

Research note

Yield stress for initial firmness determination on yogurt

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Abstract

Yield stress and apparent residual stress were measured in laboratory-made yogurts containing different gum concentrations and in seven retail yogurts. Yield stress exhibited significant correlation ($p < 0.001$) with the sensory initial firmness perceived by trained panelists in both laboratory-made ($r = 0.99$) and retail ($r > 0.97$) yogurts. Apparent residual stress was significantly correlated with sensory viscosity for retail yogurts ($r > 0.90$). The yield stress had more power to detect differences in initial firmness (lower CV(%)), thus requiring fewer samples and was considered a good predictor of the sensory initial firmness perceived by panelists. The use of yield stress also avoids the need for training panelists and conducting sensory panels, and facilitates data analysis by eluding relative scales and lack of homogeneous variances associated with sensory panelists. Additionally, the determination of the yield stress and residual stress offer the potential to manufacture yogurts with targeted yield stress (Pa) and viscosity properties as identified by consumer panels.

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1. Introduction

Different descriptors have been used to characterize the texture of yogurt (Martin, Skokanova, Latrille, & Beal, 1998; Tamime & Robinson, 1999) but no standard procedure exists for the sensory evaluation of this fermented product. Firmness, creaminess, viscosity, sliminess, curdness, chalkyness, and syneresis are considered the most important descriptors for the textural perception of yogurt and other acid dairy products (Muir & Hunter, 1992; Tamime & Robinson, 1999). Nevertheless, the cost associated with the training and running of sensory panels, in addition to difficulties in data analysis associated with data variability, heterogeneous variances (Kuehl, 1994) and use of relative scales, makes the search for instrumental measurements a continuous effort by researchers.

Yogurts are non-Newtonian highly structured materials and their characterization through the measurement of

fundamental rheological properties is not an easy task. In many cases, empirical or imitative methods have been preferred to characterize the textural properties of yogurts (Benezech & Maingonnat, 1994; Fiszman & Salvador, 1999; Hellinga, Somsen, & Koenraads, 1986; Skriver, Holstborg, & Qvist, 1999).

With the exception of drinkable yogurts, all undisturbed yogurts behave as viscoelastic materials or so-called gels. The classification of yogurt is elusive since its structure is the result of disulfide bonding between κ -casein and denatured whey proteins (chemical interaction) and the aggregation of caseins as the pH drops to the isoelectric point of the casein proteins during fermentation (physical aggregation). The determination of viscoelastic properties is cumbersome since yogurts behave as very weak gels with a narrow linear viscoelastic region (Keogh & O'Kennedy, 1998). To avoid differences due to structural breakdown during handling of the yogurt, Ozer, Bell, Grandison, and Robinson (1997) stirred the samples before measurement and reported viscoelastic properties of the residual or recovered structure. The draw back of this method is

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that a different material is being characterized. Yogurts exhibit yields stress, the minimum stress required to initiate flow (Steffe, 1996). Yield stress is also an elusive property to measure, because it depends on the sample itself and on the characteristic time of the process to which the sample will be subjected. There is no standard procedure to measure yield stress and several methods have been suggested (Cheng, 1986; Nguyen & Boger, 1983; Steffe, 1996; Yoshimura, Prud'home, Princen, & Kiss, 1987). The measurement of yield stress in weak materials having abundant whey release is difficult since cutting and placing the sample on the rheometer may affect its structure and micro-syneresis. The “vane method” has been used to study the yield stress of various foods (Yoo, Rao, & Steffe, 1995). Advantages of the vane include (1) slip on the wall is avoided since the material yields within itself, (2) insertion of the vane involves less structural damage when compared to other geometries, (3) narrow annular gaps of conventional geometries are avoided, and (4) It allows the material to yield under quasi-static conditions (Barnes & Nguyen, 2001; James, Williams, & Williams, 1987; Nguyen & Boger, 1983). The most important assumption when measuring yield stress using a vane rotor is that the material yields on the edges of the vane, thus the geometry can be analyzed as a cylinder having the same dimensions of the vane. Nguyen and Boger (1985) demonstrated that the assumption of uniform end stress distribution (i.e., $\tau_e(r) = \tau_w$) is valid. In this way, the yield stress (τ_o) can be expressed in terms of the maximum torque (M_o) by Eq. (1).

