Comparison of techniques for measuring the rheological properties of labneh (concentrated yogurt)

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The effectiveness of conventional rheological techniques (destructive) and the oscillatory dynamic test (non-destructive) for the study of the physical properties of concentrated yogurt (labneh) was studied. Six different types of labneh (control (cloth bag method), ultrafiltrated (UF)-after and -before fermentation, reverse osmosis (RO)-after and -before fermentation and direct reconstitution from whole milk powder) were examined. Dynamic rheological studies revealed that labneh is a viscoelastic system in which its elastic characteristic is more dominant than its viscous properties. The elastic and viscous attributes of the control labneh were significantly different from the rest of the test samples. In general, the samples with low protein content (RO-after and -before fermentation and direct reconstitution labneh) produced weaker gel structures than their UF counterparts. The penetrometer and viscometer (destructive techniques) failed to reveal expected differences between the samples, and the results did not correlate with the oscillatory dynamic tests. In the light of these results, it could be suggested that dynamic studies are much more reliable than the destructive rheological techniques for the study of the physical properties of labneh.

INTRODUCTION

The yogurt gel is a heat induced acid casein gel, and consists of a permanent network composed mainly of non-covalent protein bonds (eg, hydrophobic and electrostatic binding) as well as covalent thiol-disulphide bonds. There are many factors which affect the physical properties of yogurt, such as the type and number of the protein interactions, the size and shape of the protein network and the distribution of whey proteins and caseins in the aqueous phase. All these factors are affected by the technical applications, such as heat treatment, incubation temperature and pH, type of starter culture, methods of manufacture, as well as type of milk.[1]

Because this combination of factors plays a determinative role in the rheology of the resulting gel, the method which is selected to study the physical properties of yogurt should, ideally, be sensitive enough to measure the viscoelastic properties of the test samples without disturbing the nature of the gel forming components.

Over the last two decades, conventional techniques, such as the Plummet device,[2] the Posthumus funnel,[3] the falling ball,[4] the Namatre vibrator[5] and the Rheomat,[6] have been almost universally accepted for the measurement of the physical properties of set or stirred yogurts, and rotational viscometers, such as the Haake[7] and the Brookfield[8] have become widely used as well.

For set yogurt, different types of penetrometer/consistometer, such as the curd tension-meter,[9] Instron testing machine,[10] Stevens Texture Analyser[11] and the SUR-penetrometer PNR[12] have been widely used to assess the firmness of the body/gel.

However, traditional techniques (1) measure the physical properties of residues of the gel after mechanical agitation, (2) give single point measurements (fixed rate, strain or both) which do not mirror the actual rheological characteristics of yogurt and (3) cannot provide sufficient data to calculate the exact shear rate/strain for stirred yogurt.[13]

Consequently, a great deal of research has investigated the rheology of normal, set- and stirred-yogurts with the non-destructive dynamic rheometer.[1,14–20] In particular, the weak viscoelastic nature of yogurt gel is well established[1,14] and the rheological properties of yogurt can be explained by measuring its viscous and elastic moduli. The most widely employed viscoelastic tests are creep, stress relaxation and dynamic oscillatory testing.[15] However, while the stress relaxation and the creep tests are mostly used to measure the rheological characteristics of stationary gel networks, such as cheese, the dynamic oscillatory tests are more suitable for monitoring the gel properties of time dependent gel systems. Nevertheless, the gel properties of concentrated yogurt have not been subjected to dynamic oscillatory tests. Thus, the aim of the

Original paper.
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present work was to study the effect of increasing total solids by various techniques on the rheology of concentrated yogurt. Additionally, the comparability of traditional techniques and the dynamic oscillatory test for the study of the rheology of concentrated yogurt was investigated.

MATERIALS AND METHODS

Materials

Full fat milk powder supplied from Adams Food Ingredient Ltd (Staffordshire, UK) was used in the production of the test samples. The powder was stored at 4°C until used.

