RHEOLOGICAL PROPERTIES OF CONCENTRATED YOGHURT (LABNEH)

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ABSTRACT

The effect of six techniques for the elevation of the total solids on the rheological properties of labneh were investigated using controlled-stress dynamic rheometer. All samples exhibited a weak viscoelastic gel structure with the storage modulus (G') higher than the loss modulus (G") over all of the measured range. None of the experimental materials produced the same overall gel strength as the control made by draining with traditional cloth-bags. The changes in the storage modulus (G') as a function of amplitude sweep were mirrored by changes in the loss modulus (G"). Considerable differences in the loss tangent (G"/G') values of the various materials were observed at higher stress amplitudes. Rheological differences in the overall gel strength at low amplitude and frequency suggest that, although the type of protein interactions in each case may be similar, there are differences in the degree of interactions. Subsequent breakdown at higher amplitudes and frequencies suggests that the overall domains of the treated proteins may have been reduced, and that different methods of manufacture may be producing materials that have different space occupancy in the gel.

INTRODUCTION

Labneh (a concentrated fermented milk product) has increased in popularity during recent years. Its increasing economic importance has been achieved as a result of its perceived nutritional benefits and storage characteristics (Benezech

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and Maingonnat 1994). However, much of its consumer acceptability is dependent on its sensory properties which, in turn, seem to be heavily dependent on the method of processing of the material. It should be possible to use rheological data to modify and control the process conditions to give a product with the quality attributes desired by the consumer. Over the last 10–15 years, conventional methods such as the Posthumus funnel (Posthumus 1954), the Plummet device (Tamime and Robinson 1985) and the falling ball apparatus (Bottazi 1976) have been almost universally accepted for the measurement of physical properties of stirred yoghurt, and rotational viscometers, such as the Brookfield (Abrahamsen and Holmen 1980) and the Haake (Parnell-Cluines *et al.* 1986) viscometers, have also become widely used.

For set yoghurt, the firmness of the body/gel is considered to be the best parameter to assess the physical quality of the system. The consistometer or penetrometer apparatus have been widely used for this purpose. The penetrometer test measures the force required to push a probe into a food or depth of penetration but, as a wide variety of probes, penetration depths and temperatures may be used, it is almost impossible to compare results between laboratories. In order to standardize the results and to improve the reproducibility of the data derived from penetrometer-type measurements, a computerized Texture Profile Analyzer (TPA) is usually employed to study the texture of set-type yoghurts (Benezech and Maingonnat 1994). All of the techniques mentioned above have some advantages and disadvantages. Yoghurt is defined as a weak gel system and weak gels are unable to keep their structural integrities during high shear; even the lowest possible shear may disturb the gel when traditional penetrometer or rotational techniques are used. Dynamic oscillatory testing is a technique that is very appropriate for investigating the structure of a viscoelastic material such as yoghurt (Steventon *et al.* 1990; Xiong and Kinsella 1991).

Several studies have investigated the viscoelastic properties of yoghurt-type materials; (1) the effect of starter culture (Rohm and Kovac 1994); (2) different milk types (Biliaderis *et al.* 1992; Vlahapoulou and Bell 1993); (3) rheological characteristics of stirred yoghurt (Skriver *et al.* 1993; Rohm 1992; Ramaswany and Basak 1991, 1992). Horne (1993) found that these dynamic measurements showed good correlation with certain rheological measurements of gel strength (elastic modulus) in set yoghurts. In spite of the fact that rheological characterization of normal set and stirred yoghurts have been investigated by dynamic testing, there has been no study of the rheological properties of labneh. Labneh is traditionally manufactured using a cheese cloth bag for concentrating yoghurt after overnight refrigeration. Because it needs a long processing time (2–3 days), is labour-intensive and unhygienic, this method of manufacture is not suitable for large scale processing (Tamime *et al.* 1989). During the last decade, mechanized processes such as ultrafiltration (UF) and reverse osmosis (RO) have become wide-

ly used in the production of labneh. Especially, the concentration of warm yoghurt to the desired total solids level by UF after fermentation, is being currently practised intensively. So far, all mentioned manufacturing techniques have been studied individually and because different rheological techniques have been used in each study, it is difficult to reach a fair judgement about the physical qualities of the labnehs. The present study was designed to compare different manufacturing techniques in terms of physical characteristics of resulting products brought about by application of those techniques.

