SOME RHEOLOGICAL PROPERTIES OF WHEAT FLOUR GELS

Monica A. Louda, Alexandro Morales-Patiño, Nhan Phan-Thien
Department of Mechanical & Mechatronics Engineering,
The University of Sydney
Sydney, NSW
Australia

Vojislav Ilic

Mechanical Automation Engineering University of Western Sydney Nepean Kingswood, NSW Australia

ABSTRACT

It is well recognised that increasing the water content of dough results in a flour suspension or batter. Lack of cohesion is the common characteristic of such mixes. However, if subjected to elevated temperatures, the starch, in the wheat flour, will gelatinize and subsequently form a paste. Apart from their intrinsic interest, rheological characterisation of such mixes is desirable in order to provide specific process control to ensure the uniformity of the end product. Measurements of rheological properties are particularly difficult, because of the number of variables that can affect them, including the degree of mixing, flour batch, additives, temperature, resting time and humidity.

The overall aim of this study was to determine the nature of shear stress, the general dynamic behaviour and ascertain the existence of yield stress for commercial grade flour pastes.

The initial work reported here concentrates only on the flour/water weight fraction of 25%.

INTRODUCTION

The production of wheat based products from flour, water and other ingredients is a process in which rheological properties of the material change considerably at different processing stages. Knowledge of the rheological behaviour is important in understanding and predicting texture and flow characteristics during processing and shelf-life, as well as the quality of the raw materials and the end product.

Currently, there is a growing interest in various aspects of rheology of wheat-based products because large bakeries have become automated. So, for a quality end product, accurate prediction of properties of the product during processing are imperative [Faridi, H., 1987; Biliaderis, C.G. et al, 1992].

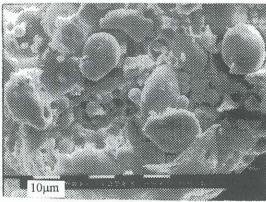


FIGURE 1: SEM OF FLOUR

The difference in the texture and tastes of bread are derived primarily from the interactions of starch and gluten. Flour is the basic product of these substances. This study was broken down to the starch in a flour water system.

The aim of this work is to characterise the behaviour of a wheat flour gel with 25% weight fraction of flour. The initial data presented here include the dynamic response determined from oscillatory tests and yield stress measured with shear vane and stress relaxation tests.

ANALYTICAL BACKGROUND OF A WHEAT FLOUR GEL

Flour and water suspensions lack cohesion, but if subjected to elevated temperatures, the starch, in the wheat flour, will gelatinize and subsequently form a paste. A scanning electron micrograph, SEM, for a flour is shown in figure 1. The large starch granules in the protein matrix are clearly visible.

Starch gelatinizes over a definite temperature range, as specific granules undergo irreversible swelling at

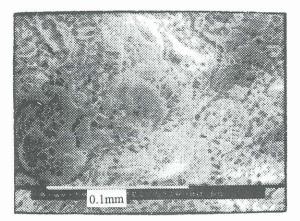


FIGURE 2: SEM OF A 25% WHEAT FLOUR GEL

different times when heated. At the end of the temperature range the granules lose their rigidity and form a sponge-like structure [Liu, J., Zhao, S., 1990]. There are theories about what precisely happens at the molecular level, and how the swelling of the granules occurs [Howling, D., 1980]. Traditionally, it was believed that the water was entering the granule and causing it to swell. An alternative theory was put forward by Miller et al.[Miller, B.S., et al, 1973] that the granules only swell slightly but secrete a substance, exudate, which forms an inter-granular matrix, the cause of the viscosity increase. They found that viscosity always increased sharply after most of the granule swelling ceased. A relationship especially noticeable with wheat starch. The scanning electron micrograph of a 25% wheat flour gel is shown in figure 2. For this micrograph the gel was freeze-dried, i.e. the sample was frozen directly in liquid nitrogen for approximately 45 seconds. Part of the sample was fractured so the internal structure of the starch granules could be viewed. The result observed supports the theory of Miller et al. that granules swell slightly, in comparison to the size of the starch granules present in the flour and secrete an exudate, which forms an inter-granular matrix. The spongy appearance suggests that most the starch granules were gelatinized.

The rheological properties of wheat flour gels are influenced by the amylose gel matrix, volume fraction and rigidity of gelatinized granules; the interactions between starch components; granule size; amylose/amylopectin ratio; physical organization of the granule, minor constituents (granular lipids); presence of solutes; pH; the starch concentration; and the shear-temperature-time requirements [Biliaderis, C.G., 1992].

Therefore, to assess the rheological properties of the wheat-flour pastes requires careful considerations of stress, strain, temperature, gelatinization temperature, time and the experimental procedure. For dynamic measurements, small deformation of the paste is imperative, to maintain linearity between stress and strain.

