RHEOLOGICAL MONITORING OF STRUCTURE DEVELOPMENT AND REBODYING OF SET-STYLE YOGHURT

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ABSTRACT
The present work was undertaken in order to evaluate the rheological changes of set-style yoghurt at different incubation times, during production. The study of rheological changes of milk destined to set-style yoghurt production, in different processing stages prior to fermentation was accomplished using steady and oscillatory shear, while the study of the influence of storage time was also carried out using steady shear. Set-style yoghurt samples with 2 and 3 hours of fermentation exhibited a major structural breakdown during upward shear rate sweep experiments as expected, since the protein network was initially intact. Both samples exhibited two points of sudden decay. For T2 the these points occured at shear stresses of 38 Pa and 77 Pa and for T3 at 47 Pa and 80-83 Pa. The first point corresponds to the main break-up of protein network and the second one to the disruption of primary-aggregates, breaking the portion of casein network that was associated with exopolyssacharides produced by lactic acid bacteria. Structural analysis performed by means of oscillatory measurements indicate that the protein network is already partially formed after 2 hours, albeit full development is only achieved after longer periods. Heat-treated milk, evaporated milk and set-style yoghurt samples with 0 and 1 hour incubation times showed an apparent yield stress value of approximately 0.4 Pa and a strong shear-thinning behaviour afterwards. At higher shear stresses (approximately 1.6 Pa), however, the behaviour changed to shear-thickening, this effect being attributed to flow-induced interactions between the milk components. For the latter sample, a further change in flow behaviour occurred at a stress of 8 Pa, which is probably due to the breakdown of the weak protein network that had formed during incubation.

ZUSAMMENFASSUNG
In der vorliegenden Arbeit wurden die rheologischen Eigenschaften von stichfestem Joghurt mit verschiedenen Inkubationszeiten untersucht. Die rheologischen Eigenschaften von Milch wurden in verschiedenen Prozessstadien vor der Fermentation durch stationäre und oszillierende Schermessungen aufgenommen, während die Untersuchung zum Einfluss der Haltezeit auch durch stationäre Schermessungen durchgeführt wurde. Stichfester Joghurt zeigt nach zwei und drei Stunden Fermentation ein deutliches Zusammenbrechen der Struktur bei zwei unterschiedlichen Schubspannungbereichen. Bei Probe T2 trat dies bei Schubspannungen von 38 Pa und 77 Pa auf, bei Probe T3 war dies bei 47 Pa und 80-83 Pa der Fall. Der erste Punkt entspricht dem Aufbrechen des Proteinnetzwerkes und der zweite dem Zerreissen der primären Aggregate des Caseinnetzwerkes, d.h. der durch die Milchsäurebakterien produzierten Exopolysacchariden. Die Strukturanalyse, die mittels oszillatorischen Messungen durchgeführt wurden, zeigt die teilweise Bildung des Proteinnetzwerks schon nach zwei Stunden, während das endgültige Netzwerk erst nach längeren Zeiten gebildet wird. Wärmebehandelte Milch, verdampfte Milch und stichfester Joghurt mit geringer Inkubationszeit zeigen eine scheinbare Fließgrenze bei näherungsweise 0.4 Pa und danach ein stark strukturviskoses Verhalten. Bei höheren Schubspannungen änderte sich das Verhalten hin zu Scherverfestigung, was strömungsinduzierten Welchselwirkungen zwischen den Milchkomponenten zugeschrieben werden kann. Bei den Yoghurtproben zeigte sich eine weitere Änderung des Fließverhaltens bei einer Spannung von 8 Pa, die der Zerstörung des schwachen Proteinnetzwerkes zugeschrieben wird.

RÉSUMÉ
Le présent travail a été entrepris afin d'évaluer les changements rhéologiques de yaourts types à différents temps d'incubation, pendant la production. L'étude des changements rhéologiques du lait destiné à la production de yaourt, à différentes étapes de la mise en oeuvre précédant la fermentation, a été menée en utilisant des cisaillements constants et oscillatoires, tandis que l'influence du temps d'entreposage a également été étudiée en utilisant un cisaillement constant. Les échantillons de yaourt type avec 2 et 3 heures de fermentation, présentent, comme prévu, une décomposition struturale majeure, lorsque une rampe montante de vitesses de cisaillement est appliquée, puisque le réseau de protéine est initiallement intact. Les deux échantillons présentent 2 points de décroissance brutale. Pour l'échantillon T2, ces points se trouvent à des contraintes de cisaillement de 38 Pa et 77 Pa, et pour l'échantillon T3, à 47 Pa et 80-83 Pa. Le premier point correspond à la rupture principale du réseau de protéine, et le second à la décomposition en aggrégats primaires, par la cassure de la portion du réseau de

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$$Ta = \frac{\rho^2 \Omega^2 (R_o - R_i)^3 R_i}{\eta(\dot{\gamma})^2} \quad (1)$$

In this, ρ is the density of the fluid, Ω the angular velocity of the inner bob, R_O and R_i the outer and inner bob radii, respectively and η the shear rate dependent viscosity. The calculations show that Ta < 90, *i.e.* well below the critical threshold and, therefore, one must rule out the existence of secondary flows as a cause for this phenomenon. The yoghurt sample with 1 hour of fermentation (*T1*) exhibited a further change in the flow behaviour at a shear stress of about 8 Pa, showing a sudden drop in viscosity, followed by the continuation of shear-thickening (this having occurred for all three experiment repetitions). This change may be due to a structure breakdown, probably of the weak protein network that had formed already during incubation. Again, secondary flows are not a factor because Ta ~ 1000.

