DETERMINATION OF DYNAMIC MODULI OF SOY DOUGHS USING AN ORTHOGONAL RHEOMETER

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ABSTRACT

The orthogonal rheometer in principle provides a relatively simple means of applying a dynamic strain to polymers to determine the dynamic storage and loss moduli ($G'$, $G''$) as functions of frequency and strain. In applying this technique to soy doughs, surface evaporation of water from the samples was found to be a serious problem, but when evaporation was controlled, good reproducibility for $G'$ and $G''$, was obtained. The moduli were very sensitive to dough moisture level, with $G'$ changing by a factor of 10 over the moisture range 40 to 50%. Plate diameter and percent strain both affected the absolute values of $G'$ and $G''$, though the effects were relatively small. Log $G'$ and log $G''$ increased linearly with decreasing nitrogen solubility index. The two major advantages of the orthogonal rheometer for such food systems are the ease of operation and the fact that sample thickness can be chosen to obviate problems associated with sample heterogeneity. Nevertheless, the effects of plate diameter and percent strain need more extensive study.

INTRODUCTION

Although extrusion is an important food processing operation, its basic mechanisms are poorly understood. In food extrusion, a raw material, such as soy dough (for example, hydrated, defatted soy flakes, or flour) is cooked and texturized using a screw extruder (Rossen...
and Miller 1973; Bruin et al. 1978; Chung 1976). The product quality can be controlled by using trial and error methods or empirical equations; but the basic properties of the raw materials are not well known and the complex processes occurring inside the extruder are not fully understood.

Recently, researchers have attempted to use plastic extrusion theories to predict extruder flow rates of soy and other food doughs (Fricke et al. 1976; Harmann and Harper 1974). Methods involving flow-through capillaries in rheometers or extruders have been applied to determine food dough viscosities (Jao et al. 1978; Remsen and Clark 1978; Gwone and Harper 1978; Morgan et al. 1978). Unfortunately, such basic studies are rare.

The orthogonal rheometer or eccentric rotating disk (ERD) geometry offers an alternative method for characterizing the rheology of soy doughs. ERD has already been used successfully in studying polymer rheology. Using ERD, it should be possible to study the rheological properties of dough at any stage of extrusion, from uncooked, raw materials to cooked products. Once these basic properties are determined, the processes occurring inside extruders can be more readily understood.

This paper deals with methodology for investigating soy dough using ERD.

**EQUIPMENT AND MATERIALS**

The orthogonal rheometer (ERD geometry) consists of two parallel disks of radius (R) with a sample between (see Fig. 1) (Macosko and Davis 1974; Ahrens and Golstein 1977; Willey et al. 1974; Maxwell and Chartoff 1965). The distance between the plates (h) can be varied to give the desired sample thickness. The upper disk is driven at a constant angular velocity (ω) and the lower disk is assumed to follow at the same velocity. Strain (a/h) is applied by displacing the lower disk horizontally by an amount (a). The sample undergoes sinusoidal shearing, and the resulting forces (F_x, F_y, F_z) along the principal axes (x, y, z) are measured by strain gauges. The force (F_y) is referred to as the elastic force and can be related to the elastic modulus by:

\[ G' = \frac{F_y h}{(a \pi R^2)} \]  

(1)

The force (F_x) is known as the viscous loss force and can be related to the viscous loss modulus by:
The elastic modulus measures the ability of a material to store energy while undergoing deformation, due to its resistance to any change in shape. As the elastic modulus increases, the resistance to a change of shape increases and the amount of stored energy increases. The viscous loss modulus measures the resistance of a material to flow while undergoing deformation, or the work that is required due to viscosity (internal friction). As the viscous loss modulus increases, the resistance to flow increases, and the amount of dissipated energy (heat) increases.

A Rheometries Mechanical Spectrometer Model KMS-71 was used. Angular velocity range of the Mechanical Spectrometer is 0.001 to 200 radians per second. The strain gauge range is 20 to 10,000 g.

\[ G'' = \frac{(F_x h)}{(a \pi R^2)} \]  

(2)

**FIG. 1. ECCENTRIC ROTATING DISK GEOMETRY**
Additional equipment used in the experiment included a clear plastic humidity chamber (Fig. 2), a hygrometer (Hygrodynamics Model 15-3050) for relative humidity measurement, a 25 mm mold (cylinder with dual pistons) for sample preparation, an Alpine Pin Mill (Augsburg Model A250C10) for grinding flakes into flour, and an autoclave (American Sterilizer Model 57CR).

