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Yield Stress Measurement of Semi-solid Foods Using the Back Extrusion (Annular Pumping) Method

presented by

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James F. Steffe
Major professor

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Yield Stress Measurement of Semi-solid Foods Using the Back Extrusion (Annular Pumping) Method

by

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A THESIS

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ABSTRACT

by

Aunur Rofiq Hadi

Three major factors in yield stress measurement using back extrusion (annular pumping) were considered: friction factor on plunger surface, ratio of plunger to sample tube radius, plunger velocity and depth of penetration.

A lubricated plunger compared to an unlubricated plunger, in evaluating yield stress revealed that friction on the plunger surface resulted in nonreproducible yield stress measurements. Smooth, lubricated plungers are recommended for yield stress determination.

As the ratio of plunger to sample tube radius approached one, a more erratic curve of force reduction during relaxation was produced. By using a wider annular gap, the erratic curve disappeared. Good results were achieved with plunger to container radius ratios less than 0.67.

Yield stress determined after subjecting a fluid to different plunger speeds or different the depths of penetration did not show significant differences. High yield stress foods needed a very long observation time (as long as four hours in this study) to reach equilibrium states, otherwise the yield stress determined using this method was still time-dependent.

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NOMENCLATURE

C_{sp}	chart speed of recorder, m/s
D_{gr}	diameter of a sample tube (gun rheometer), m
D_v	diameter of a vane, m
F	force, N
F_b	buoyancy force, N
F_T	total forces (back extrusion), N
F_{T_e}	total forces at equilibrium, N
H_v	height of a vane, m
K	ratio of plunger to cylinder radius, dimensionless
K_{HB}	consistency index (Herschel-Bulkley), $Pa\ s^n$
L	length of immersed plunger, m
L_{gr}	length of a sample (gun rheometer), m
L_{sp}	length of a spindle, m
M	mass of a dropping device (cone penetrometer), kg
P	air pressure, N/m^2
R_c	critical radius, m
R_{gr}	radius of sample tube (gun rheometer), m
R_i	radius of plunger, m
R_o	radius of graduate cylinder, m
R_{sf}	radius of plates (squeezing flow), m
R_{sp}	radius of a spindles, m
T	torque, $N\ m$
T_m	maximum torque, $N\ m$
V_{sf}	volume of a sample (squeezing flow), m
g	gravity acceleration, 0.981 m/s^2
h_{cp}	the depth of penetration (cone penetration), m
h_L	limiting height (squeezing flow), m

l_{ch} chart length, m

n flow behavior index, dimensionless

v_p velocity of plunger, m/s

Greek Symbols

α one half cone angle, degrees

$\dot{\gamma}$ shear rate, s^{-1}

$\dot{\gamma}_w$ shear rate on plunger wall (back extrusion), s^{-1}

η apparent viscosity, $Pa\ s$

μ Newtonian viscosity, $Pa\ s$

μ_0 high shear limiting viscosity (Bingham plastic), $Pa\ s$

μ_∞ high shear limiting viscosity (Ofoli et al.), $Pa\ s$

ρ density of a fluid, kg/m^3

σ shear stress, Pa

σ_0 yield stress, Pa

σ_w shear stress on plunger wall (back extrusion), Pa

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Chapter 1

INTRODUCTION

Back extrusion or annular pumping has been used for measuring rheological properties of various food materials. In this method two physical movements are involved : (1) a cylindrical plunger is forced down into a fluid and (2) the fluid flows upward through a concentric annular space. Back extrusion terminology comes from the fact that fluid motion during testing, is in the opposite direction to plunger movement. This method was first proposed to determine the characteristics of Newtonian fluids by Morgan et al. (1979). Later, a mathematical analysis of non-Newtonian fluids was developed by Osorio-Lira (1985). However, due to the fact that the calculation procedures for Herschel-Bulkley fluids are very complicated, Steffe and Osorio (1987) did not recommend these methods for industrial practice.

The back extrusion method has also been proposed as one of the techniques to measure yield stress directly (Osorio-Lira, 1985). The technique uses the stress relaxation technique in which the fluid, after being subjected to a constant shear rate, is allowed to rest and the remaining stress is taken as a function of the yield stress. The reliability of this method for measuring yield stress has been demonstrated for baby food by Steffe and Osorio (1987). However, its capacity to evaluate fluids with a high yield stress has not been explored. It is important that yield stress values calculated from back extrusion data be reproducible and independent of strain history.

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The objectives of this study, therefore, were: 1. To evaluate the capability of back extrusion testing to evaluate yield stress in fluids from "free-flow" to "semi-solid" types; 2. To determine if the yield stress measured by back extrusion is independent of plunger velocity and depth of penetration; 3. To improve experimental techniques in yield stress measurement using the back extrusion method by evaluating the effect of geometrical factors (plunger radius/container radius), lubrication and experimental surfaces (material characteristics) on results.

Chapter 2

Literature Review

2.1 Definition and Importance of Yield Stress

A yield stress may be defined as a minimum shear stress required to initiate flow. The yield stress marks the transition from elastic to viscous behavior; below the yield stress the substance is considered an elastic-solid and expected to follow Hooke's law; and above the yield stress, it is considered a fluid and may follow one of the fluid flow models such as Bingham plastic if linear, Herschel-Bulkley or Casson Eqs. if nonlinear.

Beyond the yield stress, the fluid flows with a shear rate that depends on the excess stress ($\sigma - \sigma_0$). If a fluid exhibits yield stress under the action of an applied constant shear rate, the shear stress will increase steadily until the yield stress is reached and then the stress will hold constant (Dzuy and Boger, 1983). The yield stress in this case is independent of the shear rate and can be shown as the interception of shear stress-shear rate flow curve at zero shear rate.