EXPERIMENTAL

Materials

Full cream milk powder supplied from Adams Food Ingredients Ltd. (Staff. UK) and stored at 4C was used. A freeze-dried commercial yoghurt culture (coded CH-1) from Chr. Hansen's Laboratory (Reading, UK) was used to prepare the yoghurt. The starter was a blend of *Streptococcus thermophilus* and *Lactobacillus delbruckii* sub-sp *bulgaricus* in equal proportions. The labneh samples were stored overnight at 4C before testing.

Methods

Yoghurt-making Process. Six different techniques for raising the level of total solids were applied: (1) traditional cloth bag system (control)-drainage was achieved by holding yoghurt (16% w/v solids) in bags of double layer cheese cloth at 4C for 18–20 h, (2) direct reconstitution of full-cream milk powder to 23% (w/v) total solids (direct reconstitution labneh), (3) reverse osmosis (RO) after and before fermentation, and (4) ultrafiltration (UF) after and before fermentation. Pilot scale UF and RO plants were employed for the concentration of milk and yoghurt to 23% total solids. The UF cartridge consisted of a bundle of tubular membranes and the specifications of the UF membranes were: surface area 0.8 m^2, type ES 625 (Patterson Candy, Whitchurch, Hands., UK), membranes composed of polyether sulphone, and nominal molecular weight cut-off 25,000 dalton; and the RO membranes were: surface area 1.2 m^2, type ZF 99 (Patterson Candy International), membrane composed of polyether sulphone. For the membrane processes, the temperatures of milk and yoghurt were 50C and 42C, respectively; the RO plant was operated at 20 bar for yoghurt and 25 bar for milk systems. For all samples, a standard yoghurt making procedure proposed by Tamime and Robinson (1985) was followed. The initial total solids content of the control, UF-after fermentation and RO-after fermentation labnehs was 16% (w/v), and the final total solids content for all end products was ~23%. The standard heat treat-

ment was 85C for 20 min and, after cooling, the milks were inoculated with a liquid starter culture (2% v/v) at 42C. Incubation was halted when the pH dropped to 4.3 for samples concentrated post-incubation, and 4.0 for previously concentrated samples; the final pH of all samples was 4.0. In the determination of the effect of the level of protein on the gel properties of labneh, milks were concentrated with UF up to 10% protein level. Then, serial dilutions from 1 to 9% protein content were made using the same permeate in order to keep the ionic environment identical in each sample.

Chemical Analyses. Chemical analyses were done according to the methods outlined in the British Standard Institution as follows: protein, BSI Part 10:1 (Kjeldahl N × 6.38) (BSI 1988); fat, Gerber method BSI 696: Part 2 (BSI 1989) and ash, BSI 1741: Part 9 (BSI 1988).

Dynamic Rheological Measurements. Dynamic rheological tests were performed with a Rheotech International controlled-stress oscillator rheometer using parallel plate geometry (10 mm plate radius and 1 mm gap setting). All samples were stirred by means of a controlled-speed stirrer at low speed before measurement in order to standardize between set and stirred labnehs. Each sample was loaded into the rheometer and allowed to relax and equilibrate to measuring temperature (2 min, 25C) prior to testing. A strain sweep used angular frequency, ω, from 10^{-3} to 10^{1} Hz at 0.07 mNm torque and sweeping amplitude from 1.5×10^{-2} to 1.5×10^{-1} mNm at 0.25 Hz. The rheometer was thermostatted by a water circulator connected to a temperature controller (Haake, UK). Measurements are the average of five replications and from each replication ten readings were taken. Standard errors are less than $\pm 8\%$ of average values for each sample.

Statistical Analyses. Differences between the samples were tested by single factor variation analysis using the data analysis tool kit that is supplied with Microsoft® Excel spreedsheet Version 5.0. Statistical differences between the groups were determined by DUNCAN test.

RESULTS AND DISCUSSIONS

Chemical composition of labnehs are illustrated in Table 1. In the RO and Direct reconstitution labnehs, the protein and fat contents increased relatively less than in the control and UF treated samples. The lactose concentration was reduced to 4.53 to 4.16% in the control and UF samples, whereas in the RO and direct reconstitution labnehs, this component increased with the concentration factor.