Other possibilities for this behaviour would be viscous heating, the onset of turbulent flow and the development of purely elastic instabilities (different in nature from the secondary flows described above). However, the authors do not believe these to be relevant factors in the present situation because there is no apparent reason for it only being present for *T1* and not for the remaining samples. Furthermore, some quick calculations show that the flow characteristics obey the stability criteron for isothermal, laminar flow. Regarding the onset of turbulent flow the Reynolds number, Re, given by

$$Re = \frac{\rho \Omega (R_o - R_i) R_o}{\eta(\dot{\gamma})} \quad (2)$$

is of the order of 120, *i.e.* well below the 50000 critical value [18]. The reduction in torque expected from viscous heating is given by:

$$\frac{M}{M_o} = 1 - \left(\frac{bBr}{12n}\right)^n \quad (3)$$

In this, M_O is the torque under isothermal conditions, b is the temperature sensitivity factor of the viscosity (see Eq. 5 below) and Br is the Brinkman number, that is defined as:

$$Br = \frac{m_o (\Omega R_i)^{n+1}}{k_T T (R_o - R_i)^{n-1}} \quad (4)$$

where k_T is the thermal conductivity, T_O the reference temperature and m_O a constant derived from the temperature dependence equation that is defined as:

$$\sigma = m_o \dot{\gamma}^n e^{-b(T-T_o)/T_o} \quad (5)$$

The estimated reduction in torque, calculated from Eq. 3, is lower than 0.5% (the Brinkman number, Br, is of the order 10^{-5} - 10^{-4}) and, therefore, negligible. Finally, the onset of purely elastic instabilities that may cause very fine, time periodic cells depends on a critical Weissenberg number, We

$$We = \frac{\Omega \lambda R_i}{(R_o - R_i)} \quad (6)$$

where λ is the longest relaxation time of the fluid. In the present case λ could be, in principle, estimated either from steady shear data

($\lambda = \lim_{\dot{\gamma} \to 0} \frac{N_1}{2\sigma\dot{\gamma}}$, where N_1 is the first normal stress difference) or from oscillatory shear data

($\lambda = \lim_{\omega \to 0} \frac{G'}{G''\omega}$).

Unfortunately, in the latter case it was not possible to reach the final terminal region of G' and G'' and, in the former, N_1 was too small to be measured accurately (the lower limit of the normal force transducer yields an accuracy of approximately 100 Pa for the measurement of N_1, at low shear rates). At the inflection point, *i.e.* at a shear stress of 8 Pa, the first normal stress difference can be measured to within 160 Pa (assuming the second normal stress difference, N_2, is zero), which means that whatever the relaxation time may be it is small and, at most, 0.02 s (the value calculated from the worst-case conditions, *i.e.* N_1 = 160 Pa and σ = 8 Pa). Assuming this value as an upper limit for λ yields We < 10, at the inflection point which, again, is well below the commonly agreed critical value of approximately 30.

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4 CONCLUSIONS

The aim of this study was the rheological monitoring of structure development and rebodying of set-style yoghurt, which was performed by means of steady and oscillatory shear experiments. Heat-treated milk, evaporated milk and set-style yoghurt samples with 0 and 1 hour incubation times showed an apparent yield stress value of approximately 0.4 Pa and a strong shear-thinning behaviour afterwards. At higher shear stresses (approximately 1.6 Pa), however, the behaviour changed to shear-thickening, this effect being attributed to flow-induced interactions between the milk components. For the latter sample, a further change in flow behaviour occurred at a stress of 8 Pa, which is probably due to the breakdown of the weak protein network that had formed during incubation.

Set-style yoghurt samples with 2 and 3 hours of fermentation exhibited a major structural breakdown during the upward cycle as expected, since the protein network was intact. Both T2 and T3 yoghurt samples exhibited two points of sudden decay. These points correspond to critical values of shear stress for which significant structure breakdowns occurs. The first point correspond to the major break-up of protein network and the second one to the disruption of primary-aggregates, breaking the portion of casein network that was associated with exopolyssacharides produced by lactic acid bacteria. Structural analysis performed by means of oscillatory measurements, showed, as expected, that sample T3 has a higher storage modulus than sample T2, but in general, the two strutures exhibited a similar elastic structure, as shown by tan δ, indicating that the protein network is already partially formed after 2 hours, albeit full development is only achieved after longer periods.

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