individual measurements [7].

Relationship Between Shear Modulus and SFC

The elasticity of a fat crystal network is dependent on its microstructure, including the spatial distribution of mass within the network [4, 5, 8]. The solid-like microscopic properties of materials structured as flocculated colloids, in particular elasticity, are highly dependent on their structure in the nanometer range (nanostructure), as well as micrometer range (microstructure). The shear modulus scales with the volume fraction of solids in a power law fashion, from which the following relationship is obtained:

$$G \sim \phi^{\mu}\ , \tag{1}$$

where G is shear modulus, ϕ is the solid mass fraction, ϕ = SFC/100, and μ is the exponent of the volume fraction term which is related to the structure of the fat crystal network [9–11]. Based on this relationship, an estimate of fractal dimension of the system can be obtained from the slope of a log G versus log ϕ plot, assuming a weak link regime, as shown in Eq. 2:

$$G \sim \phi^{1/(3-D)}\ , \tag{2}$$

where D is the fractal dimension.

Theory

The Wood Equation [12] a well-known theoretical equation, was used to determine ultrasonic velocity through the different phases of a multiphase material. This equation can be used to explain the relationship between the ultrasonic velocity (v) of a partially crystalline fat and its solid fat content:

$$\frac{1}{v^2} = \sum_{j=1}^{n} \phi_j \rho_j \sum_{j=1}^{n} \frac{\phi_j}{v_j^2 \rho_j}\ , \tag{3}$$

where ρ_j, ϕ_j and v_j are the density, solid mass fraction, and acoustic velocity of the j phase, respectively [6, 12]. This equation assumes that $\sum_{j=1}^{n} \phi_j = 1$, i.e., that the components form an ideal mixture.

Wood Equation in Fat Systems

Since the densities for the various components of fat systems in this study are approximately similar, the Wood equation can be simplified to Eq. 4 [13–15]:

$$\frac{1}{v^2} = \sum_{j=1}^{n} \frac{\phi_j}{v_j^2}\ . \tag{4}$$

For partially crystalline fats which consist of liquid and solid phases Eq. 4 can be expressed as follows: [13]

$$\phi = \left(\frac{1/v_{\text{mix}}^2 - 1/v_{\text{l}}^2}{1/v_{\text{s}}^2 - 1/v_{\text{l}}^2} \right)\ , \tag{5}$$

where v_{s} and v_{l} are ultrasonic velocity at the measurement temperature in solid and liquid oil phases respectively, and v_{mix} is the velocity in the two-phase fat system.

As an ultrasonic wave travels through a material, it is attenuated, i.e. its amplitude decreases with distance traveled. The major causes of attenuation are absorption and scattering. Absorption occurs due to thermodynamic relaxation mechanisms, which convert energy from the ultrasonic wave into some other forms, ultimately heat [16]. Scattering occurs when some of the ultrasonic wave incident upon a discontinuity in a material (e.g. a particle) is scattered. Scattering of ultrasound is important in many systems since it can have a significant effect on the measured ultrasonic properties, making the velocity and attenuation dependent on particle size as well as concentration.

A high signal attenuation was observed during fat crystallization at high concentration [17]. Therefore, Eq. 4 could be used to model the relationship between ultrasonic velocity and SFC in partially crystallized fats (early stages of crystallization), or in low solid fat content systems, SFC < 20% [17].

Ultrasonics and the Theory of Elasticity

The propagation of sound in a material depends on the physical properties of that material such as the local energy, pressure and temperature of the medium [6]. When a longitudinal sound wave passes through a narrow structure with small cross-sectional dimensions compared to the wavelength, the ultrasonic velocity is given by:

$$v = \sqrt{\frac{E}{\rho}}\ , \tag{6}$$

where E is the Young's modulus.

According to the theory of elasticity, the Young's modulus is equal to:

$$E = K + \frac{4}{3} G\ , \tag{7}$$

where K is bulk modulus and G in the shear modulus. Therefore, the sound velocity can be also expressed as:

$$v = \sqrt{\frac{K + (4/3)G}{\rho}}\ , \tag{8}$$

where ρ is the sample density. In principle, from knowledge of the ultrasonic velocity, the density, the bulk modulus, and the shear modulus could be determined.