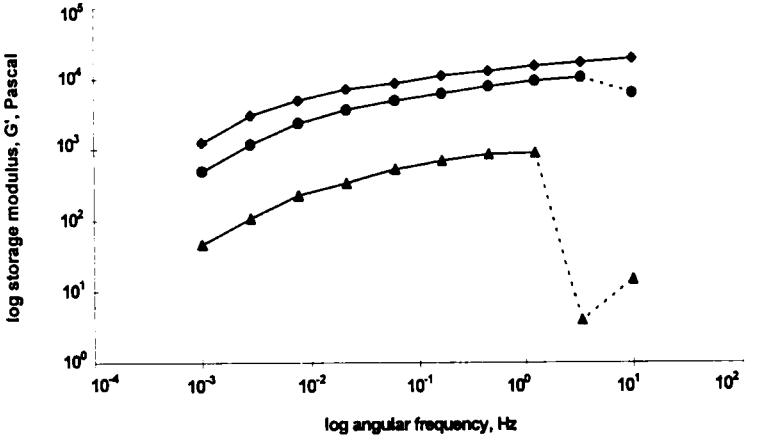
TABLE 1.
CHEMICAL COMPOSITION OF LABNEHS TESTED

Samples	Total solids %	Protein %	Lactose * %	Fat %	Ash %
Control labneh	23.31	9.20	4.16	9.18	0.79
UF after fermentation labneh	22.64	8.80	4.53	8.45	0.86
RO after fermentation labneh	22.22	6.38	8.24	6.60	1.00
UF before fermentation labneh	22.24	9.00	4.26	8.20	0.78
RO before fermentation labneh	23.22	6.82	9.07	6.25	1.08
Direct reconstitution labneh.	22.50	6.38	8.72	6.10	1.30

(*) Lactose was determined by difference.

The rheological analyses showed the labneh was a typical weak viscoelastic gel whose storage modulus (G′) was greater than loss modulus (G″) over the measured range and showing a frequency-dependence. None of the samples produced the same gel properties as the control sample, in spite of the fact that UF-treated samples (after and before fermentation) had approximately the same chemical composition as the control. A statistically significant difference was found between the samples ($P < 0.05$). Overall, the control was the stiffest sample followed by UF treated samples. RO and direct reconstitution applications created weaker structures in the final gels than control and UF treated ones. A structural breakdown was observed in all samples, with the exception of the control, at different point of frequency sweep (Fig. 1a and b). No slipping was observed in the measuring system. In general both network moduli increased with frequency in the linear region and higher protein content samples had higher storage and loss moduli. These profiles are consistent with the behaviour of weak viscoelastic gels (Ferry 1980; Benezech and Maingonnat 1994). Inhomogeneous particulate gels are generally more frequency-dependent than their homogeneous counterparts (Stading 1993), *i.e.*, the distribution of gel-forming components in the aqueous phase influences the frequency dependency of the gel. In the present case, a considerable frequency-dependency in all labnehs was evident. Therefore, it is justified to assume that the labnehs have an inhomogeneous particulate structure. The increase of network moduli with frequency, ω, indicates a relaxation of protein bonds during the period of measurements. Storage modulus (G′) is determined by the number and/or strength of nonrelaxing protein bonds, whereas loss modulus is determined by rapidly relaxing bonds (Roefs 1986). In the present case, until gel structure was broken down, the storage modulus was always higher than the loss modulus in all samples indicating that nonrelaxing bonds

(a)



(b)