EXPERIMENTS

Wheat Flour Gel Preparation

The wheat flour gel, with a flour/water weight fraction of 25%, was prepared from flour and distilled water. The

flour that was used was weak flour, supplied by the CSIPO

Apparatus. The flour was sifted through a 210 micron sieve. The flour and water were mixed in a magneto-mixer, at room temperature, and then transferred to the Haake Rheometer, RV-3, fitted with a specially designed four bladed vane to stir the flour and water mixture while it was being heated up.

Method. The distilled water in a 250 ml beaker, was placed on the magneto-mixer and mixed at speeds between 700-900 rpm. The sifted flour was gradually added as the solution was mixed. Once it was mixed, the solution was poured into the Rheometer cup at 90°C. The rotational speed of the vane was set at 1rpm for the 8 minute duration.

Oscillatory Tests

In order to determine the dynamic behaviour of the gel, oscillatory tests were done on the Carrimed, CSL 50, Rheometer, with 4cm parallel plate geometry. The frequency range was 0.001 to 40 Hz, with initial gap set at 1.5mm.

The oscillatory test involves applying a sinusoidal shear strain, γ , of small amplitude γ_0 and frequency ω , given by : $\gamma=\gamma_0$ * exp(i ω t). The shear stress is sinusoidal and proportional to, but out of phase with the shear strain: $\tau=\tau_0$ *exp(i ω t+ δ). The complex shear modulus, $G^*(\omega)$, is a complex dynamic function which relates the shear stress to the shear strain in the following manner :

$$G^* = \frac{\tau}{\gamma} = (G' + i^*G")$$

G', the storage modulus; and G", the loss modulus, are material functions of the fluid.

The storage modulus, G', is a measure of the elastic energy stored per cycle of deformation; the loss modulus, G'', is a measure of the viscous energy dissipated per cycle. The ratio of G'' to G' gives the tangent of the phase angle difference, δ , between the applied stress wave and the strain response:

$$\delta = \tan^{-1}\left(\frac{G''}{G'}\right)$$

For the perfectly elastic solid : G" = 0, which from figure 3 gives δ = 0°, while for an inelastic viscous fluid : G' = 0 and implies that δ = 90°. In viscoelastic fluids both storage and loss moduli are non-zero. The phase difference, between 0° and 90°, depends on the relative proportions of elasticity and viscosity [Nguyen, Q.D., et al., 1992].

<u>Yield Stress Tests</u>

The yield stress of the gel was obtained by the following methods:

 Shear Vane Tests. The vane shear test apparatus used in these experiments was manufactured by Wykeham Farrance Engineering, Harmondsworth,

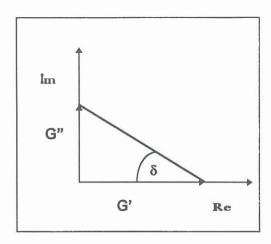


FIGURE 3: THE EXPLANATION OF THE SHEAR COMPLEX MODULUS AND DELTA

England. This test consists of placing a flour-bladed vane into the gel and rotating it to determine the torsional force required to cause the cylindrical surface to be sheared by the vane.

The yield stress was calculated from the measured maximum torque and the known cylindrical surface of yielding, defined by the dimensions of the vane. [Dzuy, N.Q., et al 1983]. For the gel measurements a vane 25.4mm diameter by 25.4mm in length and the torsion spring No.1, range 0-1.5lb.in, were used. The spring was calibrated [Zdilar, A.M., 1988], so the spring deflection, measured in degrees, could be converted to a torque, for yield stress calculation. The assumption was made that a uniform shear stress distribution existed over the end surfaces, which is true for only extremely small vanes [Dzuy, N.Q., et al, 1983]. The error in this assumption was approximately between 5-14%.

2) Shear Stress Relaxation Tests. Constant shear rate tests were performed on the Bohlin Rheometer for varying lengths of time. The yield stress was determined as the residual stress.

RESULTS AND DISCUSSION Oscillatory Tests

The oscillatory test performed on the Carrimed, CSL 50, Rheometer gave the result as shown in figure 4. From the test the value of δ was found to be approximately constant at 10°. This low value indicated the greater contribution of the elasticity effect to the gel than the viscous effect. The value of the storage modulus, G', increased linearly from ~ 6500 Pa at low frequencies to 10900 Pa at 4.6 Hz. From this frequency G' increased to 17720 Pa at 40 Hz, in a non-linear fashion. The loss modulus, G", remains fairly constant at 850 Pa until a frequency of 0.1 Hz. Then G" slowly, but steadily, increases to value of 2950 Pa at 40 Hz. The decreasing dynamic viscosity with increasing frequency is similar to the shear-thinning behaviour of the power law fluid. It has not been possible to compare the results quantitatively because of the large amount of different experimental variables.

