Rheological characterization of modified foodstuffs with food grade thickening agents

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Abstract. This work describes a rheological characterization in terms of shear and extensional properties of whole milk, modified with food grade thickening agents (xanthan and carboxymethyl cellulose) with the purpose of being utilized in dysphagia treatment. Shear viscosity of the thickened fluids (2% wt. of xanthan and CMC) were measured in a stress-controlled rheometer and for extensional viscosity, a custom-built orifice flowmeter was used, with elongation rates from 20 to 3000 s⁻¹. Such elongation-rate values represent the entire swallowing process, including the pharyngeal and esophageal phases. The steady-state shear and extensional flow curves were compared with the flow curve of a pudding consistency BaSO₄ suspension (α=05), typically used as a reference fluid for the specialized commercial dysphagia products. The modified fluids presented non-Newtonian behavior in both, shear and extensional flows, and the comparison with the reference fluid show that the thickened milk prepared here, can be safely used for consumption by patients with severe dysphagia.

1. Introduction
Dysphagia is the difficulty for swallowing solids and liquids¹, which may result in malnutrition, dehydration and pneumonia². This alteration can appear at any age and may be related to some diseases as Parkinson and a few others³. In many cases, modifications of rheological properties of foodstuffs and beverages are prescribed⁴, taking into consideration both, the barium sulfate suspension fluids (BSS) as reference to find out the bolus viscosity that would be safe for swallowing⁵⁶ and also, the National Dysphagia Diet (NDD) guidelines for thickened dietary supplements. Unfortunately, it is common that the preparation and assessment of the different thickened drinks and foods is subjective and qualitative in the majority of cases².

In order to produce adequate foodstuff for dysphagia patients, it is necessary to study flow in the deglutition process, for example, it has been determined that the bolus during deglutition is subjected to a significantly higher shear deformation in the pharyngeal step (shear-rates of 260-930 s⁻¹) than in the esophageal phase¹⁵ (5 s⁻¹). Furthermore, shear rheological properties of dietary food has been subjected of thorough research¹⁵, while extensional response has barely received any attention, although there is definitive evidence that the bolus is also extensively stretched when passing from the mouth to the esophagus⁵. Therefore, the aim of this study is to measure and quantify not only the steady state shear flow response, but also the extensional rheological properties of two beverages thickened with xanthan gum and carboxymethyl-cellulose (CMC), all with the objective to as certain suitable concentrations for safe swallowing by severe dysphagic patients, by matching their rheological properties with those exhibited by the pudding BSS reference fluid. Once the characterization procedure is finished, parameters such as apparent viscosity, consistency coefficient (k-value) and flow behavior index (n-value) are obtained for each test fluid.
2. Materials and methods

2.1 Materials
Varibar® Pudding from E-Z-EM, Canada Inc., (Lake Success, NY) was used as reference fluid (BSS) while whole milk was used as food matrix. Food grade xanthan and CMC were used as thickening agents. Different concentrations of thickening agent (0.5 to 6 wt% for xanthan and 0.5 to 3 wt% for CMC) were used to determine the most suitable concentration required to achieve a pudding consistency.

2.2 Rheological measurements and statistical analysis
Shear viscosity tests were carried out from 0.1 to 1000 s\(^{-1}\) in a stress-controlled rheometer (MCR101, Anton Paar Physica, Austria) equipped with a plate and plate geometry (50 mm diameter, 1 mm gap). All measurements were made in triplicate at 25°C (as recommended by NDD). Flow curves of the thickened milk were matched with the pudding BSS one. Data were analyzed by a paring one way analysis of variance. Dunnett’s test was used to determine whether a significant difference existed (p < 0.05). For the selected concentration, frequency ranges between 0.03 and 100 rad/s were assessed within the linear viscoelasticity region for each sample. Extensional viscosity was measured in a custom-built orifice flowmeter. The test was again performed at room temperature (25ºC) with 10 measuring points for two intervals of strain rates 30 – 300 and 300 – 3000 s\(^{-1}\). The parameters such as consistency coefficient (k-value) and flow behavior index (n-value) were obtained for each thickened fluid as well as the Trouton ratio as function of the effective strain rate.

3. Results and discussion
Figure 1 shows the viscosity curves of the whole milk thickened with xanthan and CMC (2% wt.). For both fluids, this concentration provides the closest match with the BSS pudding although the CMC-thickened milk follows closely the BSS product trend. All samples exhibited shear-thinning behavior. Note also the significant differences exhibited by the CMC thickened milk after 100 s\(^{-1}\) and larger; the importance of such differences must be assessed for safe consumption; since these shear rates are inside the typical values of the pharyngeal steps of the swallowing process.

![Figure 1. Steady-state viscous flow curves for whole milk, whole milk thickened with xanthan and with CMC 2 wt% and BSS pudding](image)

In addition, the frequency test reported that both thickened milk solutions behave as a true polymer solution since storage moduli were larger than the loss ones. Extensional viscosity measurements were also performed. The results are shown in Figure 2, where a decrease in viscosity with increasing elongation rate is observed for all fluids; however, whole milk thickened with xanthan shows slightly lower extensional viscosities at low elongation rates, while the CMC milk product exhibits much...
larger degree of softening in the higher extensional-rate zone. With such observations, it can be said that for lower extensional-rate zone, CMC thickened milk follows closer the BSS pudding trend and for higher rates, it is the xanthan product that reproduce better the reference fluid behavior. Note that even with the differences in softening trend shown by the CMC fluid, the extensional viscosity of the three fluids are of the same order of magnitude. To our knowledge these are the first reported extensional viscosities of this kind of thickened beverages.

Shear and extensional data for the thickened milk solutions were fitted with the Ostwald de Waele model, giving an $R^2$ value greater than 0.95 for all cases (Table 1).

Figure 3 shows the Trouton ratio of the thickened milks as a function of the effective strain rate. From this figure, it can be observed that the presented viscosities are above the theoretical Newtonian limit ($\dot{\gamma}r = 3$); such an observation is expected for valid measurements. For the all the tested fluids, the Trouton ration exhibits an increasing trend with increasing effective strain and again, the CMC thickened milk seems to follow closer the BSS curve. In addition, it is worth to point out that
the results presented in Figure 3 show the elastic nature of the milk thickened products, which for shear-thinning fluids, an increasing trend in Trouton ratios is a definitive proof of elasticity.

Table 1. Ostwald de Waele model parameters for thickened milk solutions at pudding consistency

| Thickener     | Shear viscosity | Extensional viscosity |
|---------------|-----------------|-----------------------|
|               | k    | n     | k    | n    | k    | n    |
| BBS           | 210.71| 0.133 | 209.59| 0.405 | 230.32| 0.437 |
| CMC 2 wt%     | 170.21| 0.178 | 114.82| 0.547 | 3499.1| 0.045 |
| Xanthan 2 wt% | 238.04| 0.162 | 107.0 | 0.541 | 145.07| 0.499 |

Figure 3. Trouton ratio as function of the effective strain for the BSS and pudding thickened milk solutions

4. Conclusions
A systematic technique for thickening beverages was developed to be recommended for local hospitals and nursing homes. 2% wt. of xanthan and CMC in whole milk matched well the steady-state flow and extensional pudding BSS curves so these concentrations may be safe for consumption by patients with a severe difficulty for swallowing. CMC is the thickener agent that best fit the flow curves of BSS pudding.

References
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