A freeze dried yogurt culture (coded CH-1) from Chr Hansen's Laboratory (Reading, UK) was used in the manufacture of yogurt. The starter was a blend of *Streptococcus thermophilus* and *Lactobacillus delbrueckii* subsp *bulgaricus* in equal portions.

Methods

Six different labnehs were analysed. Labneh made by the traditional cloth bag method, ie, hanging yogurt having 16% total solids in a double layer cheese cloth bag for about 18–20 hours at 4°C until the final product reached 23%, was chosen as the control.

Ultrafiltration was employed either before or after fermentation, and the products are referred to as UF-before fermentation labneh and UF-after fermentation labneh, respectively. The UF cartridge consisted of a bundle of tubular membranes, surface area 0.8 m^2; type ES 625, Patterson Candy, Whitchurch, Hants, UK, polyether sulphone; nominal molecular weight cut-off 25 000 daltons.

Reverse osmosis was applied either before or after incubation, and the products are referred to as RO-before fermentation labneh and RO-after fermentation labneh, respectively. The specifications of the RO membrane were: surface area 1.2 m^2; type ZF 99, Patterson Candy, membrane composed of polyether sulphone.

During the application of membrane processes (both UF and RO) the temperatures of milk and yogurt were maintained at 50°C and 45°C, respectively. The inlet and outlet pressures for the UF plant were 4 and 2 bar, respectively; the RO plant was operated at 20 bar for yogurt and 25 bar for milk systems.

The last treatment was produced by reconstituting the required amount of full cream milk powder in softened water at 40°C (direct reconstitution labneh).

The yogurt making procedure proposed by Tamime and Robinson[2] was followed. The initial total solids level of the milks and yogurts was 16%, and after concentration 23% total solids; the standard heat treatment was at 85°C for 20 minutes. Acidification was achieved by inoculation with the starter culture (2% v/v) at 43°C. Incubation was halted when the pH dropped to 4.3 for samples concentrated postincubation, and 4.0 for previously concentrated samples; the final pH of all samples was 4.0.

Determination of the gel firmness was by means of a Stevens Texture Analyser (C Stevens and Son Ltd, Herefordshire, UK), fitted with a chart recorder model BS 271. A cylindrical probe (1.2 cm in diameter and 4.5 cm in height) was used. The speed of the probe, penetration depth and chart speed were 0.5 mm s^{-1}, 15 mm and 30 mm min^{-1} at 200 mV, respectively.

The apparent viscosity was measured with a Brookfield rotational viscometer (Brookfield Engineering Laboratories, Inc, Massachusets, USA), model LVT, with a helipath stand. Approximately 350 ml of yogurt sample was analysed at low rotation speed, ie, 0.6 rpm with T-bar spindles.

The dynamic rheological studies were carried out using a stress-controlled rheometer (Rheotech International, UK). The rheometer was set up with a parallel-plate (10 mm radius and 1 mm gap setting). The temperature of the samples was maintained at 25°C using a circulating water system.

Statistical evaluations were completed using the Excel software program, and statistically significant groups were determined by the Duncan test.

RESULTS AND DISCUSSION

Preliminary studies indicated that labneh (concentrated yogurt) is a typical weak viscoelastic gel system in which the elastic modulus (G′) is greater than the loss modulus (G″).

Variations in the viscoelastic components of the test samples over the range of amplitudes applied are presented in Figs. 1 and 2. Strikingly, none of the treatments produced the same gel properties as the control sample, in spite of the fact that UF-before and -after fermentation labnehs had similar chemical composition to the control (see Table 1). This significant difference between the control and the rest of the test samples may result from the compact structure of the control sample resulting from gravity drainage. UF-before and -after fermentation labnehs to some

TABLE 1

Chemical composition of the test samples; all figures as g/100 g of sample

Samples	*Total solids*	*Protein*	*Lactose*	*Fat*	*Total mineral*
Control	23.31	8.0	5.16	9.18	0.79
UF-before	22.44	8.25	5.21	8.20	0.78
UF-after	22.64	8.13	5.50	8.45	0.86
RO-before	23.22	6.82	8.98	6.25	1.08
RO-after	22.22	6.38	8.84	6.60	1.00
Direct reconstitution	22.50	6.38	8.72	6.10	1.30