A yield stress is commonly found in highly concentrated emulsions under conditions where interparticle interactions bring about mutual attractions among individual particles (Dzuy and Boger, 1983). Princen's study (1985) with foams and highly concentrated emulsions found that the magnitude of yield stress was given by an interfacial tension, a volume fraction of the dispersed phase and the surface-volume mean drop radius. The yield stress may also be a result of physical entanglement of molecules or particles due to high degree of branching or irregular s

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shape, a network formation, a covalent or a secondary interparticles interaction, or an increased non-specific interaction between molecules or particles (Rha, 1978).

The experiment done by Barnes and Walters (1985) showed that a Bingham plastic fluid measured at very low shear rates (10^{-3} - $10^{-5}s^{-1}$) did not exhibit a yield stress. That experiment has raised the question of the existence of a yield stress. However, the concept of yield stress is still generally accepted by experimental rheologists. Cheng (1986) addressed the question using practical reasoning by noting that, when measuring the yield stress, it is not necessary to go to the lowest or zero shear rate, because yield stress is more important when related to process design or industrial purpose which has a limited residence time. Ofoli et al. (1987) also included a yield stress parameter in their model due to the fact that food manufacturing processes have strict time limitations.

The exact value of the yield stress should be known when food companies want to increase the efficiency of their operation or obtain better quality products. The qualities of food products which have a yield stress include shape retaining ability (cheese or gelatin), coating characteristics (chocolate products) and spreadability (margarine or butter). The thickness of salad dressing or tomato ketchup and the softness of some dairy products are also altered by the value of the yield stress. In the beverage and drug industry, the yield stress prevents the small particles from settling during storage.

Yield stress must be taken into account in process design and equipment specification due to possible adherence on vessels or pipes, the occurrence of dead regions during mixing operations, and the alteration of F-values in aseptic process due to unique velocity profiles in heat exchangers.

2.2 Measurement of Yield Stress

Yield stress may be obtained by an extrapolation of the rheogram to zero shear rate. If the rheogram is linear, such as found with the Bingham plastic model, the yield stress can be determined accurately. Unfortunately, many fluids exhibit nonlinear behavior at shear stresses above the yield value. As a result, one should go to as low a shear rate as possible to obtain a true yield stress.

In practice, it is difficult to measure the shear stress at a very low shear rate due to the limited capability of many rheometers. Direct measurement of yield stress without measuring shear rate, therefore, is very attractive. Numerous methods have been established, but many are tedious and need very specific conditions. It is not unusual that a yield stress obtained by one technique is non-reproducible and comparable to that obtained using a different technique. Therefore, some researchers believe that the yield stress may be time-dependent (Lang and Rha, 1981 and Cheng, 1986). It is difficult to define the yield stress as an absolute rheological parameter. Cheng (1986) also recommended that determination of yield stress must be made relevant to practical application. To minimize sedimentation in suspending fluid, for example, one should measure a yield stress at an extremely low shear rate. In contrast, to determine start-up power requirement in pipe line transportation or mixing operation, one should consider higher shear rates and a short measurement time. Table 1 shows yield stresses of food and nonfood materials which have been measured using different methods.

Table 1. Lists of some yield stress measurement methods.

Method	Material	σ_0 (Pa)	Reference
Indirect methods (extrapolation)			
Herschel-Bulkley	fish paste	1 800	Nakayama et al. (1980)
Herschel-Bulkley	meat batter 11 % fat 18 % fat 26 % fat	 587 239 148	Burge and Acton (1984)
graphically Bingham plastic Herschel-Bukley Casson	Titanium dioxide (37.3 % solid)	128 234 125 128	Dzuy and Boger (1983)
graphically Bingham plastic Herschel-Bukley Casson Heinz-Casson	miracle whip	26 54 30 39 23	Ofoli et al. (1987)

Method	Material	σ_0 (Pa)	Reference
Direct measurement	**Food**		
cone penetrometer @ 20 C	margarine cheese peanut butter	10 000 6 800 600	Tanaka et al. (1971)
squeezing flow	tomato paste ketchup mustard mayonnaise	120 26 56 84	Campanella and Peleg (1987)
balance plate @ room temp.	condensed milk corn syrup molasses ketchup mayonnaise tomato paste	2 2 3 16 25 84	De Kee et al. (1980)
stress decay stress to initiate flow	guar gum 2 % cornstarch 4.3% cornstarch 4.3%	4 20 12	Lang and Rha (1981)
cone and plate	mayonnaise	70	Elliot and Ganz (1977)
back extrusion	fruit-dessert baby food	20	Steffe and Osorio (1987)
	Nonfood		
squeezing flow	toothpaste Titanium dioxide (37.3 % solid)	4 128	Covey and Stanmore (1981)
concentric cylinder	kaolin Titanium dioxide	24 40	Vocadlo and Charles (1971)
stress relaxation	Titanium dioxide (37.3 % solid)	106	Dzuy and Boger (1983)
vane device	red mud (66 % solid)	168	Dzuy and Boger (1983)

2.2.1 Indirect Measurement

Determining the yield stress by extrapolation of shear stress-shear rate data is an indirect method since it requires rheological data from a previous measurement. There are two techniques which can be used in extrapolation: (1) to extrapolate raw data of the relationship between shear stress and shear rate directly, graphically; and (2) to extrapolate the data using a mathematical model determined via linear or nonlinear regression. Most common rheological instruments may be used to collect data to determine yield stress using indirect methods: capillary, concentric cylinder, cone-plate or parallel plate viscometers, etc.

