

Destructive Effects of Classical Viscosimeters on the Microstructure of Yoghurt Gel

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Abstract: The effects of empirical viscosity measuring techniques that are commonly used in the determination of the viscosity of weak viscoelastic gels such as yoghurt on the microstructure of yoghurt were investigated by scanning electron microscopy (SEM). In the measurement of viscosity, Brookfield and Haake viscosimeters were employed at 2 different measuring periods (15 and 30 s). The results obtained indicated that the increase in the measuring period (15 s versus 30 s) stimulated the further destruction of the casein gel network. Additionally, the Brookfield viscosimeter was more destructive than the Haake viscosimeter. In general, lower viscosity values were in good harmony with higher destruction in the gels, as shown by SEM micrographs.

Key Words: Viscosity, Yoghurt, Destruction

Klasik Viskozimetrelerin Yoğurt Mikroyapısı Üzerindeki Tahrip Etkisi

Özet: Bu çalışmada, yoğurt benzeri zayıf viskoelastik jellerin pıhtı stabilizasyonunun ölçümünde sık kullanılan empirik viskozite ölçüm tekniğinin yoğurdun mikroyapısı üzerine etkileri tarayıcı elektron mikroskobu (scanning electron microscopy, SEM) yardımı ile incelenmiştir. Viskozite ölçümlerinde Brookfield ve Haake viskozimetrelerinden yararlanılmış ve iki farklı ölçüm süresi (15 ve 30 saniye) deneme kapsamına alınmıştır. Elde edilen sonuçlar ışığında, ölçüm süresinin artması ile birlikte kazein jellerinin pıhtı tahribatı da artış göstermiştir. Ayrıca, Brookfield viskozimetresinin Haake viskozimetresine oranla daha fazla deformasyon etkisi yarattığı da belirlenmiştir. Genel olarak, SEM fotoğraflarından da görüldüğü üzere, düşük viskozite değerleri ile jel yapısındaki parçalanma düzeyi arasında önemli bir ilişkinin varlığı saptanmıştır.

Anahtar Sözcükler: Viskozite, Yoğurt, Parçalanma

Introduction

Rheology is a science related to the flow of fluids and deformation of matter (Prentice, 1992; Steffe, 1992). In many disciplines of science and technology, including geology and mining (Cristescu, 1989), construction technology (Tattersall and Banfill, 1983) and plastics processing (Dealy and Wissburn, 1990), rheological tests are used. However, compared with other disciplines of science and technology, the food industry (especially the dairy industry) is the area in which rheological tests are most extensively employed. Since milk is able to be converted into many milk-based products with different physical properties, rheological studies in the dairy industry are of special importance. Understanding the rheological behavior of dairy products is important in the

following steps during manufacturing: (i) quality control of ingredients and finished products, (ii) design and evaluation of processing equipment, unit operations and process parameters, (iii) adjustment of time x temperature x flow rate selection of fluid dairy products and (iv) characterization and development of dairy products for consumer acceptability, and elucidation of the structure and relationship among structure and textural properties (Rao, 1977; Rao and Skinner, 1986; Kokini, 1992; Barbosa-Cánovas et al., 1993).

In the evaluation of the rheological properties of a viscoelastic gel, it is necessary to define both elastic and viscous characters at the same time. During the last 20-30 years, the rheological evaluation of viscoelastic dairy foods has been achieved by empirical techniques such as

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the Plummet device (Tamime and Robinson, 1985), Posthumus funnel (Posthumus, 1954), falling ball apparatus (Bottazi, 1976), Namatre vibrator (Parnell-Cluiness et al., 1988), Rheomat (Danenberg and Kessler, 1988), Instron device (Schmidt et al., 1985) and Stevens texture analyzer (Tamime et al., 1989). The use of Haake (Parnell-Cluiness et al., 1986) and Brookfield (Abrahamsen and Holmen, 1980) viscosimeters in the determination of viscosity is also common. However, although these devices are cheap and easy to handle, they produce rheological data after the gel structure has been degraded and, therefore, it is not always possible to make a fair judgment regarding the rheology of the dairy food under investigation. In addition, since there is a high risk of obtaining non-reproducible and, to some extent, erroneous results from empirical viscosimeters, the decision-making process for the selection of unit operations and equipment design may become rather difficult.