$$\tau_o = \frac{2M_o}{\pi D_v^3} \left(\frac{H}{D_v} + \frac{1}{3} \right)^{-1} \quad (1)$$

The objective of this research was to measure the yield stress of undisturbed laboratory-manufactured set yogurts and retail yogurts using the vane method and to correlate this rheological property with the sensory initial firmness as perceived by trained panelists. Additionally, the apparent residual stress was correlated with the sensory viscosity perceived by trained panelists in stirred yogurt samples.

2. Materials and methods

2.1. Yogurts and yogurt manufacture

Fresh milk (3.55–3.60% milk fat, 3.25–3.27% protein) was purchased from the Washington State University (WSU)

Creamery and used to manufacture yogurt. Yogurts having different initial firmness were prepared by adding 0, 3.34, 6.67, 10.00, 13.34, 16.67, and 20.00% v/v of a mother gum blend in fresh milk kept at 43 °C. The mother gum blend was prepared by adding 1.5% (w/v) of Agaroid RS-507® (agar, locust bean, Xanthan, and Carageenan; TIC gums, Belcamp, MA, USA) in milk warmed to 80 °C. After stabilizing the temperature to 50 °C, 10% of a yogurt mother culture blend was added. The yogurt mother culture blend was prepared using five grams of YC-087 frozen culture (CHR Hansen Inc., Milwaukee, WI) per liter of milk and kept at 5 °C. The yogurt mix containing the gum and yogurt culture was poured in 170 mL sterile containers and fermented for 5 h at 43 °C, then stored at 5 °C for 72 h before rheological and sensory determinations.

From preliminary experiments, seven retail yogurts (Table 1) were selected based only on covering a wide range in yield stress, and kept at 5 °C before rheological and sensory determinations. Different types of yogurts were selected to demonstrate that the sensory initial firmness vs. yield stress correlation exists regardless other properties of the yogurt. A 3-digit randomly assigned number was given to each brand and containers were completely covered before used for sensory evaluation. The two extremes in terms of yield stress were assigned as “Hi” and “Lo”.

2.2. Yield stress determination

The yield stress was determined using a MCR-300 rheometer (Paar Physica, Ashland, Virginia, USA) equipped with a 6-blade vane geometry (22 mm diameter, 16 mm height). Yield stress was recorded as the peak stress in a vane inserted 10 mm below the surface of the sample, and rotating at 1 rpm for 100 s. The sample and geometry were kept at 5 °C until each determination. In all cases the yield stress was recorded within the first 20 s of measurement, making changes in temperature negligible. The average stress during the last 10 s of rotation was recorded and defined as apparent residual stress, considered an empirical measurement since the assumption of the geometry being a cylinder does not hold after long rotation time and extensive structural damage of the sample. A completely randomized design with two and three replications was used for laboratory made yogurts and retail yogurts, respectively. Apparent viscosity was measured after shearing the sample for 10 s using a cone and plate geometry

Table 1
Retail yogurts used for initial firmness and yield stress determinations

Sample no.	Brand/Flavor	Fat (%)	Thickener	Size (ounces)
984	Lucerne/lemon chifflon	<0.5	Starch, gelatin	8
216	Tillamook/vanilla bean	1.5		6
321	IGA/peach	1.5	WPC, gelatin, starch	8
257	Dannon LaCreme/vanilla	4	Starch, gelatin	4
684	Dannon LaCreme/raspberry	4	Starch, gelatin	4
Lo	Dannon Light'n fit creamy/lemon chifflon	<0.5	WPC, gelatin, starch, pectin	8
Hi	Dannon/plain	<0.5	Pectin	8

(50 mm diameter, 2° angle, 0.2 mm gap) rotating at a constant shear rate (50 s^{-1}). Since appreciable differences were observed only for the case of retail yogurts, results for lab-made yogurt are not shown.

