TA Instruments

Thermal Analysis & Rheology

Thermal Analysis Application Brief

Dynamic Mechanical Analysis of Food Products

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SUMMARY

The processing and handling of food products can significantly affect the material's texture, flavor, and appearance. Historically, the methods used to evaluate and/or predict these properties have been somewhat arbitrary and non-quantitative. The use of analytical instrument techniques such as thermal analysis provides a more quantitative, reproducible way for characterizing food products. Dynamic Mechanical Analysis (DMA), for example, can provide information about the mechanical properties of food and how they are affected by various processing conditions.

INTRODUCTION

Dynamic Mechanical Analysis (DMA) is a sensitive and versatile thermal analysis technique which measures the modulus (stiffness) and damping (energy dissipation) properties of materials as the materials are deformed under periodic stress. DMA has been employed for some time to measure the structural and intrinsic property changes in polymeric materials [l]. But the sensitivity of DMA as well as the ability to measure properties of larger samples (relative to DSC) has lead to use of the technique in other fields, including metals [2] and biological materials [3]. The processing and handling of food products greatly affect the texture, flavor and appearance of the final edible product. Dynamic mechanical analysis can be used to gain insight into the factors affecting food quality through simulation of processing conditions. There have been, however, relatively few studies on the dynamic mechanical properties of food, although Roulet et al [4] did perform a comparative DMA study on starches of wheat.
In this study, DMA experiments were performed on two food products, commercial white bread and dried pasta. The DMA storage and loss moduli obtained provide valuable information about the softness and keeping properties of bread, as well as the cooking characteristics of pasta.

EXPERIMENTAL

In DMA, the sample is clamped between the ends of two parallel arms which are mounted on low-force flexure pivots allowing motion only in the horizonal plane. The distance between the arms is adjustable by means of a precision mechanical slide to accommodate a wide range of sample lengths (from <1 mm up to 65 mm). In addition, a variety of clamping configurations is available to accommodate different material types. An electromagnetic motor attached to one arm drives the arm/sample to a strain (amplitude) selected by the operator. As the arm/sample system is displaced, the sample undergoes flexural deformation. A linear variable differential transformer (LVDT) mounted on the driven arm measures the sample's response to the applied stress and uses that information to calculate the modulus and damping properties of the material. The rate of deformation (frequency) can be selected by the operator from a wide range (0.001 to 10 Hertz). A frequency of 1 hertz is often used to provide the best compromise between sensitivity and time of analysis.

Samples of a commercially packaged white bread were exposed to atmosphere (temperature ca. 23°C and ca. 70% relative humidity) for various times (no exposure, 3 hours, and 18 hours). The samples were then compressed from original thickness ca. 14 mm to ca. 2 mm to enhance thermal conductivity and signal strength. Samples were cut into strips and mounted into the standard vertical clamps of a TA Instruments DMA 983 Dynamic Mechanical Analyzer. Typical sample dimensions as mounted were 8 mm by 13 mm by 2 mm. Mechanical properties were measured at a fixed frequency of 1 Hz with an oscillation amplitute of 0.8 mm. Temperatures were ramped from -40 to 50°C at a rate of 2°C/min.

The experimental configuration for the pasta product was similar to that of Dillman and Seferis [5]. The DMA 983 was turned on end with the arms pointing downward. Experimental evidence has shown that this arrangement does not affect the measured properties. Horizontal low mass clamps were chosen for this study because of their sensitivity to small changes in sample properties. Typical sample dimensions were 10 mm by 1.25 mm by 2.8 mm. The ends of the samples were moistened slightly before mounting to enhance clamping. Measurements were taken at a fixed frequency of 1 Hz and an oscillation amplitude of 0.5 mm. Samples were immersed in tap water heated to ca. 73°C. According to Dillman and Seferis, the effect of the water on the modulus is independent of the interaction of water with the sample. The fluid correction factors were calibrated using an elastomeric material that did not react with the water.

The drive and phase signals of the calibration material at 73°C are shown in Table 1 below.