Thus, in the present study, it was intended (i) to demonstrate the destructive effects of 2 common viscometers, the Brookfield and Haake viscometers, on the yoghurt gel microstructure by means of scanning electron microscopy (SEM) and (ii) to investigate the relationship between the viscosity and the microstructure of the final products.

Materials and Methods

Yoghurt production was carried out according to the method proposed by Tamime and Robinson (1985).

In the viscosity measurement of the yoghurt samples, Brookfield (model LVT, Brookfield Engineering Laboratory, Massachusetts, USA) and Haake (model RV12, attached with PG 142 power supply, Haake GmbH, Karlsruhe, Germany) viscosimeters were used. Two different measurement times (15 s and 30 s) were applied and the measuring temperature was kept constant at 25 °C using a circulating water bath.

Yoghurt samples were prepared for SEM studies according to the method proposed by Brooker and Wells (1984). Two micrometer sections of previously solidified yoghurts in an agar medium were cut with a glass knife microtome (Reichert Ultracut E, Leica UK, Milton Keynes, United Kingdom), placed on a drop of 10% acetone on a circular 10 mm diameter cover slip, and dried by gentle heating. Etching of the samples was achieved by a method

proposed by Lane and Europa (1965). The specimens were dried by means of liquid CO₂, and the dried samples were coated first with aluminum and then with gold under a vacuum sputter coater (Edwards High Vacuum S150, Crawley, United Kingdom). Sections were examined in a SEM device (Hitachi L750, Nissei Sangyo Co. Ltd., Tokyo, Japan) operating at accelerating voltages from 5 to 30 kV.

The study was repeated 5 times (n = 5). Statistically different groups were determined by Duncan's multiple range test (Steel and Torries, 1980).

Results and Discussions

Figures 1a-e show how a classic viscosimeter had a destructive effect on the gel structure of yoghurt. As can be seen from the relevant figures, with the increasing destruction, the protein bonds lost their structural integrity and continuity. A continuous structure was evident in the yoghurt sample before viscosity measurement (Figure 1a). The more detrimental effect of the Brookfield viscometer was clearly seen after a period of 30 s (Figure 1c) compared with the shorter period application (15 s) (Figure 1b). Similar conclusions could also be drawn for the Haake viscometer, albeit to with a lesser extent. Extending the measurement time caused a greater and unrecoverable deformation in the delicate gel structure. These findings are in line with theoretical approaches to yoghurt gel formation and destruction by the high shearing effect. SEM micrographs showed that the deformation created by the Haake viscosimeter was less pronounced than that created by the Brookfield viscosimeter (Figures 1b and c versus Figures 1d and e). As a result of shearing during the viscosity measurement, the casein aggregates pulled apart, and the tiny thread-like structures between the strands in the samples subjected to 30 s measurement (in both Brookfield and Haake viscosimeters) may be a result of stretching. Figures 1c and e indicate that the longer period of viscosity measurement may have caused a 'ripping', not only of the casein aggregates but also in the clusters and micellar chains. There was a clear harmony between the SEM images and viscosity measurements (Table 1). The viscosity values of the experimental yoghurts measured by the Brookfield viscosimeter for 15 s and 30 s were 225.0 poise and 189.0 poise, respectively. Similarly, the viscosity values determined by the Haake viscometer for 15 s and 30 s were 248.0 poise and 237.0 poise,