2.3. Sensory analysis

Sensory analysis by trained panelists was approved by the WSU institutional review Board. Each descriptive panel was composed of individuals who were trained to identify sensory initial firmness and viscosity in yogurts. Individuals were recruited from the Biological Systems Engineering and the Food Science and Human Nutrition Departments at WSU. Five individuals were selected for judging initial firmness of laboratory-made yogurts and 15 for the retail yogurts. One training session was held to familiarize the panelists with the experiments. During training, the panelists were instructed to (1) judge the yogurt samples only for initial firmness, ignoring other properties of the samples, (2) use a plastic spoon to judge the initial firmness of the sample i.e., judge this property by slowly breaking the structure of the yogurt samples, avoiding stirring or extensive structural breakdown, (3) judge the initial firmness based on two anchor (reference) samples for “very firm” and “very weak” yogurt. Each panelist in a private booth received a sheet with six line marking scales (Lawless & Heymann, 1999), 13 cm each, labeled as “weaker” and “firmer”. The first line was used to mark the anchors and the remaining five for each of the samples. After receiving and evaluating the initial firmness of the two anchors, five samples with different initial firmness were handed to each judge in random order. While the anchor samples remained with the judge during the session, judges had to completely judge a sample before receiving the next one.

For retail yogurts, after the panelists finished judging for initial firmness, each of the five samples and the two anchors were stirred 20 times in 10 s ($\sim 120 \text{ rpm}$) using a plastic spoon by a single investigator. After completely breaking the gel’s structure, all the samples were simultaneously handed back to each judge. Panelists were instructed to ignore attributes other than the viscosity as perceived by gently stirring the sample with a plastic spoon. Two variables were obtained from this arrangement: (1) apparent viscosity as centimeters in a 13 cm scale from “less viscous” to “more viscous”, analyzed as completely randomized ANOVA, and (2) yogurt rank from 1 (less viscous) to 7 (more viscous), analyzed using Friedman ANOVA.

3. Results and discussion

Increasing concentration of gums in lab-made yogurt resulted in both an increase of the sensory initial firmness by the panelists and the yield stress (Fig. 1, Table 2). While homogeneity of variances was observed for both initial firmness and yield stress, the panelists exhibited a higher coefficient of variation in their response to increased levels of gums (Table 1). While panelists were able to detect a difference of 2.35 cm in a 13 cm sensory firmness scale (Table 2), the measurement of yield stress allowed the detection of differences equivalent to 0.3 cm in the same scale. The lower power to detect differences in sensory initial firmness vs. yield stress results in the need for more replications and thus larger panels in order to detect a given change in gum concentration in the yogurt. In this way, a panel of $n > 20$ trained judges would be required to obtain the same power to detect differences as the yield stress method for a similar increase in yogurt firmness.