The advantage of direct extrapolation of raw data is that it does not depend on any specific mathematical model whose validity must be established separately. A yield stress is simply determined from an extrapolation of the shear stress to zero shear rate or the shear stress where the apparent viscosity goes to infinity on a plot of apparent viscosity versus shear stress.

To get a more accurate result, Kaletung-Gencer and Peleg (1984) used a digitizer aided determination. However, the value of yield stress obtained from this technique can not be confirmed using any statistical method. In addition, the accuracy of the yield value depends on the reliability of the rheological data at low shear rates.

The other method is the extrapolation of a mathematical model which has been established separately based on experimental data. The most common Eqs. used to express behavior of fluids with a yield stress are the Bingham plastic, Herschel-Bulkley, Casson and Heinz-Casson models. The recent model proposed by Ofoli et al. (1987) is a generalization of those traditional models (Table 2).

Table 2. Mathematical models used to express rheological behavior of fluids with a yield stress.

Model Name	Shear stress	Apparent viscosity
Bingham plastic (Bingham, 1922)	$\sigma = \sigma_0 + \mu_0 \dot{\gamma}$	$\eta = \sigma_0 \dot{\gamma}^{-1} + \mu_0$
Herschel-Bulkley (Herschel and Bulkley, 1926)	$\sigma = \sigma_0 + K_{HB} \dot{\gamma}^{n}$	$\eta = \sigma_0 \dot{\gamma}^{-1} + K_{HB} \dot{\gamma}^{n-1}$
Casson (Casson, 1959)	$\sigma^{.5} = \sigma_0^{.5} + (\mu_\infty \dot{\gamma})^{.5}$	$\eta = \left((\sigma_0 \dot{\gamma}^{-1})^{.5} + (\mu_\infty)^{.5}\right)^2$
Heinz-Casson (Heinz, 1959)	$\sigma^{n} = \sigma_0^{n} + (\mu_0 \dot{\gamma})^{n}$	$\eta = ((\sigma_0 \dot{\gamma})^{n} + (\mu_0)^{n})^{1/n}$
Generalized model (Ofoli et al. 1987)	$\sigma^{n_1} = \sigma_0^{n_1} + \mu_\infty \dot{\gamma}^{n_2}$	$\eta = \left(\left(\frac{\sigma_0}{\dot{\gamma}}\right)^{n_1} + \mu_\infty \dot{\gamma}^{n_2 - n_1}\right)^{1/n_1}$

The value of yield stress predicted by each model (Table 2) from the same rheological data may be significantly different (Ofoli et al. 1987). In this case, the yield stress is strongly determined by a selected model and curve fitting technique rather than physical characteristics of the fluid itself. Although the mathematical model is very important for process design, a fundamental characteristic of the fluid like yield stress should be determined independent of those models for the most accurate result.

2.2.2 Direct Measurement

It is important to measure yield stress directly, independent of a particular mathematical model. Ofoli et al. (1987) showed that a specified yield stress can alter the value of the other rheological parameters for every model. Much attention has been given to direct measurement and many methods have been proposed. Unfortunately, most methods are still not reliable due to their limited applications and non-reproducible results. The methods are only consistent for specific materials or experimental conditions.

Two major methods applied in direct measurement involve the use of a force to generate flow and stress relaxation after an applied force. Another proposed method which has a very limited application is the oscillatory test (James et al. 1987). A yield stress may be obtained from response waves, where, below the yield stress, the response is a sine wave and above it a flat wave.

2.2.2.1 Force to Initiate Flow

The cone penetration test has been used to measure shearing stress since 1949 (Tanaka et al., 1971), and more recently was used to measure yield stress of margarine or butter (Dixon and

Parekh, 1979). The principle of this method is based on the assumption that, by applying a constant weight, the cone (Fig. 1) will penetrate into the material until an equilibrium point is reached. The shear force is given as $Mg\cos\alpha$ and the conical area of penetration is $\pi h_{cp}^2 \tan\alpha/\cos\alpha$. The yield stress, then, can be calculated from the equilibrium point:

$$\sigma_0 = \frac{gM\cos^2\alpha}{\pi h_{cp}^2 \tan\alpha} \tag{1}$$

where:

g - gravitation acceleration, 0.981 m/s^2

M - mass of a dropping device, kg

h_{cp}- the depth of penetration, m

α - one-half cone angle, degrees

Tanaka et al. (1971) found that the exponent of h_{cp} was not exactly two, but fell between 1.4 and 2.0 depending on the type and the temperature of the food. Dixon and Parekh (1979) used different cone angles for measuring the firmness of butter, and found that the expression of the yield stress was

$$\sigma_0 = \frac{M(2\alpha)^{-1.65}}{h_{cp}^2} \tag{2}$$

In addition, Keentok (1981) mentioned that the equilibrium might require penetration time of 12 hours or longer.