Material chosen for study was Nutrisoy defatted soy flakes [Archer Daniel Midland, Nitrogen Solubility Index (NSI) = 91]. Most of the work was done using soy flour prepared by grinding soy flakes into flour (Table 1). A series of lower NSI soy flours was prepared by autoclaving the original soy flour at 110°C for 5, 10, 20, and 40 min.

**FIG. 2. MECHANICAL SPECTROMETER AND HUMIDITY CHAMBER**

<table>
<thead>
<tr>
<th>U.S. Standard Screen Number</th>
<th>70</th>
<th>80</th>
<th>100</th>
<th>120</th>
<th>140</th>
<th>200</th>
<th>270</th>
<th>325</th>
<th>pan</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cumulative Percentage Retained</td>
<td>2.4</td>
<td>3.5</td>
<td>4.2</td>
<td>5.3</td>
<td>7.0</td>
<td>41.6</td>
<td>89.0</td>
<td>91.3</td>
<td>100.0</td>
</tr>
</tbody>
</table>
PROCEDURE

Initial experiments were done without a humidity chamber at 23°C. In the experiments using a humidity chamber (23°C), soy flour (12.2 g, 9.1% moisture) was hand mixed with distilled water (10 g) for 1 min. The sample was quickly placed between the disks (within 1 min) and the humidity chamber was closed. The sample was compressed to the desired sample thickness (from 1 to 8 mm). When the relative humidity in the chamber reached 90%, excess material extending beyond the disk edges was carefully removed with a cutting device. When trimming was done properly, very little gouging or cracking of the sample surface occurred. Occasionally, a sample with a dry or crumbly nature (40% moisture only) made compression and trimming difficult. In an alternative procedure, the material was preformed into a disk by placing 20 kg on the top piston of the 25 mm mold. The usual compression and trimming techniques were then used.

After either procedure (approximately 10 min after mixing), the sample was rotated at the chosen angular velocity (0.1 to 40 radians/sec). The $F_x$ and $F_y$ strain gauge ranges were set and recorder pens were zeroed. The lower disk was then displaced in the $+y$ direction to the chosen strain of 1.5% and subsequently moved by increments of 0.5% to a strain of 1.5% in the $-y$ direction. After displacement was reduced to zero, a new angular velocity was chosen and the strain experiment was repeated.

Machine variables studied included, $\omega$, $h$, $R$, and % strain. Product variables studied included moisture, NSI, and particle size. Other variables included time, relative humidity in the humidity chamber, and application of greases on the sample surface to reduce moisture loss. Values of $G'$, $G''$ were determined graphically. Instrument compliance due to transducer deflection was corrected for by using the method of Macosko and Davis (1974).

RESULTS AND DISCUSSION

Initial experiments yielded erratic results. The cause was traced to surface drying with crust formation. Thus, surface drying of the sample had to be controlled before other work could proceed. The soy dough samples lost moisture at a rapid rate, the edge surface becoming very hard and the moduli increasing dramatically (see Fig. 3) with time when drying was allowed to occur. The drying effect masked all other effects.
It was found that a thin coating of Apiezon grease (also petroleum jelly or vegetable shortening) would retard drying. However, the sample still dried slightly before coating, coating thickness and application time varied considerably, and cleanup was time consuming.

It was found that surface drying could be controlled by using a humidity chamber with 90% relative humidity (RH). Changes in moduli with time were small (Fig. 4). The elastic modulus appeared to decrease steadily with time in both the coating and humidity chamber experiments. It was not clear if this was due to continuing hydration, or what effect the relative humidity had on the phenomenon. The elastic moduli decreased by about 4% in a typical 30 min experiment. Moduli were relatively stable at 90% RH (from 0 to 60 min), but increased when the relative humidity decreased. \( G' \) appeared to be more sensitive than \( G'' \) to RH changes. When the RH was adjusted upward (to 85% RH at 70 min), moduli decreased slowly. The RH around the sample is critical during the measurement of moduli. If a sample is allowed to dry before or during ERD measurements, the moduli measured will be incorrect.
Moduli for dough (50% moisture) with a coating are compared to those for dough in the humidity chamber in Table 2. Results are similar, but the humidity chamber method was preferable due to its ease of operation and other factors mentioned previously. Especially important during the coating process was the tendency of the dough surface to dry. Moduli increases of up to 20% occurred in as little as 1 min, making speed during the trimming and coating steps critical.