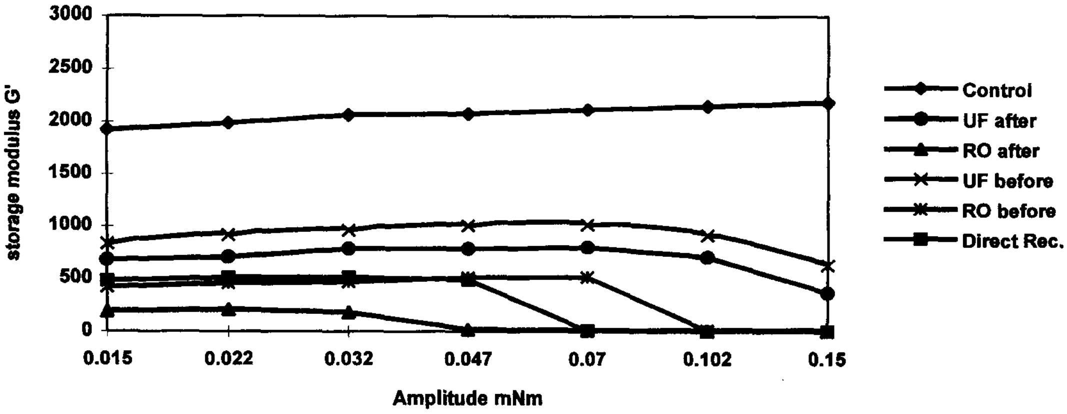

Fig. 1. Typical storage modulus pattern of labnehs after overnight storage. Results are the average of eight separate runs (standard errors less than symbol dimensions). Test conditions are: amplitude range 0.015–0.15 mNm, frequency 0.25 Hz, parallel-plate (10 mm radius and 1 mm gap setting), 25°C measuring temperature. Direct Rec. = direct reconstitution.

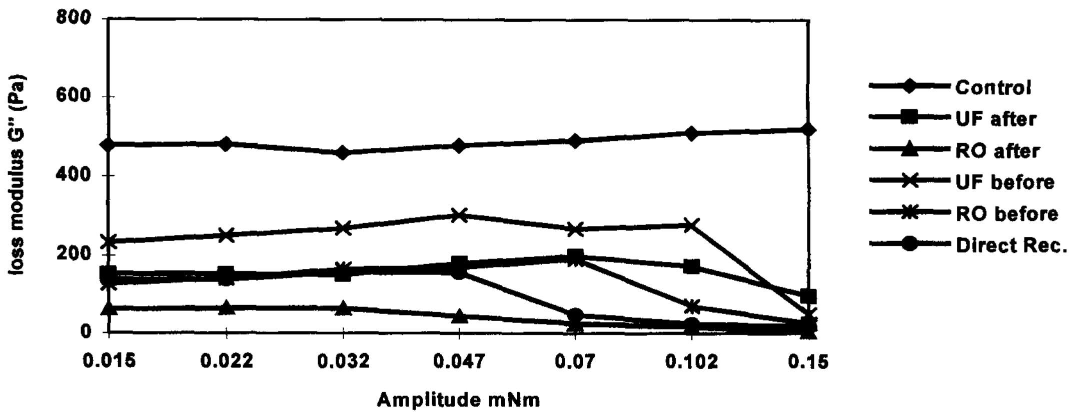

Fig. 2. Typical loss modulus pattern of labnehs after overnight storage. Results are the average of eight separate runs (standard errors less than symbol dimensions). Test conditions are: amplitude range 0.015–0.15 mNm, frequency 0.25 Hz, parallel-plate (10 mm radius and 1 mm gap setting), 25°C measuring temperature.