Table 1

Drive Signal and Phase Angle of Calibration Standard

	Drive Signal (mV)	Phase (rad)
Dry sample	457.9	0.0382
Immersed sample	427.7	0.0441

The equations of Lear and Gill [6] for DMA modulus are modified to include the fluid correction factors F' and F" [5]:

$$G' = (2Jk^2 - 2K' - 2F') \times (\beta^2 + \gamma^2)/\beta^2 \times (L/B^2 A) - \gamma G''/\beta$$

$$G'' = (2JwD - 2K'' - 2F'') \times (\beta^2 + \gamma^2)/\beta^2 \times (L/B^2 A) + \gamma G/\beta$$

The explanation of the individual components can be found elsewhere [5]. Since the modulus of a material unaffected by the fluid does not change with immersion, the values of F' and F" can be calculated by subtracting the above equations for a dry sample (F' and F" are both 0) from that of an immersed sample . The resulting equations for a fixed frequency experiment are:

$$F' = 8J\pi^2 C' (V_d \cos\delta_d - V_l \cos\delta_l)/a$$

$$F'' = 8J\pi^2 C' (V_l \sin\delta_l - V_d \sin\delta_d)/a$$

Where J = moment of inertia, C" = drive signal constant, V = drive signal, δ = phase, a = oscillation amplitude. The subscripts d and 1 refer to dry and liquid phases respectively.

In this experiment J = 1.6 g-m^2, C' = 0.0094 mm/mV-sec^2, and a= 0. 5 mm. The values for F' and F" are 0.073 N-m and 0.0032 N-m respectively.

Experiments on pasta were performed at immersion times from 1 minute to 14 minutes. Each sample was removed from the DMA 983 rapidly at the end of immersion and placed on the stage of a TA Instruments TMA 2940 Thermomechanical Analyzer. TMA force ramp experiments were performed using the knife-edged flexure probe. The force was ramped from 0.01 to 1 N at 0.1 N/minute and indentation depths were measured.

RESULTS

The DMA curves for the bread samples are shown in Figures 1, 2, and 3 and the key results are summarized in Table 2. Three significant changes occur with increasing exposure time: the subambient storage modulus (E') decreases, room temperature E' increases (albeit modestly), and the subambient peaks in E" and tan δ decrease in magnitude, disappearing altogether in the "overnight" sample. The probable source of these three changes is the loss of loosely bound water with exposure to atmosphere. The large subambient storage modulus in the first two samples is likely caused by crystalline water in this phase. The E" and tan δ peaks likely arise from the melting of