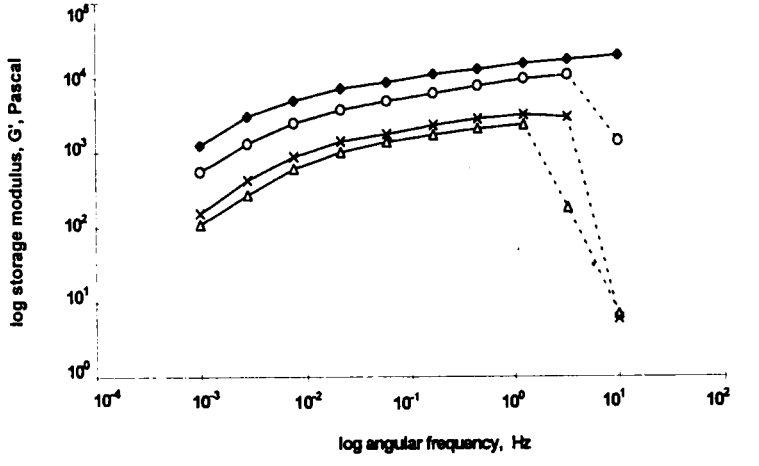


FIG. 1. STORAGE MODULUS OF SAMPLES CONCENTRATED (a) POST-FERMENTATION, (b) PRIOR TO FERMENTATION AS A FUNCTION OF ANGULAR FREQUENCY

Standard deviations are less than symbol dimensions. (♦) control, (▲) RO-after fermentation labneh, (●) UF-after fermentation labneh. (△) RO-before fermentation labneh, (○) UF-before fermentation labneh, (×) Direct reconstitution labneh.

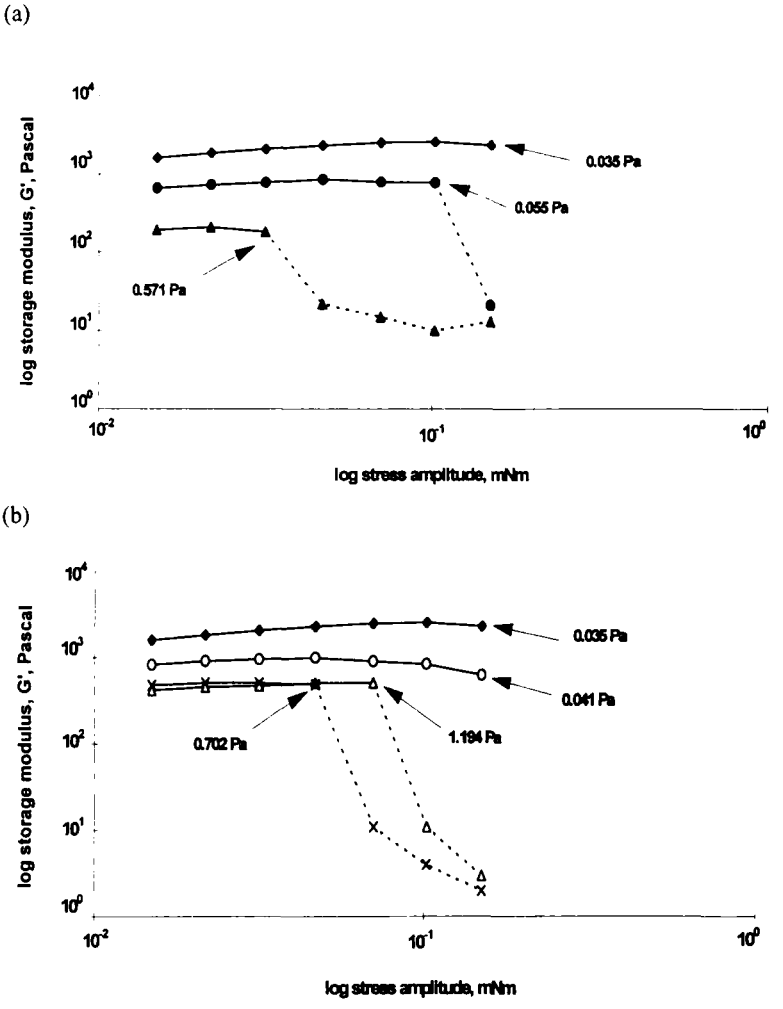


FIG. 2. STORAGE MODULUS OF SAMPLES CONCENTRATED (a) POST-FERMENTATION, (b) PRIOR TO FERMENTATION AS A FUNCTION OF AMPLITUDE SWEEP

Standard deviations are less than symbol dimensions. (♦) control, (▲) RO-after fermentation labneh, (●) UF-after fermentation labneh, △) RO-before fermentation labneh, (○) UF-before fermentation labneh, (÷) Direct reconstitution labneh. Points indicated give the amplitude strain at that stress.

dominated over rapidly breaking and reforming weak bonds, and this domination depends upon macromolecule (casein) concentration because the higher protein content samples had the higher storage moduli.