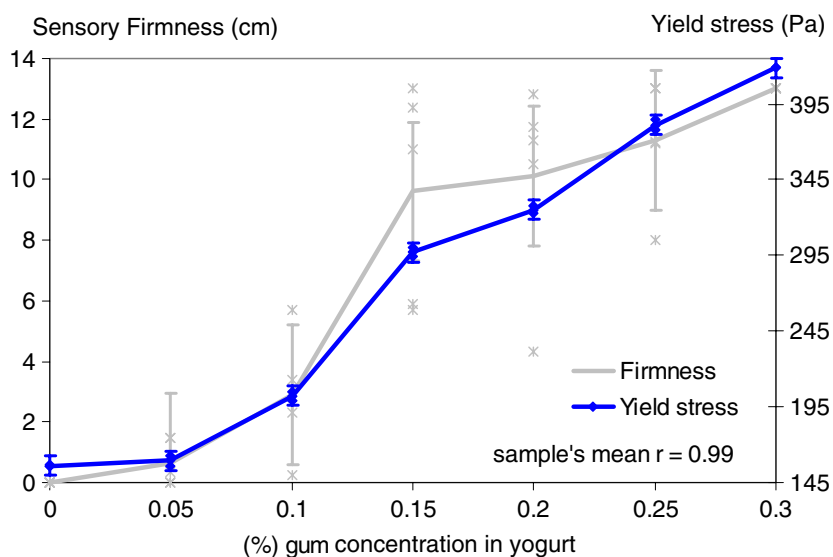


Fig. 1. Sensory initial firmness and measured yield stress of yogurts having selected gum concentration. Bars are 95% confidence intervals for the mean. Stars are individual panelist determinations and diamonds are measured yield stress replications.

Table 2

Estimated parameters for the effect of increased gum concentration (0.0–0.3%) on sensory initial firmness and measured yield stress of yogurt

Estimated parameter	Variable	
	Firmness	Yield stress
Homogeneity of variances – Bartlett	$p > 0.09$	$p > 0.14$
ANOVA for gum level effect	$p < 0.0001$	$p < 0.0001$
Coefficient of variation	36.85%	1.4%
Least significant difference ($\alpha = 0.05$)	2.35 cm	6.3 Pa

Since both, sensory initial firmness and yield stress are destructive determinations, no paired correlation was done between these two variables, i.e., yield stress and firmness were not measured in the exact same sample. Nevertheless, the sample’s average yield stress was highly correlated with the average sensory initial firmness perceived by the judges ($r = 0.99$; $p < 0.001$). Since the true mean (population mean) for both variables is not known, the observed correlation value has to be considered as an indication of good association between these two variables while the true correlation is not known.

Yield stress values were within the range reported by Kovalenko and Briggs (2002) for soy based yogurt using the vane method, but were higher than values reported

by Ramaswamy and Basak (1991), Benezech and Maingonnat (1994), Harte, Amonte, Luedecke, Swanson, and Barbosa-Canovas (2002) using other methods for measuring yield stress. Causes for these differences are (1) structural damage associated with measuring yield stress using cone-plate geometries since the sample had to be “cut” and placed in to the measuring system, (2) potential for microsineresis and slippage in this geometry as demonstrated by Benezech and Maingonnat (1994), (3) pre-shearing of samples promoting extensive structural damage (Ramaswamy & Basak, 1991), and (4) determination of yield stress as the departure from linearity in a stress vs. strain ramp (Harte et al., 2002) and from flow curves yields, resulting in lower values than the single point vane method (Barnes & Nguyen, 2001).

While gum concentration had a significant effect ($p < 0.01$) on the apparent residual stress (i.e., the average stress after 100 s shearing), the maximum observed difference between the yogurt containing 0.3% gum and the gum-free yogurt was only 11 Pa. This small textural difference was below the minimum threshold detectable by trained panelists and thus no further tests for sensory viscosity were done.

The seven retail yogurts tested by the panelists were selected to cover a wide range in sensory initial firmness based on previous yield stress determinations. The sensory initial firmness did not exhibit homogeneous variances for the different retail yogurts tested (Table 3, Fig. 2) due to (1) the effect of specific physical and textural properties of some yogurts (e.g., color, ropyness) affecting the appreciation of the sensory initial firmness by panelist (e.g., sample 321 was the only sample containing fruit pieces), and (2) the so called end effects (Lawless & Heymann, 1999) or errors of central tendency (Stone & Sidel, 1985) due to

Table 3

Estimated parameters for the effect of seven retail yogurt samples on sensory initial firmness and measured yield stress of yogurt

Estimated parameter	Variable	
	Firmness	Yield stress
Homogeneity of variances – Bartlett	$p < 0.008$	$p > 0.9$
ANOVA for yogurt brand	–	$p < 0.0001$
Coefficient of variation	17–110%	5.4%
Least significant difference ($\alpha = 0.05$)	–	21 Pa