After drying effects were controlled, machine variables were investigated. Material response decreased slightly with strain (Fig. 5). The value of $G'$ was found to remain fairly constant when sample thickness was varied (Fig. 6). However, $G'$ varied somewhat with $R$ (Fig. 6, for 25 and 72 mm). The reason for this difference is not clear, and more work in this area should be done. The effect of angular velocity on moduli was as expected (Fig. 7), with moduli increasing as angular velocity increased.

Sample variables were then investigated. Soy flakes gave much larger values of moduli than did soy flour (Table 3). If the true differences in moduli between two doughs are to be measured, the flour samples must have the same particle size distribution.
Table 2. Soy dough moduli (25 mm disks, h=4 mm, ω=40)

<table>
<thead>
<tr>
<th>Moduli</th>
<th>Using Moisture Barrier of Petroleum Jelly</th>
<th>Using a Humidity Chamber (90% RH)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$G'$ (dynes/cm²)</td>
<td>$3.27 \times 10^5$</td>
<td>$3.31 \times 10^5$</td>
</tr>
<tr>
<td>$G''$ (dynes/cm²)</td>
<td>$1.44 \times 10^5$</td>
<td>$1.40 \times 10^5$</td>
</tr>
</tbody>
</table>

FIG. 5. MODULI VERSUS PERCENT STRAIN

Moisture content of the dough affected moduli (Fig. 7). Dryer doughs gave larger moduli, as might be expected. The complex interactions between soy protein and water on the molecular level are not understood, although the ERD method should prove invaluable in elucidating these interactions (Lumry 1973). Investigating doughs with 40% moisture or less involved experimental problems. Smaller disks were needed to compress the sample, and the sample usually had to be preformed into a disk (due to cracking and crumbling). Better methods of preparing the sample are needed.

The last dough variable to be studied was Nitrogen Solubility Index (NSI). NSI is a rough measure of protein solubility (denaturation is
FIG. 6. MODULI VERSUS h AT TWO RADII

FIG. 7. MODULI VERSUS $\omega$ FOR DIFFERENT MOISTURE DOUGHS
usually caused by heating in the presence of moisture). NSI of raw soy flakes is about 90 and decreases to 10 or less with protein denaturation. Figures 8-10 show that both moduli increased as flour NSI decreased. It seems reasonable that the protein denaturation (with unfolding of protein and formation of aggregates) would increase both moduli (Huang and Rha 1974; Wolf and Tamura 1969; Circle et al. 1964; Fleming et al. 1974; Catsimpoolas and Meyer 1970).

ERD may be useful in future work to characterize soy and other food doughs. The effect of additives and shear on moduli could determined using the ERD technique. Studying changes in a sample being subjected to high temperatures would be of value.
FIG. 9. VISCOUS LOSS MODULUS VERSUS $\omega$ WITH VARYING NSI

FIG. 10. MODULI VERSUS NSI
SUMMARY AND CONCLUSIONS

Eccentric rotating disks were used to measure dynamic moduli of soy doughs. However, drying of the sample surface had to be controlled. One way was to use a humidity chamber with an RH of at least 90%.

Moduli of soy doughs were very sensitive to both dough moisture NSI. and particle size. Moduli depended on disk radii to a lesser degree. These relationships warrant further study.

REFERENCES


ABBREVIATIONS

\(a\) = displacement in y-direction (mm)

ERD = eccentric rotating disks

\(F\) = force in x, y, or z direction

\(G'\) = elastic modulus (dynes/cm\(^2\))

\(G''\) = viscous loss modulus (dynes/cm\(^2\))

\(h\) = thickness of sample (mm)

NSI = nitrogen solubility index

\(\pi\) = pi (3.14 ...)

\(R\) = radius of disk (mm)

RH = relative humidity (%)

\(\omega\) = angular velocity (radians/second)