Polymer melts have shown to possess, at intermediate frequencies, a constant loss modulus, G'= G'0 probably caused by a temporary network of entanglements, formed in a melt by long macromolecular chains, which appears to be permanent for this intermediate frequency bandwidth. [Marin,G., 1988].

Since our gel is made of flour, containing many molecular chains of complex molecules: such as proteins, lipids, and starch, it could be considered a special type of polymer melt. The scanning electron micrograph of the gel supports this view.

Shear Vane Tests.

Eight fresh samples of 25% wheat flour gel were subjected to a shear vane test. The values of the angular displacement obtained from these tests were recorded and the average calculated to be 18.46°. This value was converted to a value of torque, by the following equation:

$$T_m = K * \theta$$

$$\label{eq:where:K} \begin{split} \text{where}: \ K &= 1.009*10^{\text{-3}} \ \text{Nm/deg, calibration constant} \\ \theta &= \text{angular displacement, degrees} \\ T_m &= \text{maximum torque, Nm} \end{split}$$

To determine the yield stress from the maximum torque, the following equation was used [Dzuy, N.Q., 1983]:

$$\tau_{y} = \frac{T_{m}}{\frac{\pi * D^{3}}{2} \left(\frac{H}{D} + \frac{1}{3} \right)}$$

where : D, H = Vane diameter and height, respectively τ_y = yield stress

These equations gave the value for the yield stress as (542 +/- 11) Pa.

Shear Stress Relaxation Tests

These tests were carried out on the Bohlin Rheometer. The final value of the shear modulus, G, from the tests was used to obtain the yield stress from : $\tau = G*\gamma_0 = (515.45 +/- 27.38)$ Pa. This compares well with the value obtained by the shear vane test.

CONCLUSIONS AND RECOMMENDATIONS

The 25% wheat flour gel is a viscoelastic material with a very high elastic component, because of its low G" and delta. The relatively constant value of G" at intermediate and low frequencies seems to suggests a similar quasi elastic behaviour as a polymer melt. This is supported by the complex molecular structure of the gel. Further oscillatory tests are being undertaken with a varying gap size, and the possibilty that the gel could model a polymer melt investigated thoroughly.

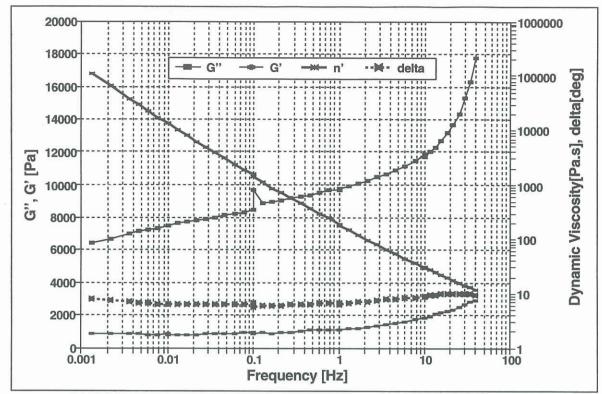


FIGURE 4: THE OSCILLATORY TEST RESULTS FOR A 25% WHEAT FLOUR GEL

The magnitude of the yield stress obtained from the shear vane test (542 +/- 11) Pa and the stress relaxation tests (515.45 +/- 27.38) Pa were compatible.

ACKNOWLEDGMENTS

We thank Dr Finlay MacRitchie of the CSIRO for his help.

Dr Tony Romeo, from the SEM unit at Sydney University, provided the SEM data.

REFERENCES

Biliaderis, C.G., Alexander, R.J., Zobel, H.F., 1992, Developments of Carbohydrate Chemistry, American Association of Cereal Chemists, United States of America

Dzuy, N.Q., Boger, D.V., 1983, "Yield Stress Measurement for Concentrated Suspensions", *Journal of Rheology* 27: 321-349

Faridi, H., 1987, Rheology of Wheat Products, American Association of Cereal Chemists, United States of America

Howling, D., 1980, 'The Influence of the Structure of Starch on its Rheological Properties', Food Chemistry 6: 51-61

Liu, J., Zhao, S., 1990, 'Scanning Electron Microscope Study on Gelatinization of Starch Granules in Excess Water", Starch 42: 96-98

Marin, G.,1988, "Chapter 10: Oscillatory Rheometry" in "Rheological Measurement", edited by Collyer, A.A, Clegg, D.W., Elsevier Applied Science, Great Britain

Miller, B.S., Derby, R.I., Trimbo, H.B., 1973, "A pictorial Explanation for the Increase in Viscosity of a Heated Wheat Starch-Water Suspension", Cereal Chemistry 50: 271

Nguyen, Q.D., Boger, D.V., 1992, "Measuring the Flow properties of Yiels Stress Fluids", Annual Review of Fluid Mechanics 1992, 24: 47 - 88

Zdilar, A.M., 1988, " Determination of the Yield Criterion of Grease", B.E. Thesis.