

## New Insights into Stringiness from Extensional Rheology

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### ABSTRACT

The final arbiter of consumer acceptance of a food product are sensory studies, which can be roughly categorized into liking studies and quantitative sensory analysis. In the latter sensory qualities of a food product are evaluated by panelists on a defined scale. These are invaluable instruments in setting targets for product development, but also as the final quality arbiters for a new product. Instrumental analysis, be it texture analysis or shear rheology, have served as valuable tools to accelerate food and food ingredient R&D where correlations between instrumental analysis and sensory science can be established. However, there are sensory attributes which are not easily described by shear rheology and other techniques are needed. This is especially true if the kinematic of the deformation at the root of the perception is not matched by shear rheology. This is for example the case with texture length (also called ropiness or stringiness). Despite the relevance of this perception aspect to the quality of the food item, formulation is guided by shear rheology. This bears the risk that formulation recommendations are off target and can lead to costly iterations. We will present a small study which connects careful extensional rheology to a sensory study on a condensed milk model. While the number of samples is small, this study demonstrates the general approach one can take. It points to which attributes are uniquely captured by extensional rheology and therefore where this technique adds distinct value and where conventional shear rheology suffices.

### INTRODUCTION

Instrumental to sensory correlations have played an important role in food science and in particular, shear rheology and tribology have been intensively employed to predict sensory in mouth texture attributes like thickness, slipperiness and smoothness.<sup>1-4</sup> For these quantities Kokini et al. derived a set of proportionalities between sensory attribute and physical properties.<sup>1</sup> They considered relevant sensorial attributes to be described by viscous and friction forces measured at the relevant load and thus, rheological and tribological measurements should be sufficient to describe these in-mouth attributes.

The field of study regarding instrumental to sensory correlations has grown substantially in recent years and techniques have become ever more sophisticated to emulate in-mouth conditions more closely, for which researchers have adapted ever more intricate designs to emulate these conditions.<sup>5-16</sup>

The value in conducting instrumental tests and building correlations to sensory attributes in particular food systems lie in a substantial acceleration of the product development cycle and

savings of costly sensory panel work. This also means that models have greater value if they have higher predictive power and are as generally applicable as possible. The complexity of human perception with all five senses acting together means that any one technique can only ever describe limited aspects of the sensory experience.<sup>17,18</sup>

Focusing on in-mouth texture, and eliminating taste, a set of techniques which adequately describe the flow conditions in the mouth in its complex and changing geometry will be needed. One can consider the most important mechanisms by which a fluid interacts with the oral surfaces and our tactile sense. The fluid needs to spread, it needs to be moved around and is forced to change shape and it gets between two surfaces in close contact. In other words, interface, viscous and elastic, and friction forces matter. Due to the complexity of the mouth geometry, the fluid experiences mixed flows which at various times during the mastication process can be dominated either by shear or by extensional flows. Furthermore, the deformations are non-linear, and the rates vary over a wide range.<sup>19</sup> This means that the adequate rheological techniques need to measure non-linear quantities, operate over the appropriate range of strain rates, and put the fluid through the right kinematic deformation. From this it becomes clear that the fluid needs to be studied under all relevant flow kinematics and hence a combination of shear and extensional rheology will provide more complete information to describe the in-mouth fluid movement and thus sensory attributes associated with it. This has been recognised by the food science community and several have focused on addressing specific questions regarding the mastication process with various types of extensional rheometric tests.<sup>20–30</sup> With the development of novel sensory methodologies, more approaches beyond traditional descriptive analysis are available to measure sensory properties of food products. It follows that different approaches beyond intensity scales have been used to explore relationships between sensory and instrumental measurements including ranking and Check-All-That-Apply (CATA)<sup>31–33</sup> The use of CATA is particularly interesting as it is not based on a true measure of intensity, but rather the applicability of a given attribute for a given product. This approach is of particular interest for attributes that are observable but not easily measurable due to either the complexity of the definition of the attribute or the complexity of the matrix.

Since this contribution will not be a deep dive into extensional rheology the authors point to the extensive literature on the topic.<sup>34–44</sup> In previous work we investigated shear and extensional rheology of a series of condensed milk samples thickened with GTF enzymes.<sup>23</sup> The focus of that publication was on establishing the extensional methodology and data interpretation for a liquid food system, focusing on the kinematics in a constant velocity extensional protocol and to show that this technique adds a differentiated perspective of the material behavior. The present study uses the instrumental data obtained in the previous study to build correlations to sensory. More background regarding GTF enzymes and instrumental methodologies can be found in Kjær et al. and O'Brien et al.<sup>23,45</sup>

In the present study we add sensory data to demonstrate how the material responses measured in extension relate to sensory attributes of in-mouth texture. It will also explore to which extent this data can assess improvements to the model when taking both, shear and extensional rheology or only one of them into account, respectively.