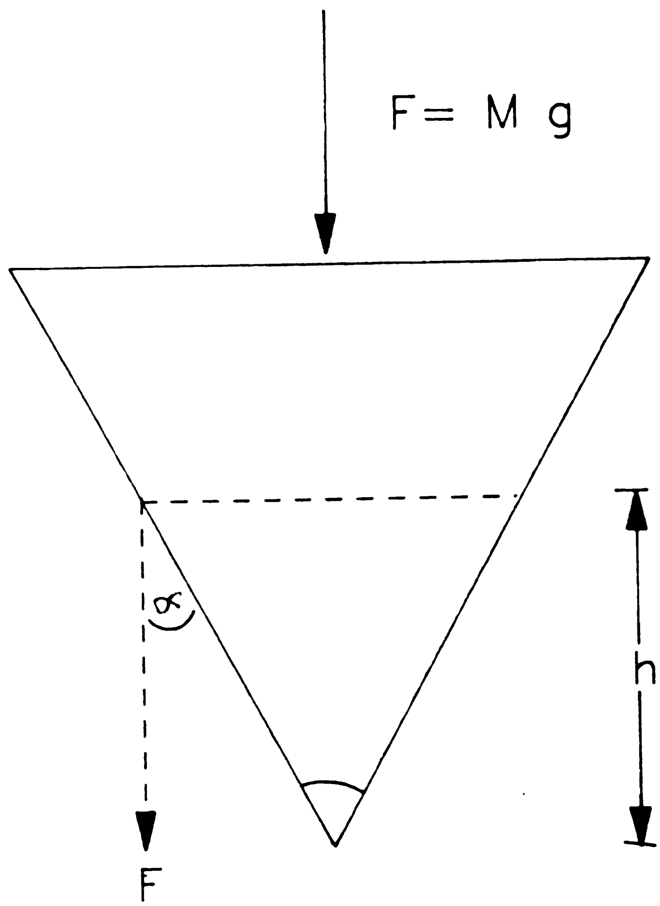

Figure 1. Cone penetrometer.

Squeezing film viscometers, which consist of parallel plates used to force substance movement with a constant force, have been called by several different names: parallel plate plastometer (Dienes and Klemm, 1946), parallel plate viscometer (Gent, 1960), transverse flow viscometer (Van Wazer et al. 1963), compression plastometer (Mooney, 1958) and squeezing flow viscometer (Leider and Bird, 1974). In squeezing flow, a yield stress can be determined from the limiting height or plate separation. The method was developed to characterize polymer melts, asphalt and other nonfood materials. Recently, Campanella and Peleg (1987) used it for measuring yield stress of semi-solid foods like tomato paste, ketchup, mustard and mayonnaise.

The theory of squeezing flow viscometer has been described in detail by Leider and Bird (1974). Measuring yield stress, in particular, using this method was described by Convey and Stanmore (1981). There are two geometrical versions called constant-volume in which the diameter of a sample is always less than the diameter of the plates and constant-radius in which the diameter of a sample is larger than the diameter of the plates.

The limiting separation is expressed by following Eqs.:
for constant-volume

$$h_l = \left(\frac{2V_{sl}^{1.5}\sigma_0}{3\pi^{0.5}F} \right)^{\frac{2}{5}} \tag{3}$$

for constant-radius, substitute $\pi R_{sl}^2 h$ to V_{sl}, so that

$$h_l = \frac{2\pi R_{sl}^3 \sigma_0}{3F} \tag{4}$$

where:

F - force applied to plates, N

R_{sf} - radius of plates, m

V_{sf} - volume of a sample, m^3

These Eqs. are good for both Bingham plastic and Herschel-Bulkley fluids since there is no motion at the limiting height situation (Covey and Stanmore, 1981). The disadvantage of the squeezing flow method is its inability to measure a fluid with a small yield stress or a free-flowing fluid.

One of the most popular viscometers is the concentric cylinder type. This instrument consists of an inner cylinder or spindle (bob) and an outer cylinder (cup). Besides measuring rheological properties under flow conditions, the concentric cylinder viscometer is also used in yield stress measurement. Three techniques may be used: stress to initiate flow; plug flow radius; and stress relaxation. The last technique will be described later in another section.

The torque on the bob of a viscometer when fluid starts to flow is a function of the yield stress. The technique was described by Lang and Rha (1981) as follows: the bob of a wide-gap viscometer was lowered into a sample, the sample was allowed to relax for ten minutes, before the outer cup was manually rotated. The stress at the onset of flow was designated as a yield stress:

$$\sigma_0 = \frac{T}{2\pi R_b^2 L_b} \tag{5}$$

where:

T - torque on bob when the fluid begins to flow, $N\ m$

R_b - radius of the bob, m

L_b - length of the bob, m

In the plug flow radius technique, a wide-gap viscometer is required, so there is a dead region of fluid when the bob is rotated. The yield stress, then, is calculated using the critical radius located between the flow and the dead region:

$$\sigma_0 = \frac{T}{2\pi R_c^2 L_b} \tag{6}$$

where:

R_c = critical radius, m

When the presence of slip is suspected, a grooved cylinder may be used along with a smooth cylinder as a comparison (Vocadlo and Charles, 1971). To eliminate end effects, Princen (1985) put mercury at the bottom of bob instead of an air bubble trap; however, this practice is not recommended for food products.

The vane device, used widely in soil mechanics, has received much attention for measuring yield stress in various chemical and food fluids. The advantages of using a vane device, instead of a concentric cylinder include no wall slip, no end effect and a minimum disturbance when the vane is introduced into the sample.

The theory and procedures of this device for measuring yield stress have been described elsewhere (Keentok, 1982, Dzuy and Boger, 1983,1985, and Keentok et al. 1985). In principle, a vane with 4 to 8 blades is immersed into a sample and then rotated very slowly at a constant speed. The torque on the vane shaft is recorded as a function of time. It reaches a maximum value when the stress applied to the sample is equal to yield stress and the vane starts to rotate.