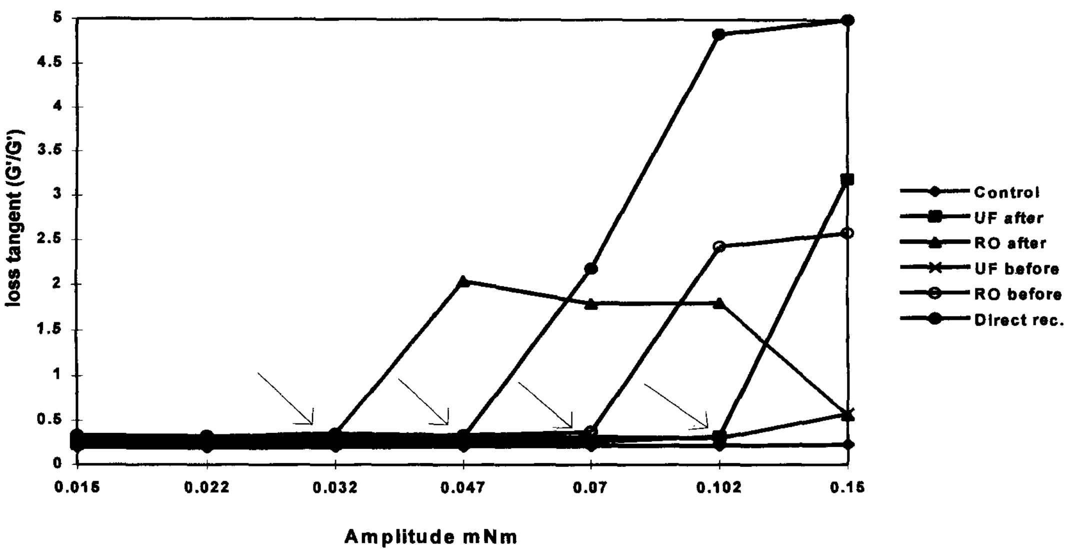

Fig. 3. Variation in loss tangent values of the samples as a function of amplitude. Arrows indicate the breaking point of structure of each sample.

extent kept their structural integrities against increasing shear. However, the rest of the samples broke down at some point within the range of amplitude applied. After the gel structure broke down, the viscous character of the samples became dominant. This effect is better seen in Fig. 3, which shows the variation in loss tangent values ($\tan \delta = G''/G'$) as a function of amplitude. Roefs[21] suggested that the tan δ value is related to the nature of the bonds forming the protein network and the relative importance of the different types of bond rather than to the spatial distribution of protein junction points. As the type of protein and the gelation conditions were identical in each system, it is reasonable to deduce that the different treatments for increasing total solids led to the differences in the levels of protein and hence relative dominance of the bonds forming the protein network.

In terms of the elastic and loss moduli, UF-before and -after fermentation labnehs were similar. The direct reconstitution and RO-before fermentation labnehs had very similar gel properties, but were weaker than the control and UF samples. The destructive effect of excess mechanical force (20 bar pressure) on the gel structure of the RO-after fermentation labneh was remarkable. This sample had a very weak and sponge-like structure, giving it an atypical appearance for labneh.

The firmness of the samples examined by the Stevens Texture Analyser is illustrated in Fig. 4. The results indicated that there was a clear difference between the control and the rest of the test samples, but the penetrometer failed to reveal possible differences between the other samples. Thus the elastic moduli of UF treated samples (Fig. 1), for example, were some 4–5 times higher than that of RO-after fermentation labneh, but the gel strengths of these samples were not significantly different ($p > .05$).

The control and the UF-before fermentation labnehs had more viscous characteristics than the others (Fig. 5), but little correlation between the penetrometer and viscometer measurements was found. This lack of correlation corroborates the theory that destructive techniques cannot mirror the actual rheology of yogurt, which has a viscoelastic nature. In other words, each penetration into or rotation in a gel network causes a breakdown in the elastically effective bonds, and the procedure thus fails to measure the actual physical characteristics of the gel. Additionally, as compared to literature data on model casein gels and microstructural findings on yogurt,[11] the results imply that