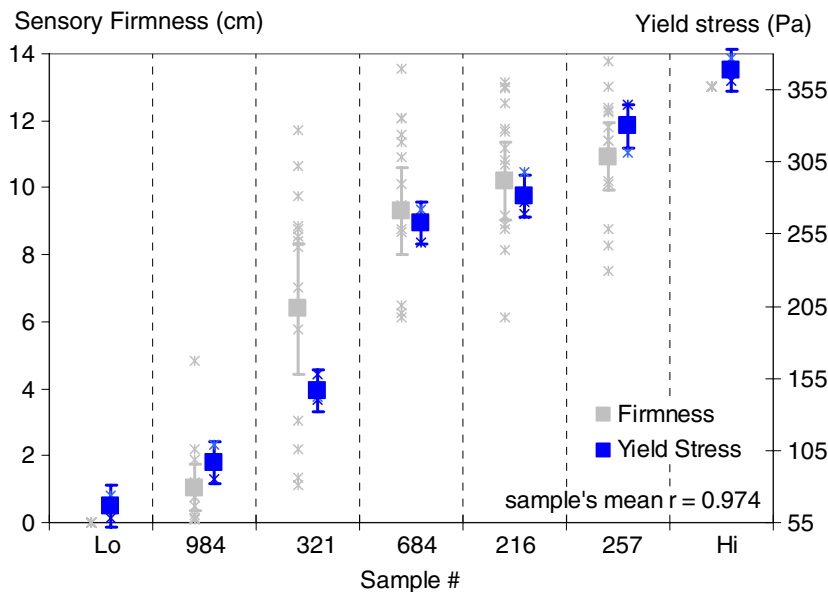


Fig. 2. Sensory initial firmness and measured yield stress of seven retail yogurts. Bars are 95% confidence intervals for the mean. Stars are individual panelists (gray) and yield stress (black) replications.

Table 4
Rheological and sensory data for seven stirred retail yogurts

Sample no.	Rheological property		Sensory viscosity	
	Apparent residual stress (Pa) ^a	Apparent viscosity (Pa s) ^a	Rank ^b	Scale (cm) ^c
257	103.6 a	2.43 ab	6.5 a	11.9 ± 0.94
684	99.3 a	2.59 a	5.9 a	11.0 ± 1.23
216	95.3 b	1.78 cd	4.3 b	6.9 ± 2.20
321	63.4 b	2.23 b	3.5 b	4.9 ± 1.17
Hi	53.8 b	1.70 d	3.9 b	6.2 ± 2.36
Lo	46.5 c	1.96 c	2.5 c	2.0 ± 1.39
984	44.4 c	1.36 e	1.5 d	0.4 ± 0.66
Bartlett ^d	$p > 0.6$	$p > 0.9$	–	$p < 0.001$
CV (%) ^e	4.4	4.4		

^a Means followed by the same letter are not significantly different using Tukey test ($\alpha = 0.05$) after ANOVA.

^b Average rank by 15 panelists from 1 (least viscous) to 7 (most viscous). Means followed by the same letter are not significantly different using Wilcoxon test ($\alpha = 0.05$) after Friedman ANOVA.

^c Average distance by 15 panelists in a 13 cm scale (0 = less viscous; 13 = more viscous). ± 95% confidence interval.

^d Bartlett's test for homogeneity of variances.

the tendency of judges to avoid the use of the end of a bound scale (e.g., sample 984). Although the panelists were trained to ignore attributes other than the initial firmness and to use the entire scale, these sources of variability are common in sensory panels.

The hypothesis of homogeneous variances was not rejected for the case of the yield stress (Table 3), thus the variability was not affected by the particular formulation of retail yogurt tested. The average value of the variable sensory initial firmness was highly correlated with the average yield stress ($r > 0.97$; $p < 0.0001$). Furthermore, excluding the yogurt 321, having high variability for initial firmness, the correlation reached $r > 0.99$ ($p < 0.0001$).