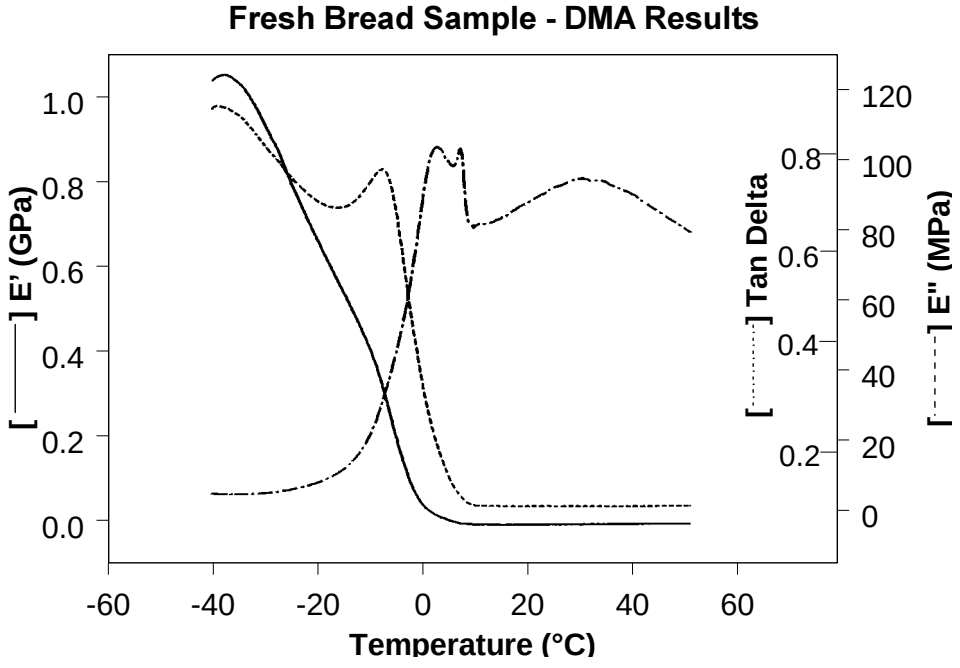

Figure 1

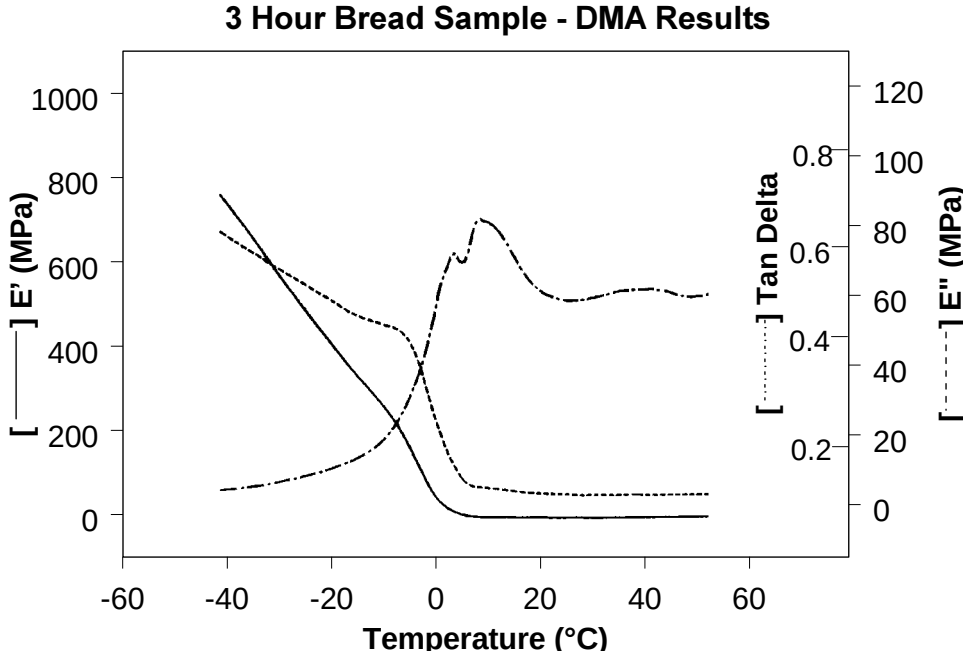

Figure 2

this phase. The tan δ doublet suggests that there may be more than one water phase, or there may be a skin/core effect in the bread which has a low thermal conductivity. The room temperature modulus, on the other hand, increases with exposure time, indicating that water acts as a plasticizer for the bread starch.

There is also a broad tan δ peak at -40°C present in all these samples which arises from the release of more tightly bound water. The magnitude of this peak decreases with exposure time indicating some loss of water from this phase.

