MEASURING RHEOLOGICAL CHARACTERISTICS AND SPREADABILITY OF SOFT FOODS USING A MODIFIED SQUEEZE-FLOW APPARATUS

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ABSTRACT

The rheological characteristics of soft foods such as peanut butter, mayonnaise and margarine were evaluated by the UW Meltmeter, a modified squeezing-flow device, applying a constant load under constant volume and changing area. Sample thickness profiles were measured as a function of time and the yield stress was calculated via a Herschel–Bulkley model as a measure of spreadability. Peanut butter exhibited the highest yield stress value (1.31–1.45 kPa) followed by margarine (0.80 kPa) and mayonnaise (0.31 kPa). Calculated values of biaxial extensional viscosity and consistency coefficient values calculated from the UW Meltmeter data followed the expected trends of relative spreadability of peanut butter, margarine, and mayonnaise. These results show that the UW Meltmeter is a relatively simple and reliable device for measuring yield stress and rheological properties of soft foods.

PRACTICAL APPLICATIONS

A new method has been proposed to measure yield stress of spreadable foods.

KEYWORDS

Rheology, soft foods, spreadability, squeeze flow, UW meltmeter, yield stress

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INTRODUCTION

Soft, spreadable foods such as cream cheese and peanut butter (PB) are elastoplastic or viscoplastic materials. Consumer acceptance of these foods depends on their textural characteristics such as spreadability – a measure of how easily and uniformly they can be deformed and spread at end-use temperatures.

The rheological properties of spreadable foods have been studied by a variety of methods. Daubert et al. (1998) and Breidinger and Steffe (2001) used yield stress and yield strain data from vane measurements to construct texture maps of spreadable foods. Citerne et al. (2001) examined the rheological and physical characteristics of PB samples by creep, dynamic oscillatory test, and with equilibrium flow curves by fitting the Casson and Bingham models. However, semiliquid and soft foods like spreadable foods are often difficult to work with conventional rotational rheometers because of the possible wall slip and excessive sample disruption during loading into narrow gaps.

The squeezing-flow rheometry has become a very attractive method to measure the rheological properties of semi-liquid and soft foods because wall slip is in fact a prerequisite for a proper test and due to its simplicity in interpreting results from sample thickness as a function of time. The technique was primarily used to measure the flow properties of highly viscous materials such as polymer melts, but has also been a useful method for semiliquid and semisolid food materials such as processed and melted cheeses, butter, dough, PB, etc. (Casiraghi et al. 1985; Wang et al. 1998; Gunasekaran and Ak 2003).

The objectives of this research were to: (1) evaluate the rheological characteristics of soft, foods by a modified squeezing-flow device, named the UW Meltmeter; (2) compare yield stress measurements of the UW Meltmeter with traditional techniques such as the vane and shear stress ramp tests; and (3) relate rheological properties measured using UW Meltmeter to relative spreadability of the foods studied.

MATERIALS AND METHODS

Spreadable Food Materials

Three store-bought commercial foods were used: Peanut Butter Delight (Aldi Inc., Batavia, IL), Shedd’s Spread Country Crock margarine (Unilever, Englewood Cliffs, NJ) and Hellman’s mayonnaise (Unilever, Englewood Cliffs, NJ). These products were chosen because they represent a range of subjective spreadability characteristics such as easy (mayonnaise [MY]), mild
1 (margarine [MG]) and semi-hard (PB), and are known to be self-lubricating because of their high fat content, which is a requirement of a proper squeeze-flow test.

PB tested was from six containers: four were labeled as C2, C3, C4 and C5, which were manufactured at the same time (lot date of January 4, 2007); the other two were labeled C1 and C6, and were from lot dates of December 19, 2006 and February 16, 2007, respectively. MY and MG tested were from only one container each. All materials were stored in a refrigerator (~4.0°C) prior to testing. MG and MY were tested at refrigerated temperatures (5.0 and 7.5°C, respectively). PB was left at room conditions for 2–3 h for temperature equilibration before testing at room temperature (22°C). Temperatures of all samples were monitored periodically to ensure that maximum variation was less than ±1.0°C. When samples reached the upper temperature limit, they were brought back to the refrigerator.

Prior to measurements, samples were allowed to rest for 5 min upon loading. In addition, all experiments were performed in triplicates \((n = 3)\) following a balanced randomized run order with respect to replicates and measurement techniques to account for structural and consistency variations throughout testing.