By considering the geometry of the yield surface, the distribution of shear rate on the surface and the assumption that the shear stress is uniformly distributed around the tip of the vane and equal to yield stress, the simple relationship between σ_0 and T_m may be determined as

$$T_m = \frac{\pi D_v^3}{2}\left(\frac{H_v}{D_v} + \frac{1}{3}\right)\sigma_0 \tag{7}$$

where:

T_m - torque maximum, $N\ m$

D_v - diameter of the vane, m

H_v - height of the vane, m

The gun rheometer method was developed at the Warren Spring Laboratory in Stevenage, England, and a commercial instrument is produced by Deimos Ltd. (Cheng, 1986). This device is a horizontal tube viscometer where air pressure is applied to produce shear stress in a sample tube. The shear stress is calculated as

$$\sigma = \frac{\pi R_{gr}^2 (\Delta P)}{2\pi R_{gr} L_{gr}} \tag{8}$$

or

$$\sigma = \frac{D_{gr}}{4}\frac{\Delta P}{L_{gr}} \tag{9}$$

where:

R_{gr} - radius of inner sample tube, m

L_{gr} - length of sample in the tube, m

D_{gr} - diameter of inner sample tube, m

ΔP - air pressure, N/m^2

The yield stress, then, is determined at a minimum pressure drop required to initiate flow. There are two ways to obtain a minimum pressure. First is to use excess pressures, the curve of velocity versus pressure is obtained from applying different air pressure to the sample. By extrapolating that graph to zero velocity, a minimum pressure to initiate flow will be obtained. The second way is to use pressure below yield stress. The yield stress may be determined by rising the applied pressure gradually until there is a flow in the tube

2.2.2.2 Stress Relaxation

A yield stress may also be obtained using stress relaxation methods. A fluid, with characteristics between Hookian solid and Newtonian liquid, is subjected to steady shear. The stress, then, grows during steady shear until an equilibrium point which depends upon the rheological properties of the fluid. If the flow is suddenly stopped, the stress will gradually decay. For a Newtonian fluid, it will decay to a zero stress, but for fluids which have a yield stress, it will decay to a nonzero point which is a function of the yield stress.

Nagase and Okada (1986) investigated the behavior of fluid deformation and stress response during application of steady shear using a cone and plate rheometer. They identified two major shearing patterns; one for time-dependent fluids which have a yield stress dependent on the shearing history and one for time-independent fluids. Unfortunately, they did not examine the fluid behavior during stress relaxation.

Two methods have been proposed for measuring yield stress with a stress relaxation technique. In one method, a concentric cylinder device consists of a rotating inner cylinder called bob or spindle and a stationary outer cylinder called cup. Two techniques may be applied with this device. First is a technique described by Lang and Rha (1981). The bob is rotated to full torque, locked and lowered into a sample. After the sample is allowed to rest for enough time, the bob is released to drift back to equilibrium. The yield stress may be calculated from an equilibrium torque as

$$\sigma_0 = \frac{T}{2\pi R_{sp}^2 h} \tag{10}$$

The second technique was described by Dzuy and Boger (1983). The sample is loaded in the gap between bob and cup, and then the bob is rotated at a constant speed until an equilibrium condition is reached with an indication that the torque remains constant with time. The stress remaining on the bob after shearing is stopped, or in the relaxed state, is taken as a yield stress. The procedure described above is repeated several times at different constant speeds to obtain a true yield stress. The same technique may also be applied using cone and plate or parallel disk viscometers.

Another method using the stress relaxation procedure is back extrusion (Steffe and Osorio, 1987). This method will be described in detail in Chapter 3.

2.3 Time-Dependent Yield Stress

Most experiments in yield stress measurement show that yield stress is not an absolute value, but time-dependent. The dependence of yield stress on time is found along with material aging time and test observation time.

A thixotropic suspension, red mud (Nguyen and Boger, 1985) or bentonite (Cheng, 1986) had yield stress recovery during aging after being mixed. The increasing rate of yield stress development gradually decrease with time but may continue for over 1,200 hours with red mud and 7 days with bentonite. Therefore, the longer the material storage time, the higher the yield stress may be. Most yield stress suspensions are likely to have thixotropic characteristics (Nguyen and Boger, 1987).

On the other hand, the value of yield stress measured with some methods decrease with observation time. The methods that usually depend upon observation time are cone penetrometer (Keentok, 1982), squeezing flow (Campanella and Peleg, 1987) and stress relaxation method (Cheng, 1986). Thus it is almost impossible to compare the value of yield stress measured with different techniques without knowing the specific conditions of each material and experiment.

Chapter 3

Theoretical Considerations in Back Extrusion

3.1 Basic Principles of Back Extrusion

Back extrusion (annular pumping) has been used for measuring rheological properties of various food materials. With this method, a cylindrical plunger is forced down into fluid causing flow upward through a concentric annular space. The velocity profile of fluid flow during testing can be seen in Fig. 2.

To establish the mathematical relationship between parameters during testing, the following conditions are assumed (Osorio-Lira, 1985): constant temperature and fluid density; homogeneous isotropic fluid, no elasticity or time-dependent behavior; laminar and fully developed flow; the cylinders are sufficiently long that end effect may be neglected and no slip at walls of the annulus.

Total forces related to the plunger when it is being forced down into the sample with constant velocity and fluid is flowing upward in the annular can be evaluated using a force balance. The balance takes into account the force due to shear stress on the plunger wall, a hydrostatic or buoyancy force and the force responsible for fluid motion in the upward direction (Osorio and Steffe, 1987), as follows