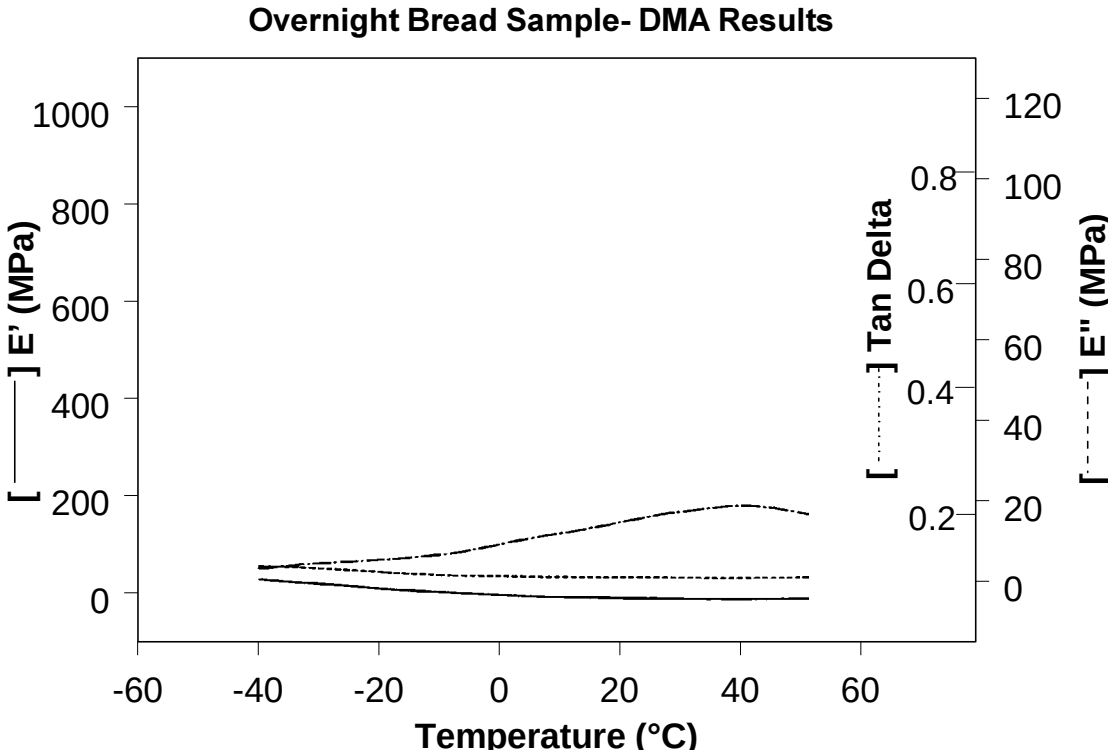

Figure 3

Table 2

Properties of Bread Samples

	Exposure Time		
	Fresh	3 hours	Overnight
E' at -40°C (MPa)	1060	682	58
E' at 25°C (MPa)	2	6	16
E" peak (MPa) at -10°C	100	50	—
Tan δ peak 1 value	0.82	0.53	—
Tan δ peak 2 value	0.81	0.60	—

All of the phenomenon seen in DMA can be related to the tendency for bread to become "stale" and more brittle under different storage conditions.

The moduli of the pasta product were calculated using a spreadsheet program and equations shown earlier. The results of this analysis are plotted in Figure 4. The storage modulus drops relatively slowly until approximately 8.2 minutes where it drops off significantly. This time can be defined as the optimum processing time, since the sharp decrease in modulus indicates an extreme weakening of the pasta, or in terms of texture a "limpness."

Thus, the DMA could be used to evaluate optimum cooking time at different immersion temperatures. These results are confirmed by the TMA experiments, where the penetration of the TMA probe (Figure 5) greatly increased between the 8 and 9 minute samples. The shape of the 9 minute sample TMA curve is virtually unchanged with longer cooking times.