Determination of Bulk Modulus

Since the partially crystalline fat system is a mixture of solid and liquid parts, the solid–liquid mixture's bulk modulus K_{mix} is calculated from the bulk modulus of the liquid phase K_l and the solid phase K_s. From the definition of the bulk modulus (acoustic pressure change per unit volume-strain change) in Eq. 9:

$$K = V\frac{P}{\Delta V}\ , \tag{9}$$

$$\Delta V = \Delta V_s + \Delta V_l\ , \tag{10}$$

the total volume change for the sample is calculated using Eq. 10: where V_l, V_s, and V are the liquid volume, the solid volume and the total volume, respectively. P is the acoustic pressure of the system.

$$\Delta V_s = V_s\frac{P}{K_s},\quad \Delta V_l = V_l\frac{P}{K_l}\ . \tag{11}$$

Substituting Eqs. 11 and 9 into Eq. 10 and rearranging yields:

$$\frac{1}{K_{mix}} = \phi\frac{1}{K_s} + (1-\phi)\frac{1}{K_l}\ , \tag{12}$$

where K_l and K_s are liquid and solid bulk moduli, respectively.

In order to obtain the bulk moduli for the solid and liquid parts, the determination of ultrasonic velocity in both phases is necessary.

In liquids the shear modulus is very small compared to the bulk modulus ($G << K$) and it is thus possible to neglect its shear modulus [6, 8]. Therefore, the wave propagation velocity (v_l) in the liquid is approximated by:

$$v_l \approx \sqrt{\frac{K_l}{\rho_l}}. \tag{13}$$

Based on the concept that the speed of sound in liquid is inversely dependent on the temperature and increases proportionally with solid fat content [18], the ultrasonic velocity in liquid oil (v_l) is obtained by:

$$v_l = ae^{-bT}\ , \tag{14}$$

where a and b are empirically determined constants [18]. Using Eqs. 13 and 14, the liquid oil bulk modulus can be determined.

Replacing the mixture and the liquid sound velocities, the solid ultrasonic velocity (v_s) is obtained from the Wood equation (Eq. 3). Thus, the solid bulk modulus K_s can be calculated by assuming Eq. 8 for that:

$$K_s = v_s^2\rho - \frac{4}{3}G_s\ , \tag{15}$$

where G_s is the shear modulus of the solid phase.

By substituting K_l and K_s in Eq. 12, the bulk modulus for the mixture is then obtained. Since the sound velocity in the sample is available from the ultrasonic experiment, by using Eq. 8, the shear modulus for the system can be determined.

Determining the Shear Modulus (G_s) of the Solid Phase and Tristearin

In order to determine K_s, the dynamic storage modulus G' was measured by rheology for each sample after crystallizing at the different temperatures. Because it is not possible to obtain a 100% solid fat sample to determine the shear modulus of the solid fraction, determination of the shear modulus by extrapolation to $\phi = 1.0$ could be a good strategy.

Blends of each sample and tristearin with canola oil were prepared in 10% increments from 50 to 100% (w/w). The samples were melted at 120 °C for 10 min to destroy any crystal memory that might influence the crystallization process. The melted samples were placed in PVC molds and stored for one day at 20, 25, and 30 °C. After one day of storage the G' and the SFC were measured. By having the G' at different solid fat contents, the shear modulus for 100% solid (G_s) was determined by extrapolation. The crystal structure of the different dilutions was characterized by powder X-ray diffraction. The melted dilutions were placed on X-ray slides and after 24 h storage at 20, 25, and 30 °C the polymorphism was determined.

Results and Discussion

Determining the Shear Modulus (G_s) of the Solid Phase

Based on X-ray analysis all samples displayed similar polymorphism, characteristic of the β and β' form for all dilutions [19, 20]. Figure 1a shows the XRD patterns for blends of sample 1 with canola oil (from 50 to 100% w/w of the sample) at 25 °C. The same patterns were observed for the other samples at all crystallization temperatures. The only exception in this analysis was 100% tristearin (Fig. 1b) which crystallized in the α polymorphic form. As a result, the value of G' for 100% SSS was not used for the extrapolation calculations.