A panel of $n > 20$ judges would be required to have the same power to detect differences in initial firmness of retail yogurts when compared to three replicates of the variable yield stress.

The yield stress determination as a single point in a controlled shear rate method may be affected by the specific shear rate (Yoshimura et al., 1987). However, the single-point controlled shear method offers less variable results than the stress-strain ramp. Furthermore, the yield stress depends on both the material and the characteristic time of the process (Barnes, 1999), i.e., the shear rate affects the yield stress by definition.

No significant correlation was observed between yield stress and apparent residual stress ($p > 0.15$) for retail yogurts indicating that two independent properties of the yogurts were characterized. While yield stress is related to the “solid like” behavior of a viscoelastic material it is hypothesized that the apparent residual stress is related to the viscosity of a Newtonian material, since the structure has been largely disrupted. In this way, while the yield stress may be greatly affected by the type of gelling agent used in the formulation of the yogurt (e.g., gelatin), once the structure is disrupted, the residual stress might depend on the total solids (mainly fat and protein) and concentra-

tion of thickening agents, e.g., full fat yogurts containing no gums may have lower yield stress and bigger apparent residual stress than fat-free yogurts containing gelatin.

The apparent yield strain exhibited very high variability, making its determination of very low use in a texture map tool (Breidinger & Steffe, 2001). The sensory viscosity as perceived by the trained panelists exhibited non-homogeneous variances and judges had difficulties ranking the seven stirred yogurts according to their sensory viscosity (Table 4). Despite these difficulties, both ranked and scaled perceived viscosities were highly correlated with the apparent residual stress ($r > 0.8$) and to a lesser degree with the apparent viscosity ($r > 0.7$; Table 5).

As mentioned before, the apparent residual stress should be considered as an empirical measurement and a rough approximation of the true stress at a given shear rate since the assumptions used to calculate the yield stress no longer hold after the material yields. The apparent residual stress should be considered as useful information under specific set of conditions and not as a fundamental rheological property. While apparent viscosity and apparent residual stress were measured in structurally damaged yogurts, the former was recorded after 10 s shearing using a cone and plate geometry (gap = 0.2 mm) promoting extensive damage to the structure and strongly lowering the apparent viscosity as reported by Basak and Ramaswamy (1994). Less damage was promoted when while measuring the apparent

Table 5
Correlation (r) between average rheological and sensory properties for stirred retail yogurt

Rheological property	Sensory viscosity measured as rank		Sensory viscosity measured as scale	
Apparent residual stress	0.903	($p < 0.005$)	0.906	($p < 0.005$)
Apparent viscosity	0.805	($p < 0.05$)	0.785	($p < 0.05$)
			0.998 ^a	($p < 0.0001$)

^a Correlation between ranked and scaled sensory viscosity.

residual stress and this conditions may explain its higher correlation with the sensory viscosity.

4. Conclusions

Yield stress of yogurt is highly correlated ($r > 0.97$) with the sensory initial firmness evaluated by trained panelists, independent of other physical or sensory properties of the yogurt. Furthermore, the empirical apparent stress after shearing exhibits high correlation ($r > 0.9$) with the sensory viscosity perceived by panelists. Several advantages exist to assess initial firmness of yogurts using the vane method: (1) less time (< 20 s for yield stress) required to run the samples (2) more power to detect differences ($CV < 5.5\%$) and thus less replication needed (3) measurements are done *in situ* and thus there is no damage associated with sample handling, (4) no use of relative scales and anchors associated with trained panels, the results are yield stress values in Pa (5) no costs associated with training and managing large panel group to have adequate sensitivity, (6) no difficulties in data analysis associated with heterogeneous treatment variances, and (7) the ability to design a product based on a target yield stress and residual stress.

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