A linear viscoelastic region was evident for all samples. With the exception of the traditional and UF-before fermentation labnehs, a structural breakdown was observed at some point of the amplitude applied. Data for comparison purposes were also collected in the form of amplitude sweeps which were considered to be another indicative parameter of the structure. The control, UF-before and, to some extent, UF-after fermentation labnehs kept their structural integrities against increasing shear (Fig. 2a and b). However, the rest of the samples broke down at some point within the range of the amplitude applied. Corresponding strain amplitude data as a function of stress amplitude applied are presented in Table 2. The changes in the storage modulus (G') as a function of amplitude were mirrored by changes in the loss modulus (G") (Fig. 3a and b). This gave rise to considerable differences in the phase angle (tan δ) values of the various materials, especially at higher amplitudes (Fig. 4a and b).

Rheological properties of the control sample should be evaluated separately from the others, because physical characteristics of this sample came into being after fermentation was halted. However, in the other samples, rheological

TABLE 2.
STRAIN AMPLITUDE DATA OF LABNEHS AS A FUNCTION OF STRESS

	Strain amplitude (Pascal)					
Stress Amplitude mNm	Control labneh	UF after fer.labneh	RO after fer. labneh	UF before fer.labneh	RO before fer. labneh	Direct reconstitution labneh
0.015	0.006	0.015	0.055	0.010	0.021	0.021
0.022	0.007	0.019	0.101	0.013	0.027	0.027
0.032	0.009	0.025	0.571*	0.017	0.039	0.040
0.047	0.012	0.029	1.302	0.021	0.702*	0.054
0.070	0.016	0.037	3.251	0.024	1.053	1.194*
0.102	0.029	0.055	6.137	0.032	2.262	2.757
0.150	0.035	0.977*	12.416	0.041	4.354	5.604

(1) Stars indicate the strain at the gel breaking point.
fer. : fermentation

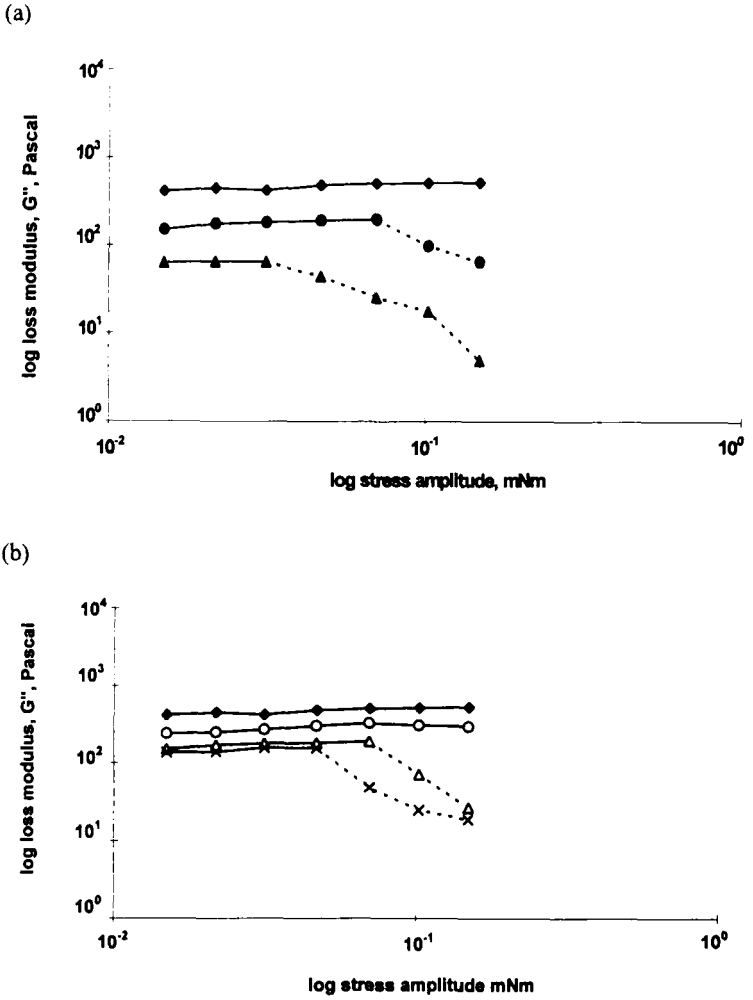


FIG. 3. LOSS MODULUS OF SAMPLES CONCENTRATED (a) POST-FERMENTATION, (b) PRIOR TO FERMENTATION AS A FUNCTION OF AMPLITUDE SWEEP