## **MATERIALS AND METHODS**

### Samples

Condensed milk samples were prepared according to standard procedures as described in more detail by O'Brien et al.<sup>23</sup> The sample set is briefly summarized in **TABLE 1**.

**TABLE 1:** Enzyme activities used to prepare various condensed milk samples of varying texture length

	<b>DS activity %</b>	<b>GS activity %</b>
<b>S1 (ref)</b>	0	0
<b>S2 (ref – stab)</b>	0	0
<b>S3 (S6- stab - seed)</b>	50	50
<b>S4</b>	100	0
<b>S5</b>	90	10
<b>S6</b>	50	50
<b>S7</b>	10	90
<b>S8</b>	0	100

### Shear and Extensional Rheology

Details of shear and extensional rheology methodology are described in detail in O'Brien et al.<sup>23</sup>

### Sensory methodology

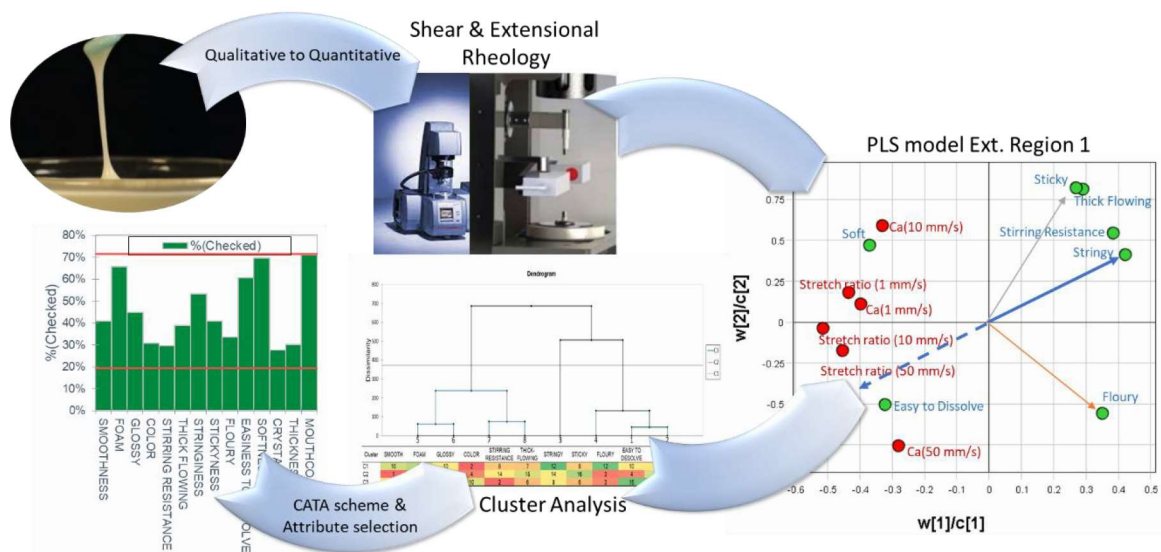
The perceived sensory attributes of the samples were evaluated using the CATA method. The products were evaluated by 5 trained sensory panelists in an initial evaluation to generate a list of descriptors that defines the sensory differentiation between the samples, specifically focusing on texture. These attributes were consolidated into an attribute list which was presented to a panel of 19 semi-trained panelists recruited from a pool of employees. Each panelist was provided with the list of attributes and their definitions and further explanations were provided where the definitions were unclear. The panelists were instructed to evaluate the applicability of the attributes for each sample within the context of condensed milk and to select all the attributes that they perceived to be applicable to each product. The samples were served in a sequential monadic presentation in a balanced complete block design. The samples were served over two sessions with four samples presented in each session to reduce sensory fatigue. The samples were served at ambient temperature. Luke-warm water and crackers were provided as palate cleansers. The data was collected using EyeQuestion software.<sup>46</sup>

### Multivariate Analysis & Statistics

PLS models were evaluated using Evince (Predictera) Software.<sup>47</sup> Data were UV-centered and normalized by their respective standard deviation. Three components were used to describe the data in a Multi-Y PLS model. Models were fully cross-validated by a leave-one-out approach.

## **RESULTS**

Feil! Fant ikke referansekilden. shows the workflow to collect both sensory and instrumental data to be combined with the appropriate statistical methods into a model. Criteria used to select variables from the sensory study followed from the frequency with which a particular attribute was selected as present in the CATA scheme, and whether the data provided enough variation between samples. Furthermore, a cluster analysis was performed to understand interrelations of different sensory attributes. Shear rheology is represented by the Cross-fit parameters for the flow curves of the samples.<sup>23</sup> Linear viscoelastic spectra were analysed by a second order polynomial in a double logarithmic representation.