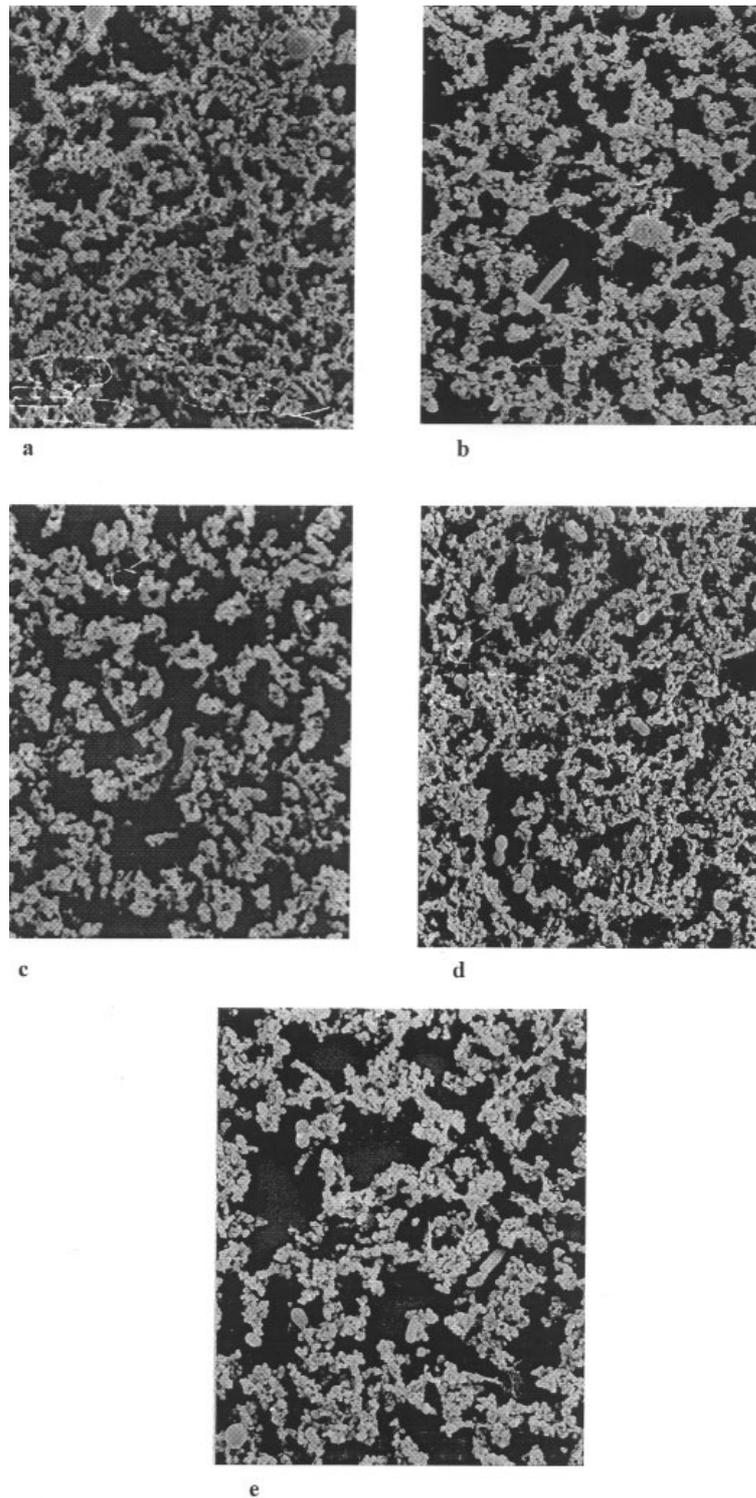


Figure 1. SEM images of experimental yoghurts. (a) before viscosity measurement; viscosity measurement with Brookfield viscosimeter (b) for 15 s, (c) for 30 s; viscosity measurement with Haake viscosimeter (d) for 15 s, (e) for 30 s

Table 1. Viscosity values ($\pm Sx$) of the experimental yoghurts (poise) ($n = 3$). Common superscripts indicate statistically indifferent groups at $P > 0.05$.