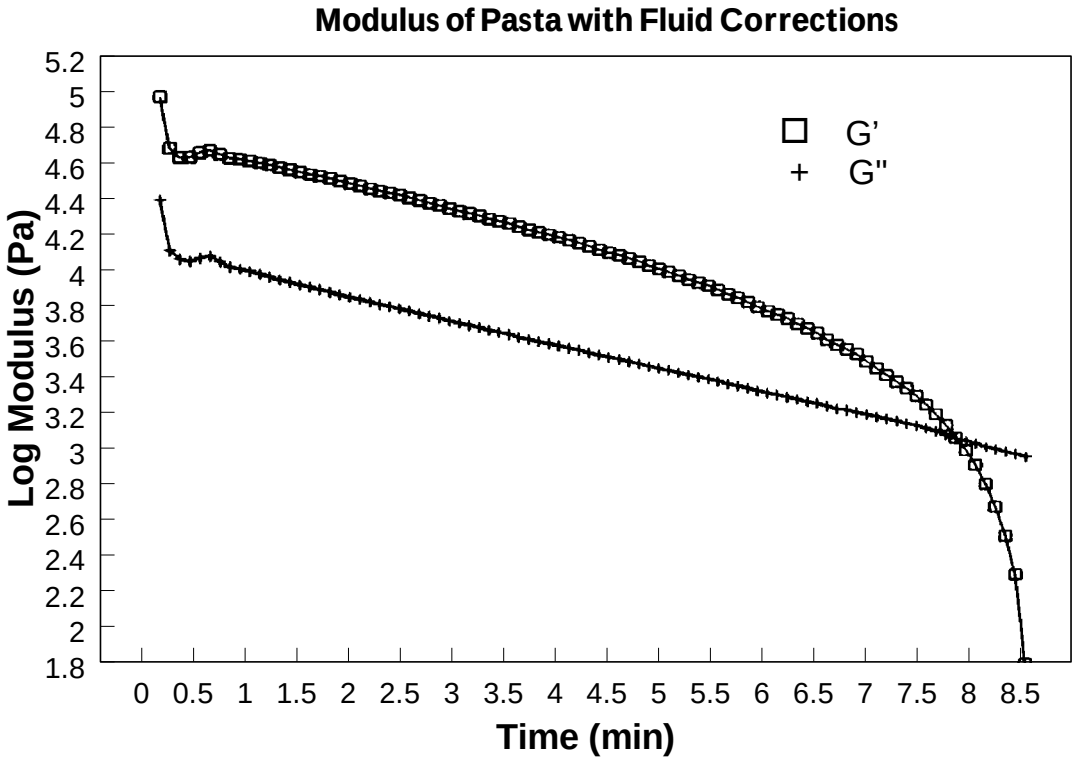

Figure 4

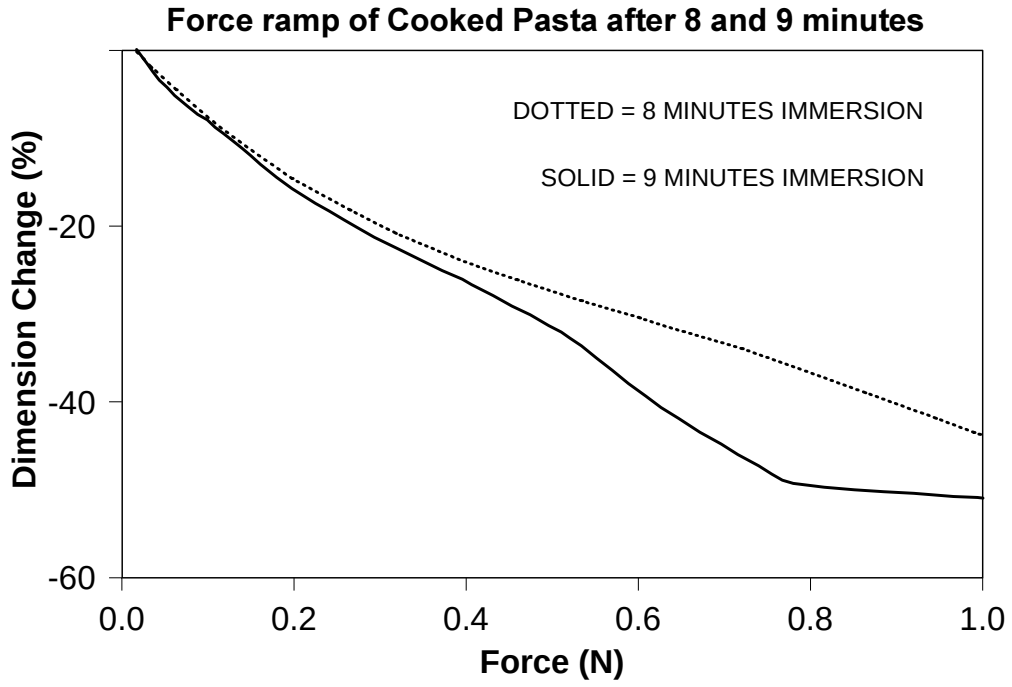

Figure 5

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