The experimental measurements of G' as a function of ϕ for 4–9 different SFC values and the extrapolated G' for the solid phase are presented in Fig. 2. Graphs for all the samples at 20, 25, and 30 °C are shown in a, b, and c, respectively. This figure shows a linear relationship between G' and SFC value for all the samples ($0.84 < r^2 < 0.99$). Since at each crystallization temperature the shear modulus in a similar range of SFC was statistically similar for all the samples, we decided to use

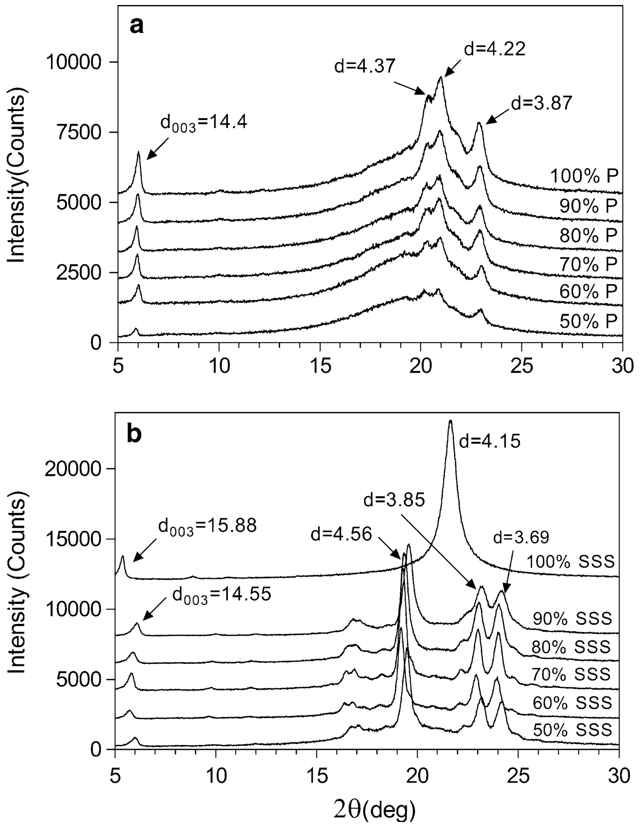
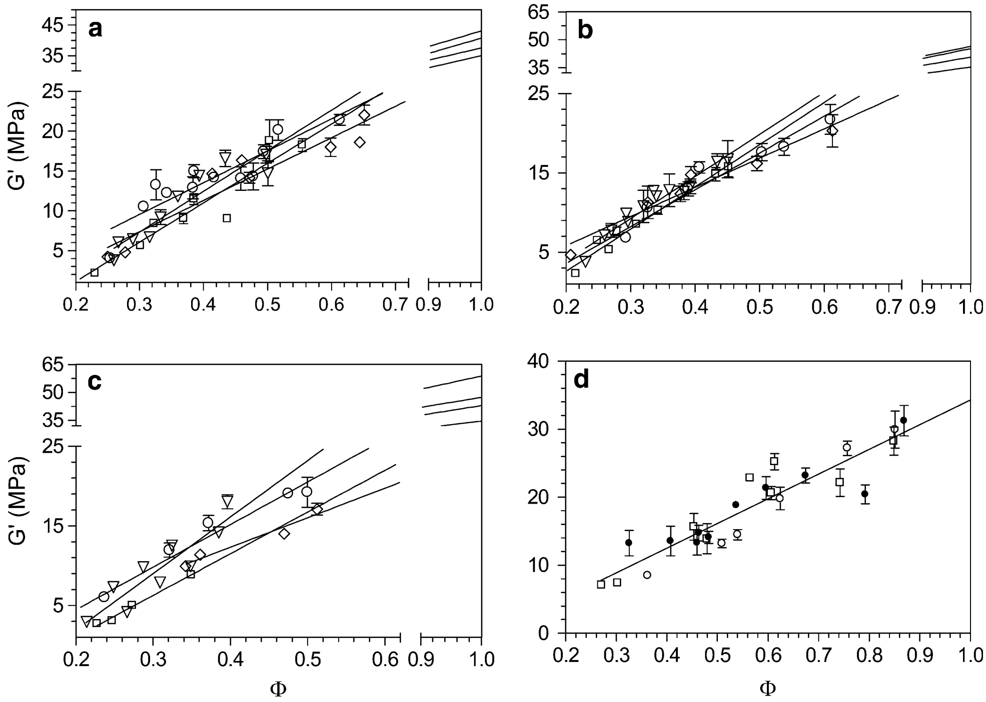