Standard deviations are less than symbol dimensions. (♦) control, (▲) RO-after fermentation labneh, (●) UF-after fermentation labneh, (△) RO-before fermentation labneh, (○) UF-before fermentation labneh, (×) Direct reconstitution labneh.

characteristics were determined during the fermentation stage. UF-after and RO-after fermentation labnehs were produced after the fermentation stage had finished but, because the samples were not cooled down after fermentation and membrane techniques were applied at incubation temperature, it may be reasonable to assume

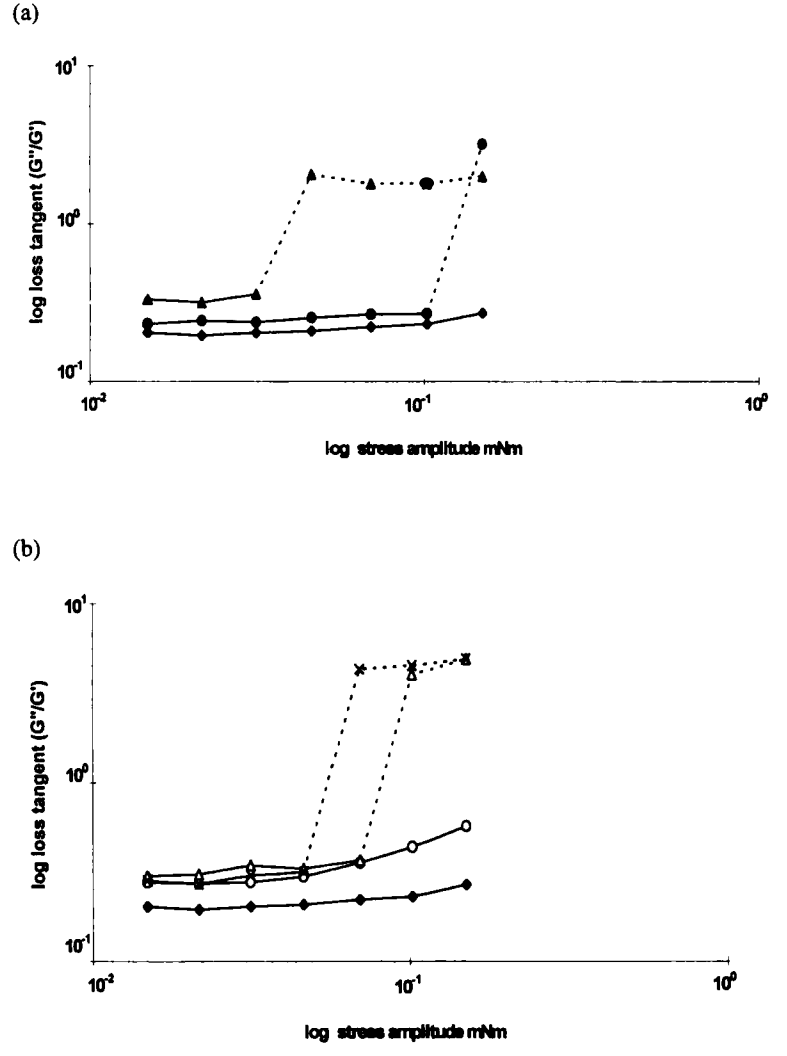


FIG. 4. LOSS TANGENT OF SAMPLES CONCENTRATED (a) POST-FERMENTATION, (b) PRIOR TO FERMENTATION AS A FUNCTION OF AMPLITUDE SWEEP

Standard deviations are less than symbol dimensions, (♦) control, (▲) RO-after fermentation labneh, (●) UF-after fermentation labneh. (△) RO-before fermentation labneh, (○) UF-before fermentation labneh, (×) Direct reconstitution labneh.

that incubation continued during membrane applications. In UF-after fermentation labneh, along with the increase in the protein level, the distribution of the protein particles may have been regulated by UF application. Mild pressure driven

effect of UF may have led to a more homogeneous distribution of protein network and, consequently, stress carrying strands over the continuous phase. It is fair to believe that UF application to warm yoghurt at low pressure (4 bar) may stimulate the increase in the number of smaller conglomerates and, therefore, number of stress carrying effective bonds. On this application, protein-protein bonds which are jammed in a large conglomerate, may be distributed homogeneously without disturbing the gel. Yet, RO application to warm yoghurt at high pressure (20 bar) caused an unrecoverable damage in the gel structure of RO-after fermentation labneh, giving in it an atypical appearance for labneh.

