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Structure development studies of soft gels using a dynamic U-tube rheometer of novel design

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Abstract

The process of structure formation of a series of food gels was studied using a patented U-tube rheometer of novel design. The instrument has been especially designed and developed to determine the shear moduli of very weak and syneresing gels, particle contained gels and foams. The rheometer is fully automatic and operates in both static and oscillating modes. The performance of the instrument was assessed by monitoring the development of the shear modulus in relation to time of gelatinised maize starch aqueous dispersions ranging in starch concentration from 6% to 12% (dry basis), gelatine gels with gelatine concentrations ranging from 2% to 12%(dry basis) as well as of native set yogurt and kefir gels. An important asset of the design is that it allows measurements to be performed on liquids as thin as water yet having the maximum strain within the limit of linear viscoelasticity. This allows a gel formation process to be monitored right from the start when the sample is still in the state of a liquid up to the point of the formation of a true gel. Another important asset of the instrument is the modular design of the sample holder allowing easy access for cleaning which moreover is fully detachable from the main body of the instrument so the sample it contains can be pretreated in an environment (heating or freezing) other than that it will eventually be measured.

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1. Introduction

The rigidity modulus of a gel can be determined by applying a known air pressure to a sample and measuring the resulting deformation. Based on this method, Saunders and Ward[1] developed a technique known as the U-tube method. The apparatus they used was made of a cylindrical glass tube in the form of a U-tube comprised of a wide limb and a capillary limb. In the wide limb a column of gel rested on

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mercury, which continued through to the capillary limb. Small air pressure was applied above the gel surface and the resulting volume deformation was calculated from the rise of the mercury in the capillary arm. Scott Blair and Burnett [2]) further improved the apparatus to permit measurements on very weak gels. The modified apparatus employed a U-tube of equal wide arms and the volume deformation was followed by the movement of a drop of coloured kerosene along a graduated horizontal capillary. The advantages of the U-tube technique included: the simplicity in construction, very little disturbance to the sample, which is important for some bio-products such as renneted milk [3,4], and tolerance to wide range of samples, some of which cannot be handled by conventional cone and plate rheometers such as gels containing particles.

This paper describes a computerized U-tube rheometer [5,6], capable of determining the rigidity of gels or other materials such as foams, in both static and oscillatory modes. Differing from common U-tube design, this U-tube consists of two identical limbs where the sample is loaded. Connected to each side of the U-tube, there is an enclosed air chamber whose pressure is measured by transducers. Connected to the air chamber, on one side, a reciprocating piston creates a driving pressure, resulting to a sample deformation, and consequently to a pressure increment in the air chamber on the other side of the U-tube. From these pressure measurements, the rheological characteristics of the sample can be determined.

The performance of the instrument was assessed by monitoring the development of the shear modulus in relation to time of series of soft and strong food gels.

2. Instrumentation

Fig. 1 shows a sketch of the U-tube rheometer built according to the principles mentioned above. The instrument comprises of the following parts: 1. The U-tube unit, which consists of three detachable parts for easy cleaning and where the test sample is placed. The U-tube parts were internally grooved to avoid wall depletion (slippage) effects.2.Temperature control is achieved by clamping the U-tube unit in between two identical thermostat units forming a sandwich type construction. 3. Connected to the left limb of the U-tube are:(a) A bleeding valve. (b) A dead volume V0*l*, which can be increased by adding extra dead volumes through valves to deliver smaller pressures. (c) An engine mechanism for converting a circular motion from a driving motor shaft to a reciprocating motion of a piston in cylinder, by which a step or oscillatory air pressure increment can be created. (d) A pressure sensor P*l*. 4. Connected to the right limb of the U-tube are:(a) A bleeding valve.(b) A dead volume V0*r* whose value was designed according to the sensitivity requirements for pressure increment versus volume displacement, namely $\Delta P r/\Delta V r$. (c) A pressure sensor P*r*.

Since a piston volume stroke is much larger than a sample volume displacement, namely $\Delta V l - \Delta V r$, the magnitude of driving pressure, $\Delta P l$, is virtually determined by dead volume selection. Furthermore, the rheometer is designed so that the volume displacement $\Delta V r$ is always very small to keep the sample under the linear strain limit.

The shear modulus of the sample can be calculated from the two pressure recordings using an analysis exactly analogous to the well-known Poiseuille's law on viscous fluid flowing through a tube. Thus the following equations can apply:

$$\gamma_{wall} = 4\ \Delta V r / \pi R^3 \qquad (1)$$

$$\tau_{wall} = (\Delta P l - \Delta P r)\ R / 2\ L \qquad (2)$$

$$G = \tau_{wall} / \gamma_{wal} = \pi\ R^4\ (\Delta P l - \Delta P r)\ / 8\ L\ \Delta V r \qquad (3)$$

Where R and L are the radius and length of the sample column respectively, γ_{wall} and τ_{wall} are the shear strain and shear stress at the tube wall, and G is the shear modulus of the sample.