**Modified Squeezing-Flow Apparatus**

A modified squeezing-flow device, originally designed and fabricated to measure the melt and flow behavior of cheeses, was used. This device, called the UW Meltmeter, was developed in the Food and Bioprocess Engineering Laboratory of the Biological Systems Engineering Department at the University of Wisconsin-Madison (Wang et al. 1998). A schematic diagram of the UW Meltmeter and photograph of the system are shown in Figs. 1 and 2, respectively. It consists of one or two sample wells (7 mm in depth and 30 mm in diameter) with a stationary inner ring and a moveable outer ring. In each unit, the inner ring is equipped with an electric heater operated by a temperature controller (CN 4400, Omega Engineering, Inc., Stanford, CT). A stationary center piston allows the outer ring to move up and down through a lever. When the outer ring is in the UP position by rising the lever, a sample well is formed in which sample can be easily loaded without excessive structural disruption (Fig. 3a). When the lever is lowered, the outer ring is in the DOWN position exposing a cylindrical sample specimen of the shape and dimensions of the sample well (Fig. 3b).

A linear variable differential transformer (LVDT; DC-E 1000, Schaevitz Engineering, Pennsauken, NJ) is attached to a 66-mm-diameter circular top plate that rests on the outer ring. By lowering the lever, the outer ring is lowered and the top of the sample specimen is in contact with the top plate.
FIG. 1. SCHEMATIC DIAGRAM OF THE SAMPLE WELL UNIT OF THE UW MELTMETER (GUNASEKARAN AND AK 2003)
LVDT, linear variable differential transformer.

FIG. 2. PICTURE OF THE MODIFIED SQUEEZING-FLOW DEVICE, THE UW MELTMETER, WITH TWO SAMPLE WELLS
LVDT, linear variable differential transformer.
The force applied by the circular top plate, the LVDT rod and any additional weights, causes the specimen to be compressed and squeezed. The UW Meltmeter test is performed applying a constant load under constant volume and changing area.

During UW Meltmeter tests PB, MG and MY samples were scooped from their original containers at respective experimental temperatures and were gently placed into the sample well. Special care was taken to load the samples consistently and to prevent the presence of voids or air pockets. A metal spatula

(Fig. 4). The force applied by the circular top plate, the LVDT rod and any additional weights, causes the specimen to be compressed and squeezed. The UW Meltmeter test is performed applying a constant load under constant volume and changing area.

During UW Meltmeter tests PB, MG and MY samples were scooped from their original containers at respective experimental temperatures and were gently placed into the sample well. Special care was taken to load the samples consistently and to prevent the presence of voids or air pockets. A metal spatula
was used to smooth the top surface and to remove any excess material. The LVDT monitors the diminishing height of the specimen during deformation. A computer data acquisition system (DAS 16G High Speed Analog I/O Board, Metabyte Corp., Taunton, MA) and software (Easyest LX Software, Asyst Technologies, Inc., Rochester, NY) were used to collect the sample height versus time data. Measurements were performed in three replicates \( n = 3 \) at a range of applied forces (e.g., 0.74–1.62 N) for each spreadable food.

**Yield Stress Measurements**

The determination of the yield stress from the UW Meltmeter is based on the residual stress after deformation and takes into consideration that a viscous-plastic fluid (i.e., Bingham plastic and Herschel-Bulkley fluid) flows until it reaches an equilibrium height or limiting thickness \( H_L \). The yield stress parameter of spreadable foods, denoted here as the apparent extensional yield stress, was determined by applying the analytical expression for Herschel-Bulkley fluids proposed by Ak and Gunasekaran (2000) shown in Eq. (1), using the \( H_L \) value obtained from the sample height versus time data.

\[
\sigma_y = \frac{WH_L}{\pi R_o^2 H_o}
\]  

From the equation above, \( \sigma_y \) is the so-called apparent extensional yield stress, \( W \) is the load applied on the circular top plate, \( R_o \) is the initial radius of the cylindrical specimen and \( H_o \) is the initial height of the specimen sample. The mean value of the last 50 data points (20 s) was taken as the \( H_L \) value, as long as the recorded height did not change by more than 5% during that time.

A dynamic rheometer, Bohlin C-VOR (Malvern Inc., Southborough, MA), was used to compare yield stress values with the vane and stress ramp tests. For the vane test, the rheometer was equipped with a four-blade vane (35-mm high and 10 mm in diameter) and was operated in the controlled-rate mode while collecting 150 data points. Samples were loaded into a standard stainless steel holding cup of 62-mm high and 27 mm in diameter. The tests were run at constant rotational speed of 0.5 rpm and angular velocity of 0.052 rad/s.