Fig. 1 Powder X-ray diffraction patterns for the different blends of samples with canola oil (50–100% w/w), at 25 °C. (**a**) Sample 1 (*P*) and (**b**) SSS dilutions

a constant shear modulus for the solid fraction, which would be independent of the sample's nature. This approach would be extremely useful because rheology measurements would be eliminated with the ultrasonics measurement method that we propose. Tristearin is a good reference because it is a long-chain saturated fatty acid with a high melting point, 73 °C [20] and a high SFC in the temperature range used in this study. Having a high SFC value can be an advantage for making the range of extrapolation narrow and giving more accurate results. Figure 2d shows the storage modulus of SSS as a function of SFC at 20, 25, and 30 °C. The extrapolated values for the SSS solid fraction elastic modulus at three different temperatures were not significantly different from each other (Fig. 3a) and therefore, we decided to pool all the data from all temperatures to verify that this was also the case for the other samples. We carried out a statistical comparison of the extrapolated shear modulus values for each sample at different temperatures (Fig. 3b–e). We observed that the values were similar for samples 3 and 4 but not for samples 1 and 2. However, the differences in those samples were relatively small; we decided to pool data from the different temperatures. In Fig. 3f one can observe the results of this analysis, using pooled data from all temperatures. Statistical analysis showed that all values were not different except for sample 2 ($P < 0.05$). Considering all these facts we decided to use the tristearin shear modulus at 100% SFC

Fig. 2 Relation between the storage modulus (G') of all the samples, 1 (○), 2 (▽), 3 (◇), and 4 (□) and their mass fraction (ϕ), at three different temperatures, 20 °C (**a**), 25 °C (**b**), and 30 °C (**c**). The shear modulus for 100% solid was extrapolated. (**d**) Tristearin Shear modulus variations with ϕ at three different temperature, 20 °C (●), 25 °C (○), and 30 °C (□)