$$F_T = 2\pi R_i L \sigma_w + \pi R_i^2 \Delta P + \rho g L \pi R_i^2 \tag{11}$$

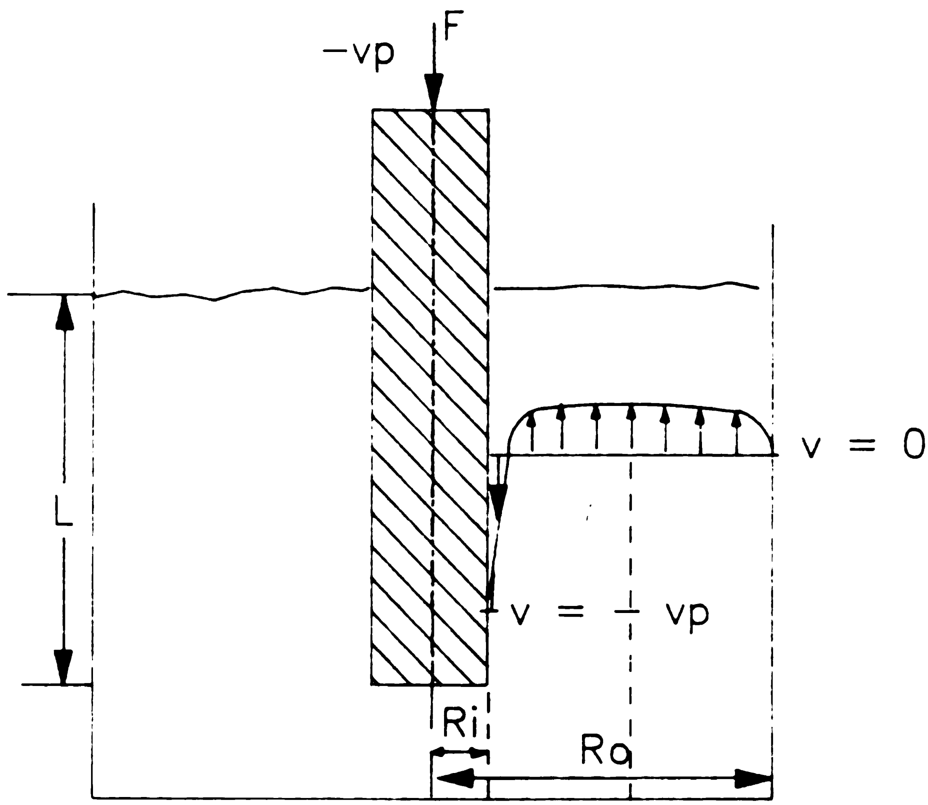

Figure 2. Profile of fluid velocity during plunger movement in back extrusion.

where:

F_T - total force employed on plunger, N

σ_w - shear stress on plunger wall, Pa

ρ - density of the fluid kg/m^3

L - length of immersed plunger, m

R_i - radius of plunger, m

3.2 Yield Stress Calculation

Back extrusion may be used to measure yield stress directly from typical Instron data (Fig. 3). During downward plunger movement with constant speed, the shear stress grows in proportion to the length of immersed plunger and the characteristics of the fluid. After the plunger is stopped the force will reach a equilibrium state (F_{T_e}). For a Newtonian or power law fluid, the equilibrium force will be equal to the buoyancy force (F_b), but for a non-Newtonian fluid with a yield stress the restraining force will be equal to the buoyancy force plus a residual force caused by the yield stress.

The force balance on plunger for Newtonian and non-Newtonian fluids may be expressed in terms of shear rate as:

during plunger movement, for Newtonian fluids

$$F_T = 2\pi R_i L(\eta\dot{\gamma}_w) + \pi R_i^2 \Delta P + \rho g L \pi R_i^2 \tag{12}$$

and for Herschel-Bulkley fluids

$$F_T = 2\pi R_i L(\sigma_0 + \eta\dot{\gamma}_w^n) + \pi R_i^2 \Delta P + \rho g L \pi R_i^2 \tag{13}$$

where:

$\dot{\gamma}_w$ - shear rate at plunger wall, s^{-1}

η - consistency coefficient, $Pa\, s^n$

σ_0 - yield stress, Pa

n - flow behavior index, dimensionless.

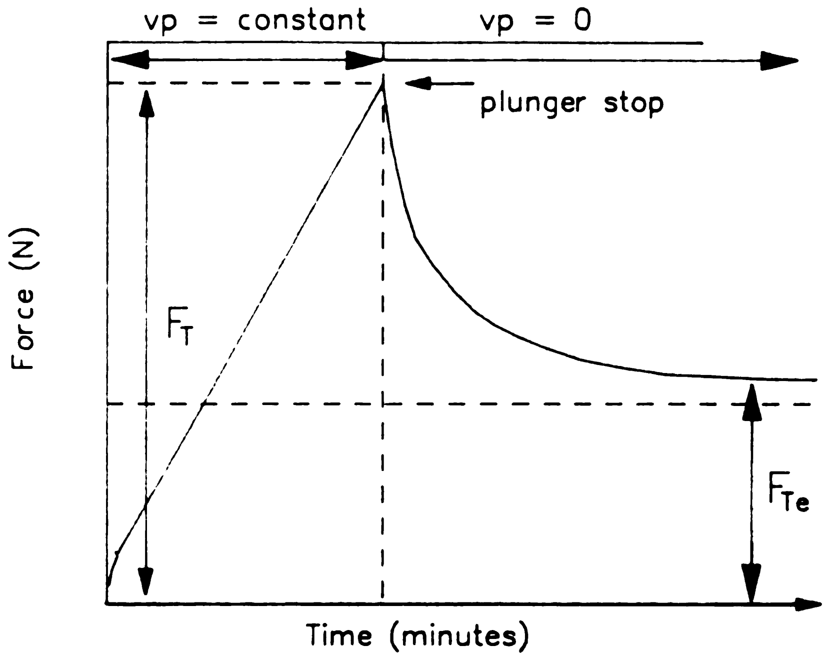

Figure 3. Typical Instron data obtained from back extrusion experiment.