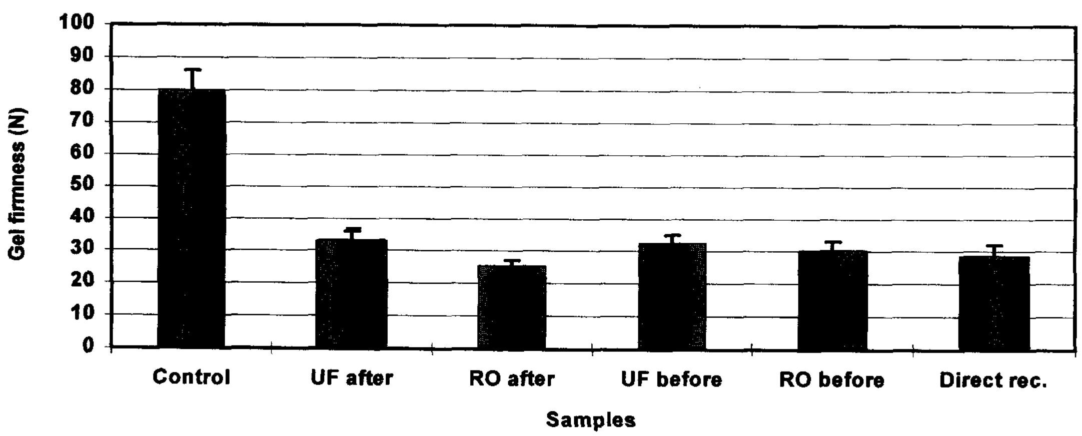

Fig. 4. Gel firmness of test samples measured by Stevens Texture Analyser. Results are the average of three separate runs.

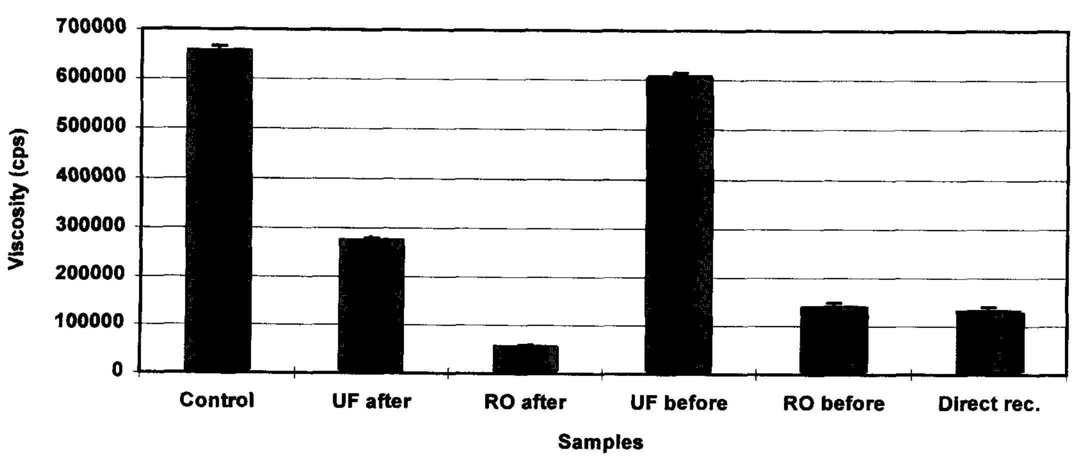

Fig. 5. Viscosity values of the test samples after overnight storage at 4°C. Results are the average of three separate runs.

yogurt is a heterogeneous particulate gel. Therefore, single point measurements are insufficient to represent the rheology of the entire gel.

CONCLUSIONS

Dynamic rheological tests (oscillatory tests) are more suitable for the study of the rheology of concentrated yogurt than traditional techniques. The key point is to preserve the natural form of the gel as long as possible. Once the gel structure is disturbed, it is rarely possible to re-form the gel structure in the same way again, because yogurt is a metastable gel and any change in its enthalpic/entropic nature creates irreversible deformation. Thus, any kind of destructive effect may lead to atypical physical properties in the yogurt, and provide erroneous results.

B Ozer thanks the Turkish Higher Educational Council (YOK) and Harran University (Turkey) for financial support.

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