| Brookfield Viscosimeter | | Haake Viscosimeter | |
|-------------------------------|------------------------------|------------------------------|-------------------------------|
| 15 s | 30 s | 15 s | 30 s |
| 225.0 \pm 11.2 ^a | 189.0 \pm 7.2 ^b | 248.0 \pm 9.2 ^c | 237.0 \pm 5.9 ^{ac} |

respectively. In general, lower viscosity values were in good correlation with higher deformation in the gel structures. This correlation was more remarkable in the Brookfield viscosimeter than in the Haake viscosimeter. Since viscosity is related to the volume of space occupied by the macromolecule particles and their interactions with each other, it was not surprising to observe lower viscosity values in the samples subjected to higher shearing.

Acid casein gels such as yoghurt gel are defined as particle gels formed by the aggregation of casein micelles (Roefs, 1986; Dickinson, 1994). The 3-dimensional structure of the particle gels is stabilized by covalent (thiol and disulfide exchange reactions) and non-covalent protein interactions (hydrophobic effect, steric effect, Van der Waals attraction/repulsion forces and electrostatic and ionic interactions) (Mitchel, 1980; Rohm and Kovacs, 1994). The rheological and microstructural properties of an acid casein gel are closely related to the casein concentration, enthalpic/entropic nature of the gel and the extent of repulsion/attraction forces between casein particles and gelation mechanism (Dickinson and McClement, 1996). In addition, the size and distribution of macromolecules (e.g. caseins) and the number of protein contact points also influence the rheology of a gel. In general, the rheological properties of a viscoelastic gel are determined by measuring the resistance of permanent protein bonds against the force applied. Additionally, non-permanent and weak protein bonds also play a role in determining the rheological properties of a weak viscoelastic gel (Dickinson, 1994). In other words, the balance between the strong and permanent protein bonds, and the weak and non-permanent bonds, determines the rheological characteristics of an acid casein gel. In the present case, it is thought that the high shearing effect created by conventional viscosimeters disturbed the balance between permanent and non-permanent bonds, and as a result of the partial destruction of the 3-dimensional structure, more water was released, leading to lower viscosity in the resulting products.

Viscoelastic gels are metastable and have a rather low minimum Helmholtz energy level, defined as the minimum energy level to required keep a gel in its metastable position and characterized as the following equation:

$$DA = dU - TdS$$

where A is Helmholtz energy; U, internal energy; S, entropy and T, temperature.

Generally, acid casein gels are not able to regain their original structure after being destroyed. With deformation, the internal energy (U) of the system increases, but the decrease in entropy is limited. This eventually leads to an increase in the Helmholtz energy level and to a permanent loss of structural unity. This point is schematized in Figure 2. In conclusion, in order to obtain rheological data that mirror the actual rheological characteristics of the gel in question, the selection of rheological method is of primary importance. In this respect, the recent developments in dynamic rheological testings have enabled more precise and objective evaluation and characterization of the physical properties of weak viscoelastic materials such as yoghurt gel.

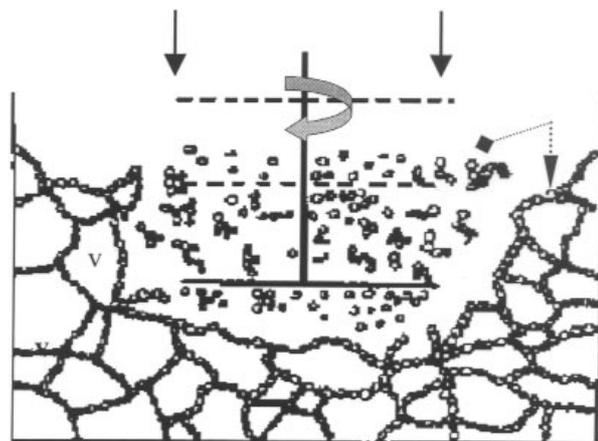


Figure 2. Schematic illustration of destructive effect of empirical viscosimeters on a weak viscoelastic gel. Legends: v, voids; - - ->, casein chains; - - -◆ degraded casein particles.

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