For the vane test, triplicate measurements were made with the top surface of vane blades aligned with the sample surface taking new samples each time. Triplicate of tests were performed using the single-point determination, based on the total torque required to overcome the yield stress of the fluid, using the following expression (Dzuy and Boger 1983, 1985)
\[ \sigma_{o,\text{vane}} = \frac{2M_o}{\pi d^3 \left( \frac{h}{d} + \frac{1}{m+3} \right)^{-1}} \]  

where, \( \sigma_{o,\text{vane}} \) is the vane yield stress, \( M_o \) is the maximum or peak torque, \( h \) and \( d \) are the height and diameter circumscribed by the tip of the vane blades, respectively, and \( m \) is a constant.

The assumption of \( m = 0 \) has shown to be reliable in previous studies (Steffe 1996; Daubert et al. 1998; Corradini and Peleg 2005); thus, the yield stress was determined with Eq. (3):

\[ \sigma_{o,\text{vane}} = \frac{6M_o}{\pi d^2 (3h + d)} \]  

For the stress ramp test, a 25-mm-diameter serrated-plate geometry (SP 25-mm, Malvern Instruments, Westborough, MA) attached to the rheometer was used with a 1-mm gap. The test was performed by applying gradually increasing stresses onto the sample under controlled-stress mode and collecting 100 data points. The instantaneous (or apparent) viscosity was monitored for the presence of an inflexion point, indicating onset of flow. The yield stress \( (\sigma_{o,\text{ramp}}) \) was given by the stress at the inflexion point in the instantaneous viscosity versus shear stress curve.

**RESULTS AND DISCUSSION**

**Yield Stress Determination**

A typical height versus time response is shown in Fig. 5 for PB (e.g., PB-C5) under the constant force of 1.62 N. The specimen height diminishes exponentially with time, and the initial rate of decrease of sample thickness is inversely proportional to the applied load.

The apparent extensional yield stress \( (\sigma_o) \) values measured with the UW Meltmeter are listed in Table 1 along with those measured by the vane and stress ramp tests. The magnitudes of yield stress determined from all three techniques followed similar trend – MY exhibited the lowest yield stress followed by MG and PB. The yield stress values of all three spreadable materials determined from the UW Meltmeter and the vane tests were in close agreement. However, this was not true when comparing the yield stress obtained from the stress ramp test, as values were lower by more than an order of magnitude than the corresponding values from the other two tests for all three foods. This suggests that yield stress values obtained from shear stress
FIG. 5. TYPICAL SAMPLE HEIGHT VERSUS TIME RESPONSE FROM THE UW MELTMETER OF PEANUT BUTTER (PB-C5) UNDER SQUEEZING FORCE OF 1.62 N

TABLE 1.
YIELD STRESS VALUES OF PEANUT BUTTER (PB-C1 THROUGH PB-C6), MARGARINE (MG), AND MAYONNAISE (MY) DETERMINED BY THE UW MELTMETER, VANE AND STRESS RAMP TESTS

<table>
<thead>
<tr>
<th>Product</th>
<th>Manufacturing lot date</th>
<th>UW Meltmeter Load (N)</th>
<th>Vane method $\sigma_y$ (kPa)</th>
<th>Stress ramp $\sigma_o$ (kPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PB-C1</td>
<td>December 19, 2006</td>
<td>1.44</td>
<td>1.27 ± 0.07</td>
<td>1.37 ± 0.04</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.51 ± 0.08</td>
</tr>
<tr>
<td>PB-C2</td>
<td>January 4, 2007</td>
<td>1.44</td>
<td>1.31 ± 0.04</td>
<td>1.35 ± 0.12</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.56 ± 0.03</td>
</tr>
<tr>
<td>PB-C3</td>
<td>January 4, 2007</td>
<td>1.62</td>
<td>1.43 ± 0.06</td>
<td>1.40 ± 0.06</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.59 ± 0.05</td>
</tr>
<tr>
<td>PB-C4</td>
<td>January 4, 2007</td>
<td>1.62</td>
<td>1.40 ± 0.06</td>
<td>1.42 ± 0.10</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.53 ± 0.02</td>
</tr>
<tr>
<td>PB-C5</td>
<td>January 4, 2007</td>
<td>1.44</td>
<td>1.31 ± 0.04</td>
<td>1.39 ± 0.07</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.56 ± 0.10</td>
</tr>
<tr>
<td>PB-C6</td>
<td>February 16, 2007</td>
<td>2.07</td>
<td>1.79 ± 0.05</td>
<td>1.71 ± 0.03</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.73 ± 0.05</td>
</tr>
<tr>
<td>MG</td>
<td>–</td>
<td>0.74</td>
<td>0.80 ± 0.05</td>
<td>0.86 ± 0.07</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.25 ± 0.02</td>
</tr>
<tr>
<td>MY</td>
<td>–</td>
<td>0.30</td>
<td>0.31 ± 0.02</td>
<td>0.34 ± 0.005</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.12 ± 0.005</td>
</tr>
</tbody>
</table>

Average of three replicates ± standard deviation.
ramp curves are to be considered as the static yield stress; the dynamic yield stress tests were performed under excessive sample disruption during loading and measurement, for example, while lowering the upper serrated plate to a 1-mm gap size.