UF-before fermentation had significantly higher storage modulus (G′) than direct reconstitution and RO-before fermentation labnehs. This is probably due to the variation in the protein contents (9% vs 6.38–6.82%, respectively). As a result of more protein-protein interactions at higher protein levels, a much denser and stronger gel structure can be expected. This is seen in Fig. 5 showing the effect of protein level on the network moduli. Protein levels were adjusted from 1 to 9% by diluting the UF-milk having 10% protein content with the same permeate in order to keep the ionic strength relatively equal in each sample. An increase in both moduli with the increase in protein level is seen. A critical protein level had to be exceeded to form a gel (minimum 3% protein). In general, network moduli in a gel are more related to the particle concentration, however, the phase angle values are more dependent on the nature of bonds between these particles. In the present case, in spite of the UF-before fermentation labneh had much higher G′ and G″ values, insignificant differences were obtained between the same samples ($P > 0.05$).

The differences in the overall gel strength at low amplitude suggests that, although the type of protein-protein interactions (mainly whey protein/κ-casein interactions) in each case may be similar, there are differences in the degree of interaction. Subsequent breakdown at higher amplitudes suggests that the overall domains of the proteins may have been reduced and that there was a different "space occupancy" of the proteins in the gel material. The breakdown as a function of amplitude persisted even during storage of the materials for up to 14 days, suggesting that any changes produced during nontraditional methods of manufacture were essentially permanent and nonreversible.

CONCLUSION

Labneh is a typical weak viscoelastic gel whose properties are strongly affected by the methods of manufacture. Different gel behaviour at lower and higher amplitudes indicates that the type of the interactions that lead to the gel network are similar, but degrees of both covalent (SH/S-S exchange) and noncovalent

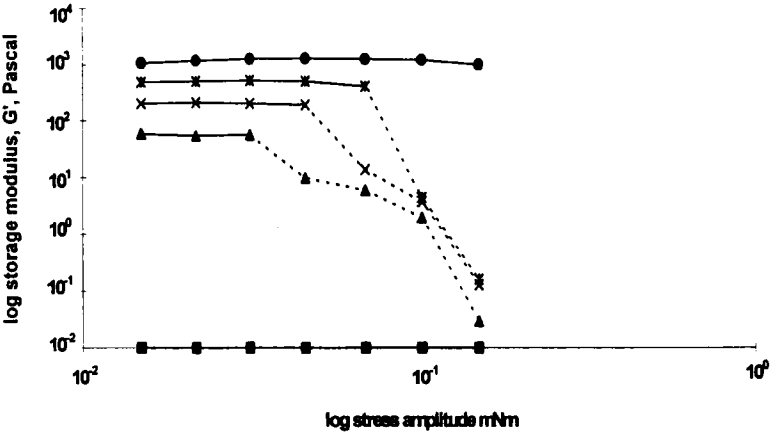


FIG. 5. EFFECT OF PROTEIN CONCENTRATION ON THE GEL PROPERTIES OF LABNEH MADE FROM UF MILK

No gel formed at the protein concentration less than 3%. Standard deviations are less than symbol dimensions. (■) 1%, (♦) 2%, (▲) 3%, (×) 5%, (*) 7%, (●) 9% protein.

(hydrophobic, electrostatic) interactions may be different as a result of the difference in protein level. The share of different gel-forming interaction forces in the formation of labneh gels is being currently investigated. Preliminary results indicate there are some differences between the samples regarding the level of protein interactions, especially the level of thiol-disulfide exchange reactions between whey proteins and κ-casein. Overall, the results suggest that UF applications can be used as an alternative method to the traditional labneh-making process (confirming the proposal of Tamime *et al.* 1989). The other production methods did not give gel properties that were close to those of typical labneh material.

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