**FIGURE 1:** Workflow of instrumental to sensory correlation: Combining sensory study to shear and extensional rheology in a PLS model

The extensional data as presented here is more complex than the shear rheology data and data reduction starts by identifying the relevant metrics to be obtained. Here, the ratio of vertical and radial Hencky strains at the onset of the Raleigh-plateau instability is taken to identify the maximum vertical extension at filament break and is called the stretch ratio  $S$ . The capillary number  $Ca$  is calculated from the surface tension of the fluid (assumed to be 50mN/m), the measured extensional stress growth coefficient and the acting Hencky strain rate in the region of interest. This is also discussed in more detail in O'Brien et al.<sup>23</sup> Values obtained from all samples and exposed to three different plate separation velocities fall onto a master curve when plotted against the capillary number based on Region 1 extensional rates and stresses. Higher stretch ratios are achieved at higher capillary number.<sup>23</sup>

**Table 2** shows the list of sensory attributes used in the study along with definitions. The frequency of use across all products and respondents for each attribute was considered and all attributes that were used fewer than 20% or more than 70% of the time were excluded. Significant differences between products were determined with Cochran Q tests and any attributes that did not show a significant difference between products were also excluded. Only one attribute, namely Mouthcoating did not pass and was therefore discarded.

**Table 2:** List of sensory attributes and their definitions used in the sensory evaluation of the products. The attributes are divided in three modalities: appearance prior to manipulation, visual texture during manipulation with a spoon and the perceived texture during oral consumption.

Modality	Attribute	Definition
Appearance	Smoothness	The surface of the product appears smooth
	Foamy	The product appears aerated
	Glossy	The surface of the product is shiny
	Color	The surface of the color is distinctly yellow
Texture on spoon	Resistance to stirring	The force required to stir the product with a spoon
	Thick flowing from spoon	The product does not flow easily from the back of the spoon
	Stringiness	When dragging a teaspoon through the product, it forms a clear string
Texture during oral evaluation	Stickiness	The product sticks to the surface of the oral cavity
	Crystals	Crystalline particles reminiscent of sugar particles is perceivable
	Soft mouthfeel	The product feels soft in the oral cavity
	Easiness to dissolve	The product does not hold together in the oral cavity but dissolves easily
	Thick	The product is viscous and not watery
	Mouthcoating	The product forms a thin film on the surface of the oral cavity
	Floury	The product has a starchy texture when consumed.

In initial models, with all rheological and sensory variables included, it was observed that Thickness and Thick Flowing attributes were highly correlated and only Thick Flowing was taken further. Additionally, a focus was put on sensory attributes which are related to flow, and thus Color, Gloss and Crystals were also excluded from further analysis.

To test which technique or combination of techniques is best suited to describe the sensory dataset, and which of the sensory attributes are best captured by the different instrumental metrics representing both shear and extensional rheology, several PLS models were evaluated on appropriately chosen sub-sets of the instrumental data. In this way, both, the correlation, and predictive relevance of models can be tested for shear rheology, extensional rheology and considering both techniques. The best correlation was found for most of the sensory attributes for the combination of both techniques, however, the predictiveness suffers as including this many instrumental metrics leads to overfitting. Instead, it is found that extensional rheology by itself leads to good correlations with a high predictive relevance for most sensory attributes. This is especially true when only considering region 1 metrics, and both stickiness and stringiness show the highest correlation  $r^2$  and prediction  $q^2$  values. Shear rheology by itself does not lead to good correlation ( $r^2 > 0.7$ ) and predictive relevance ( $q^2 < 0$ ). This suggests that extensional rheology outperforms shear rheology and has an advantage over the combination of both techniques as it produces better models for the condensed milk system. It uniquely describes stickiness and stringiness and is thus of broader relevance for dairy application development.

## CONCLUSIONS

This study demonstrates how extensional rheology can be beneficially employed to investigate cohesive properties of liquid foods and relate these to sensory perception, both in and outside the mouth. These properties are in a sense orthogonal to viscosity in the condensed milk system investigated here where linear viscoelastic properties were nearly constant with formulation, and thixotropy and shear thinning mostly help to describe attributes like thickness but fail to capture stickiness and stringiness. It is interesting to note, that extensional rheology not only leads to better models regarding stringiness and stickiness, but also leads to significant improvements with regards to Thickness of Flow, Stirring Resistance, Floury, Soft and Easy to Dissolve, all attributes which would seem more related to shear than extensional flow. The ability of extensional rheology to capture more than just Stringiness and Stickiness suggests broader relevance of extensional rheology in describing aspects of the out and in-mouth sensory experience.

With that extensional rheology introduces very sensitive observables and becomes a powerful tool to describe both, outside of the mouth and in-mouth perception. It was demonstrated that it can improve or even outperform models based on shear rheology to sensory correlations.

An interesting aspect of extensional rheology is its inherently transient nature. In extensional rheology, steady state is rarely reached and thus measured quantities obtained are presented as a function of time or Hencky strain. This offers opportunities to investigate transient sensory perceptions. In this context it is important to consider the entire transient response curve to fully describe the sensory perception. This aspect was excluded from this study because the sensory study focused on simple sensory attributes and not their temporal dependence during consumption. However, it is an intriguing question for future studies.

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