At the equilibrium state, the shear rate and ΔP are equal to zero, and Eqs. (12) and (13) become:

$$F_{T_e} = \rho g L \pi R_i^2 \tag{14}$$

for a Newtonian fluid, and

$$F_{T_e} = 2\pi R_i L \sigma_0 + \rho g L \pi R_i^2 \tag{15}$$

for the Herschel-Bulkley (or Bingham plastic) fluid

where:
F_{T_e} = equilibrium force after cessation of plunger movement, N
Rearrangement of Eq. (15) gives the direct expression for the yield stress:

$$\sigma_0 = \frac{F_{T_e} - \rho g L \pi R_i^2}{2\pi R_i L} \tag{16}$$

The reliability of the back extrusion method to measure yield stress has been demonstrated for baby food by Steffe and Osorio (1987). However, its capacity to measure fluids with high yield stress has not been fully explored. Due to the lack of information on stress relaxation in back extrusion, some suspected factors that contribute significant effects during testing should be examined. These are plunger velocity, depth of plunger penetration, a friction factor on plunger surface and annular gap size.

3.3 Influence of Different Velocity and Depth of Penetration

As seen in Eqs. (12) and (13), a yield stress may be determined when the shear rate is zero. At this condition there is no force at the bottom of plunger due to upward flow of the fluid. The buoyancy force and the force due to yield stress must be distributed uniformly along the vertical surface of the immersed plunger. This condition will be fulfilled if there is no shear force remain after the fluid is allowed to rest for sufficient time. There must also be good contact between the fluid and immersed plunger surface.

To determine if a yield stress measured with back extrusion is independent of plunger movement, some different treatments have to be given to the variables related to plunger movement such as different plunger velocities and depths of penetration.

3.4 Influence of Surface Friction

Based on the mathematical expression described by Osorio-Lira (1985), the shear stress generated during plunger movement in back extrusion is function of a ratio of plunger to tube radius, the velocity of plunger movement, the depth of plunger penetration and a friction factor on the plunger surface.

The significant role of a frictional effect in uniaxial compression has been investigated by some researchers (Chatraei et al. 1981, Christianson et al. 1985 and Bagley and Christianson, 1988). They found that a frictional effect not only altered the magnitude of shear stress, but also contributed to non-reproducible results. That condition was due to the fact that the fluid deformation in unlubricated plates did not have uniform (and reproducible) contact with the plates.

A similar response may be expected in back extrusion when the surface of the plunger has a variable friction factor. This friction factor is very important in measuring rheological properties of fluids because a no slip condition at annular wall is required in mathematical analysis (Osorio-Lira, 1985). The yield stress measured using the stress relaxation method may depend on this friction factor. In addition, small annular gap size is suspected as a factor which contributes significant effects in force distribution along immersed plunger during relaxation. Unfortunately, no study has evaluated these factors with regard to yield stress measurement using back extrusion.

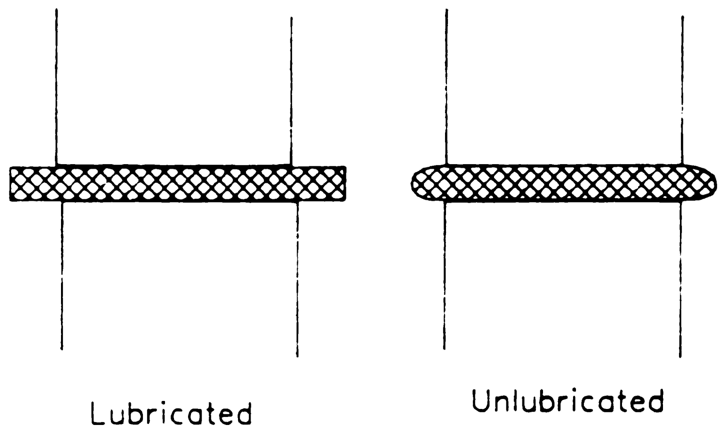

Figure 4. Profile of sample deformation in lubricated and unlubricated compression (Christianson et al. 1985).

Chapter 4
Materials and Methods

4.1 Experimental Materials

Foods with high yield stress were chosen for experimentation: tomato paste (Beatrice/Hunt-Wesson, Inc.), peanut butter (Groeb Farms, Inc.), mixed-cereal baby food (Gerber Products Co.) and chicken frankfurter prepared from mechanically deboned chicken in the Food Science Building at Michigan State University. Tomato paste (6 lb. cans), peanut butter (5 lb. buckets) and mixed-cereal (5 oz. jars) were purchased from the Michigan State University Food Store.

To prepare chicken frankfurter batter, 15 lbs block of frozen mechanically deboned chicken was cut into 2500 g blocks, wrapped in polyethylene, and stored at -30 ° C. A frozen mechanically deboned chicken block was stored at 4 ° C before being used the next day. Ingredients consisted of 2200 g mechanically deboned chicken, 33.5 g sugar, 9.2 g pepper, 54.85 g sodium chloride, 0.34 g sodium nitrite and 1.37 ascorbic acid, were weighed out.

Frankfurters were manufactured at room temperature (approximately 25 ° C. MDC, spice mix, salt and 500 g ice were placed in a cutter and chopped for 5 min. The batter, having a temperature approximately 4-7 ° C, was stuffed into graduate cylinders immediately after chopping.

In the experiments with a lubricated plunger, Vegalene pan coating (Tryson Company) was used as a lubricant.

4.2 Experimental Equipment

4.2.1 Back Extrusion Device

The Instron Universal Testing Machine (Instron Corporation), Model 4202, with a 50 N compression load cell was used as the main device in back extrusion (Fig. 5). This machine was connected to a Hewlet-Packard 86B computer as an input program device, a recorder and a printer as output devices. A plunger rod, used to force the fluid during testing, was screwed to the load cell on cross head. The Hewlet-Packard system was also used to control the Instron.