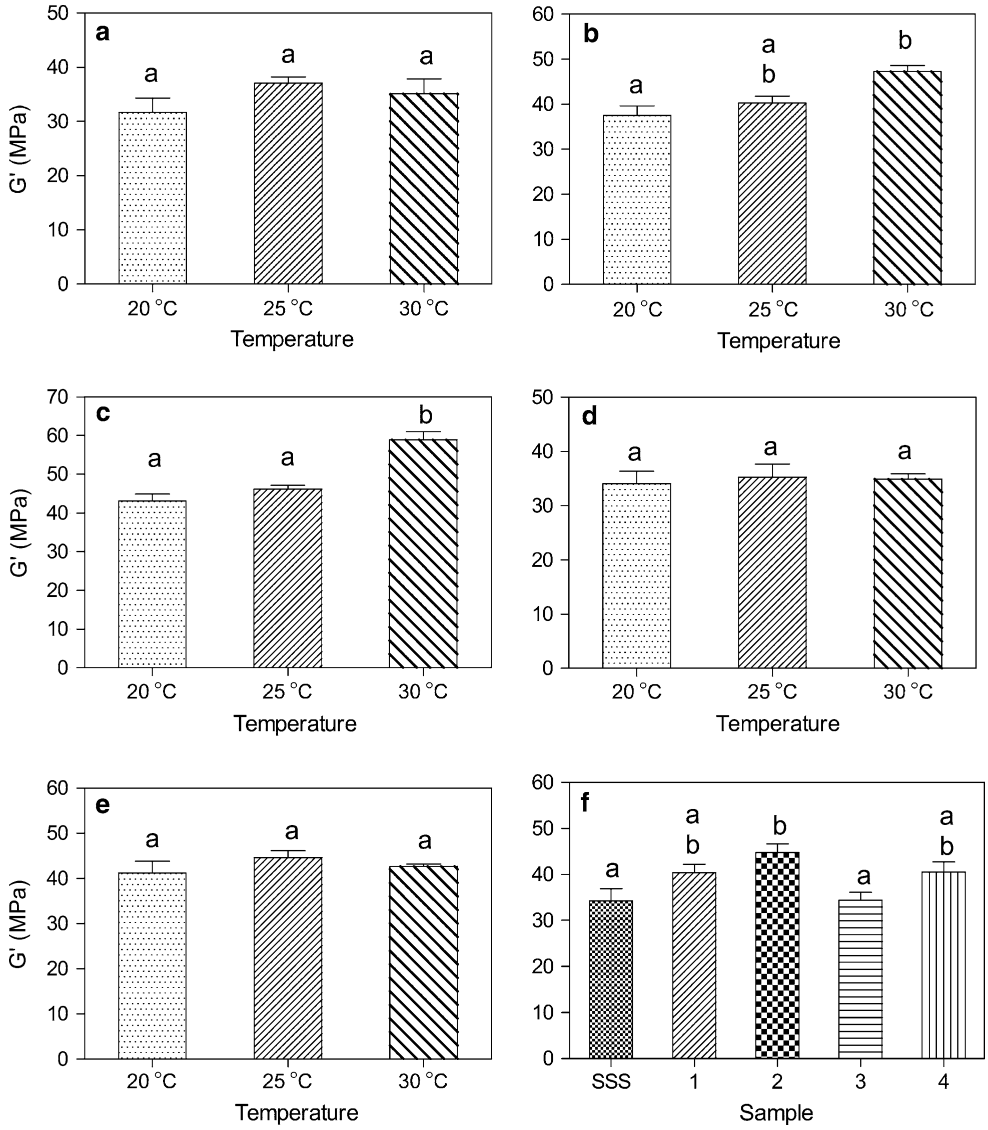


Fig. 3 The statistical comparison of the extrapolated shear modulus values of all samples, SSS (**a**), 1 (**b**), 2 (**c**), 3 (**d**), and 4 (**e**) at 20, 25, and 30 °C. A comparison for the pooled data from all temperatures for each sample is shown in (**f**)

as the modulus of elasticity of the solid phase for all samples under the conditions used in this study.

Shear modulus values of all the samples at different temperatures determined by ultrasonics and rheology are shown in Table 1. The missing shear modulus value for rheological measurements corresponds to very soft samples for which it was not possible to determine G'. This figure shows that there is reasonable agreement between the values of elastic modulus obtained by rheology and ultrasonics for the samples studied. The discrepancies between some of the values can be expected since the SFC between these experiments were similar, but not identical. It is

Table 1 Comparison between the Shear modulus determined using ultrasonics and rheology, for all the samples at 20, 25, and 30 °C

Sample	20 °C		25 °C		30 °C	
	G' (MPa) rheology	G (MPa) ultrasonics	G'(MPa) rheology	G (MPa) ultrasonics	G' (MPa) rheology	G (MPa) ultrasonics
1	4.05 ± 0.05^{a}	4.99 ± 0.11^{b}	3.45 ± 0.32^{a}	4.05 ± 0.41^{b}	$3.0\ 1 \pm 0.34^{a}$	3.29 ± 0.20^{a}
2	3.80 ± 0.23^{a}	3.64 ± 0.03^{a}	3.63 ± 0.11^{a}	3.18 ± 0.07^{a}	2.95 ± 0.09^{a}	2.54 ± 0.02^{b}
3	3.85 ± 0.05^{a}	3.53 ± 0.39^{a}	4.66 ± 0.20^{a}	4.10 ± 0.08^{a}	ND	1.66 ± 0.18
4	2.18 ± 0.16^{a}	2.36 ± 0.21^{a}	2.33 ± 0.16^{a}	2.38 ± 0.07^{a}	2.74 ± 0.09^{a}	2.53 ± 0.78^{a}

ND not determined

generally agreed that the solid content of fats is the major factor determining their rheological behavior [21].

Fractal Dimension Calculation

Fractal dimension, D, can be determined by plotting the natural logarithm of the obtained values of G and ϕ for each sample [22, 23] (Fig. 4). Equation 17 gives the D value from the slope of the line, μ, was derived by linear regression from Eq. 16:

$$\ln G = \mu \ln \phi \,, \tag{16}$$

$$\mu = 1/(3 - D) \,. \tag{17}$$

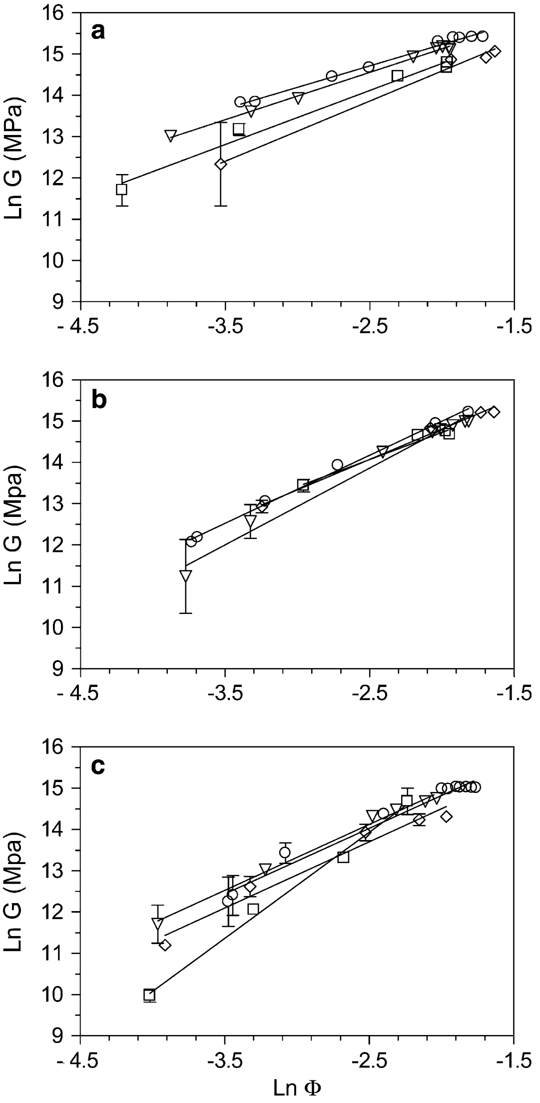


Fig. 4 Ultrasonics results for the determination of the fractal dimension. Natural logarithm of G versus natural logarithm of SFC of each sample, 1 (○), 2 (▽), 3 (◇), and 4 (□) at 20 (**a**), 25 (**b**), and 30 (**c**)°C

Assuming the weak-link regime, the fractal dimensions for all the samples at 20, 25, and 30 °C were calculated by using Eq. 17 and results are presented in Table 2 and Fig. 4.

One can observe from this figure that the ln–ln relationship between G and ϕ is linear with a high correlation coefficient ($0.97 \leq r^2 \leq 0.99$). As described by Marangoni and Narine [4], the fractal dimension is always smaller than the corresponding Euclidean dimension ($D < d$), hence in 3-D space where $d = 3$, $D < 3$. Fractal dimensions determined by rheology in a weak-link regime and ultrasonic methods were compared and results are presented in Table 3. Comparison between these two methods shows that the values obtained by rheology and by ultrasonics are not identical. The variability can be expected since by rheology the measurements were carried out after one day of storage and the fractal dimension corresponds to the final structure of the fat. By ultrasonics, we monitor the early stages of crystallization, and therefore the structure studied is not the final structure.

This also highlights the point that we can measure the mechanical properties of the system during the crystallization process. In addition, the ultrasonic technique provides the means to measure these properties in a soft fat system at low SFC, which is difficult to do by rheology. Moreover, by using ultrasonic spectrometry one could determine the shear modulus and solid fat content simultaneously in an on-line situation in real time.

Table 2 Parameters of the linear fit between the shear modulus (G), obtained by ultrasonics, and solid content for all samples crystallized at 20, 25, and 30 °C

Regression values ($\ln G = \mu \ln\phi + b$)			
Sample	μ	b	R^2
T_c = 20 °C			
1	1.04 ± 0.04	17.32 ± 0.10	0.99
2	1.15 ± 0.03	17.42 ± 0.07	0.99
3	1.46 ± 0.10	17.50 ± 0.24	0.99
4	1.31 ± 0.09	17.39 ± 0.28	0.98
T_c = 25 °C			
1	1.65 ± 0.05	18.30 ± 0.14	0.99
2	1.86 ± 0.09	18.21 ± 0.23	0.98
3	1.47 ± 0.10	17.76 ± 0.24	0.99
4	1.31 ± 0.16	17.35 ± 0.36	0.97
T_c = 30 °C			
1	1.60 ± 0.08	18.01 ± 0.21	0.97
2	1.60 ± 0.07	18.12 ± 0.19	0.99
3	1.60 ± 0.17	17.71 ± 0.51	0.97
4	2.57 ± 0.14	20.37 ± 0.43	0.99

Table 3 Comparison between fractal dimension values obtained by ultrasonics and rheology

Sample	20 °C		25 °C		30 °C	
	D rheology	*D* ultrasonics	*D* rheology	*D* ultrasonics	*D* rheology	*D* ultrasonics
1	2.31	2.04	2.49	2.39	2.47	2.38
2	2.50	2.13	2.46	2.46	2.60	2.38
3	2.40	2.32	2.25	2.32	2.16	2.38
4	2.59	2.37	2.49	2.24	2.65	2.61

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