The limiting thickness \( H_L \) decreased with initial stress (\( \sigma_i \)) applied, and correspondingly the calculated \( \sigma_y \) values increased. These trends are presented in Figs. 6 and 7, respectively. The initial applied stress was determined dividing the applied load (\( W \)) used by the initial area of the specimen (707 mm\(^2\)).

Under high applied load the material is deformed and squeezed at a great extent such that the material instantly collapses. On the other hand, under low load deformation is rather slow requiring long time to reach \( H_L \), which suggests that the material is able to resists the applied stress, and perhaps able to rearrange and rebuild its structure somewhat. Therefore, at higher loads \( \sigma_y \) may be regarded as the static measurement considering both its magnitude (i.e., higher yield stress) and state of physical structure of the material. We recognize that the magnitude of \( \sigma_y \) of the materials tested with the UW Meltmeter may be lower than the “true” yield stress, as \( \sigma_y \) accounts for the remaining material structure after squeezing.

FIG. 6. LIMITING THICKNESS VERSUS INITIAL APPLIED STRESS FOR PEANUT BUTTER (PB), MARGARINE (MG) AND MAYONNAISE (MY)
* Includes averaged values from all PB containers.
Yield Stress of PB as a Function of Manufacturing Date

Results from all the three measurement methods indicate that variations in PB manufactured on different lot dates are somewhat predictable based on yield stress (seen in Table 1). The yield stress of PB of lot dates January 4, 2007 (PB-C2, C3, C4 and C5) and December 19, 2006 (PB-C1) were similar; PB of lot date February 16, 2007 (PB-C6) had the highest yield stress. The reasons for these deviations on yield stress are unknown and beyond the scope of our study, yet possible physical and chemical changes during storage, transportation, and distribution may have contributed to structural changes of PB, not to mention variations in the manufacturing processes.

Biaxial Extensional Strain Rate and Biaxial Extensional Viscosity

The data of specimen height as a function of time (Fig. 5) obtained from the UW Meltmeter were converted into biaxial extensional strain rate $\dot{\varepsilon}_B$ and biaxial extensional viscosity $\eta_B$ with Eqs. (4) and (5), respectively, for all three products (Chatraei et al. 1981; Dealy 1995):
where \( \varepsilon_T \) is the momentary strain rate, \( F \) is the squeezing flow force, \( H(t) \) is the height at time \( t \), \( H_0 \) is the initial height of the specimen, and \( A_0 \) is the initial cross-sectional area of the specimen.

Figure 8 displays the \( \eta_B \) as a function of \( \dot{\varepsilon}_B \) for MY, MG and PB under squeezing loads of 0.30, 0.74 and 1.62 N, respectively. It was observed that increasing squeezing loads resulted in increasing \( \eta_B \), causing the curve to shift upwards; conversely, low loads resulted in lower \( \eta_B \) and curves tended to move downwards. Overall, biaxial extensional viscosity of MY was the lowest followed by those of MG and PB, which follows the expected relative spreadability values for these foods.
Power-Law Constants

The consistency coefficient \( K \) and flow behavior index \( n \) were determined assuming the squeezing-flow behavior of the selected spreadable foods follows the power-law equation (Table 2). The \( n \) values were in the order of 0.010 to 0.024 indicating highly shear thinning behavior for all three spreadable products. These values were much lower than previously reported in the literature based on shear flow measurements (Campanella and Peleg 1987; Steffe 1996). On the other hand, coefficient \( K \) represented well the differences in consistency between products, as values for PB were about two and six times greater than those of MG and MY, respectively.

CONCLUSIONS

The rheological properties of three soft, spreadable foods were measured with a modified squeezing-flow device, the UW Meltmeter. Information of sample height versus time and the resulting limiting thickness \( (H_L) \) obtained were used to calculate the apparent extensional yield stress parameter based on a Herschel–Bulkley model. In general, decreasing deformation rate of sample thickness as a function of time was proportional to the increase in applied squeezing force; and decreasing \( H_L \) resulted in increasing yield values. The magnitude of yield stress increased in the following the order: MY (0.31 kPa); MG (0.80 kPa); and PB (1.31–1.45 kPa). Additionally, yield stress values of selected spreadable foods measured with the UW Meltmeter were in good agreement with values obtained with the vane method. The biaxial extensional
viscosity calculated from UW Meltmeter data followed the expected relative spreadability values for the three foods tested.

REFERENCES