Rheological characterization of stirred yoghurt.  
Viscometry and oscillation.

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ABSTRACT
Rheological characteristics of stirred yoghurt, produced according to an experimental design, were evaluated using a Bohlin VOR Rheometer. Flow curves from viscometry tests were described by a new QRS-model. Principal component analysis of data from oscillation tests demonstrated that G* and δ at one frequency contained all the essential information in frequency sweeps.

INTRODUCTION
There have been many investigations on the flow behaviour of yoghurt (e.g. Parnell-Clunies¹; Ramaswama & Basak²). These studies have demonstrated that yoghurt exhibits pseudoplastic behaviour (sometimes with a yield stress). Two models and variants of these have been used to describe the flow behaviour: The Casson model which has two parameters and the Herschel-Bulkley model which has three parameters. Several studies have evaluated the textural characteristics of set-type yoghurt, but only a few are available on the rheology of stirred yoghurt. Investigations on the flow behaviour of stirred yoghurt have generally been limited to low shear rates. Dynamic measurements of acid casein gels has been used to a limited extent and a linear relationship between log(frequency) and log(G') has been demonstrated (Arshad et al.³).

The objective of this research was to characterize stirred yoghurt with a few parameters containing the essential information from viscometry and oscillation test.

MATERIALS AND METHODS
Yoghurt preparation
The stirred yoghurt was prepared from whole milk as described earlier (Skriver et al.⁴). Two factorial experiments, each replicated three times were performed. In experiment 1 bacteria culture (4 types) and incubation temperature (2 levels) were varied. In experiment 2 bacteria culture (3 types) and dry matter content (3 levels) were varied. All together 51 samples were examined.

Rheological measurements
The viscometry and oscillation measurements were performed using a Bohlin VOR Rheometer system (Bohlin Reolog, Science Park Ideon, Lund, Sweden). Measuring temperature was 5°C and a coaxial measuring system (C25) was used. In the viscometry test the shear rate was varied from 29.22 s⁻¹ to 920.7 s⁻¹ in 16 steps with equal distance on a logarithmic scale, and the corresponding shear stress values were measured. In the frequency sweep the frequency was varied from 0.01 to 0.5 Hz in 5 steps at strains in the linear viscoelastic range (determined from a strain sweep at 0.5 Hz). Storage modulus (G'), loss modulus (G''), complex modulus (G*), dynamic shear viscosity (η') and loss angle (δ) were obtained.

Statistical analysis
The flow curves were fitted to the Casson, Herschel-Bulkley and the new QRS model using linear or non-linear regression. Principal component analysis (PCA) was used to locate the essential information from frequency sweeps. The Statistical Analysis System program package (SAS) was used for all
statistical evaluations (PROC NLIN, PROC PRINCOMP).

RESULTS AND DISCUSSIONS

Viscometry

A typical flow curve for stirred yoghurt is presented in Fig. 1.

![Figure 1. Typical flow curve for yoghurt and fits to three models.](image)

Generally the yoghurts exhibited pseudoplastic behaviour, apparently with a yield stress. Data from all of the 51 flow curves obtained from experiment 1 and 2 were initially fitted to the Casson model and to the Herschel-Bulkley model. The quality of all fits were evaluated by careful examination of the residuals. The Casson model in general gave a relatively poor fit with $R^2$ values (multiple correlation coefficient) ranging from 0.87. Further, the residual error was highly systematic (see Fig. 1). The Herschel-Bulkley model gave a better fit with $R^2$ values generally above 0.97. Still, the Herschel-Bulkley model fit is unsatisfactory. After testing numerous models on the experimental data, a new model, inspired by the Michaelis-Menten equation, was found:

$$\sigma = \frac{Q \times \dot{\gamma}}{(R + \dot{\gamma})} + S$$

$S =$ yield stress, $Q + S =$ hypothetical asymptotic value for shear stress and $R =$ shear rate at shear stress equal to $S + Q/2$.

This model gave a better fit to experimental data from pseudoplastic flow curves with a yield stress ($R^2 > 0.99$). All three parameters can be given an interpretation. As demonstrated in Fig. 1 there is some systematic variation in the residuals with the QRS-model, but compared to the Herschel-Bulkley and Casson models this is negligible. Number distribution of $R^2$ values for fits to the Casson-, Herschel-Bulkley- and the QRS-models data are presented in Fig. 2, from which it is evident that the new QRS model gave the best fit.

![Figure 2. Number distribution of $R^2$-values for the three models fitted to the experimental data.](image)

Oscillation

Fig. 3 shows a typical result for the frequency dependence of $G'$, $G''$, $G^*$ and $\delta$. $G'$ was higher than $G''$ at all values of $\omega$ employed, indicating elastic properties.

![Figure 3. Example of a frequency sweep.](image)
Further, the slopes obtained from linear regression of \( \log G' \) or \( \log G^* \) versus \( \log \omega \) indicated that yoghurt behaves like a weak gel. Although many parameters (\( G' \), \( G'' \), \( \delta \), \( \eta^* \), \( G^* \) etc.) can be obtained in oscillation measurements \( G^* \) and \( \delta \) are sufficient to describe all the rheological informations. Plots of \( \ln (\text{frequency}) \) versus \( \ln (G^*) \) for all the yoghurt samples seems to be parallel lines, indicating \( G^* \) at one frequency contains all the informations. This was confirmed by principal component analysis of \( G^* \) for all 51 the yoghurts. One principal component explained 97.5% of the variation in the data set and indicated that measurements of \( G^* \) at more than one frequency are needless. Similar results are observed for \( \delta \).

The Cox-Merz rule was not obeyed for stirred yoghurt. There is a considerable deviation between the log \( \eta_\alpha \) vs. log \( \gamma \) and log \( \eta^* \) vs. log \( \omega \), eventhough the two lines are parallel to each other.

A relationship was found between the fitted yield stress (S) from the viscometry measurements and \( G^* \) from the oscillation measurements (see Fig. 4), indicating that these parameters describe the same properties in the yoghurt \( (R^2=0.86) \).

![Graph](image)

Figure 4. Plot of \( G^* \) versus the yield stress (S).

**CONCLUSION**

Viscometry: Generally stirred yoghurt exhibit pseudoplastic flow behaviour apparently with a yield stress. Over a wide shear rate interval (29 to 920 s\(^{-1}\)), the often applied Casson and Herschel-Bulkley models gave an insufficient fit. A new model, the QRS-model gave a much improved fit.

Oscillation: In frequency sweeps yoghurt showed a behaviour typical of weak gels. Principal component analysis demonstrated that \( G^* \) and \( \delta \) at one frequency contained all the information of a frequency sweep.

Cox-Merz rule was not obeyed for stirred yoghurt but a relationship between the yield stress and \( G^* \) was found.

**REFERENCES**