Three different diameters of smooth aluminum plunger rods were used: 20 mm, 24 mm and 28.58 mm. Two plunger rods (6 total) with the same diameter (28.58 cm), but different surface characteristics, smooth and grooved, were made from plexiglass, teflon and aluminum. Graduate cylinders of 100 ml, 250 ml and 500 ml capacity with diameters of 25.62 mm, 35.68 mm and 46.46 mm, respectively, were used as sample holders.

4.2.2 Gun Rheometer

A gun rheometer (Fig. 6) based on principles described by Cheng (1986) was built at Michigan State University. As shown schematically (Fig. 6) the wall of a pressure vessel was made of PVC (8.16 mm thickness) with a diameter of 15 cm and height of 38 cm. Top and bottom were also covered with plexiglass (19.18 mm thickness).

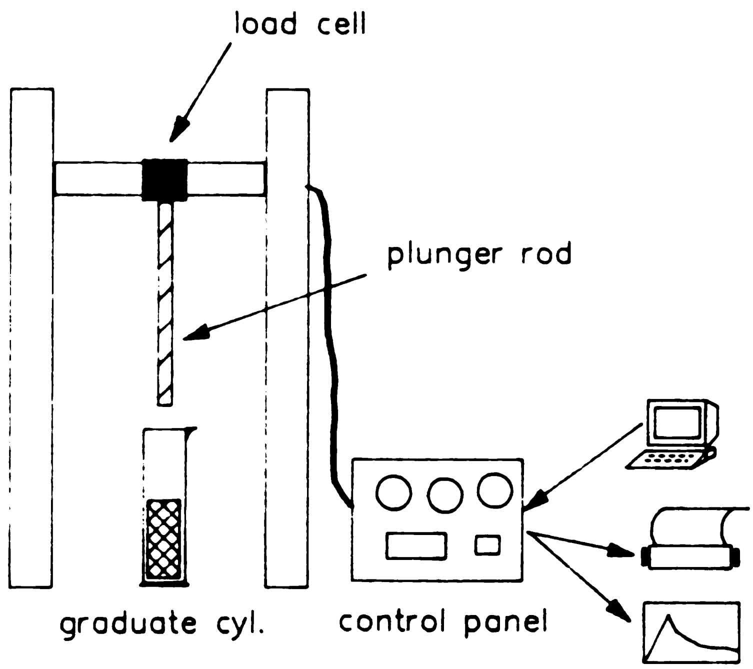

Figure 5. Schematic diagram of back extrusion device using an Instron Universal Testing Machine to collect experimental data.

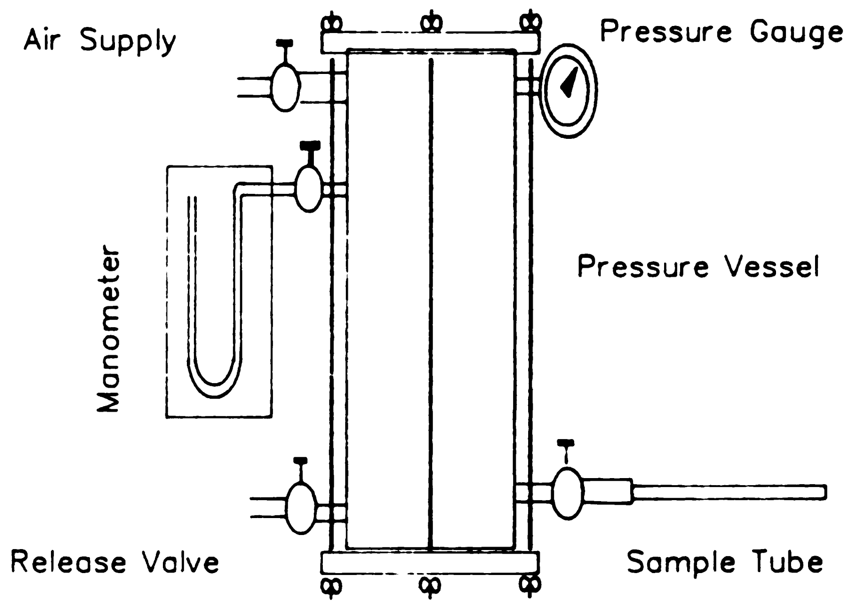

Figure 6. Schematic diagram of gun rheometer system.

To prevent the pressure vessel from leaking when subjected to high air pressure the top and bottom covers were sealed with rubber and screwed together with threaded steel bars and wingnuts. A safety valve was installed to release air if the pressure in the vessel because too high. The system was connected to a laboratory air supply. To measure air pressure in the vessel, a water manometer along with a dial pressure gauge were used. The dial pressure gauge was used when the air pressure was higher than 5 kPa.

Sample tubes, connected to vessel using a rubber tube, were made of plexiglass with 10 mm inside diameter, 2.75 mm wall thickness and 300 mm length.

4.2.3 Vane Device

The vane device (Fig. 7) used to measure yield stress in this study was similar to the device used by Dzuy and Boger (1985). The vane consisted of four blades centered around a small shaft. The diameter of the vane was 24.5 mm and the length of blades was 50.8 mm giving a H/D equal to 2.06. This ratio was still less than 3.5 as recommended by Dzuy and Boger (1985).

A Brookfield HBTD viscometer with full scale of 54,496 dyne cm was used as a rotational motor. The torque generated during testing was registered using a strip chart recorder and the maximum torque was calculated from the peak of the curve.

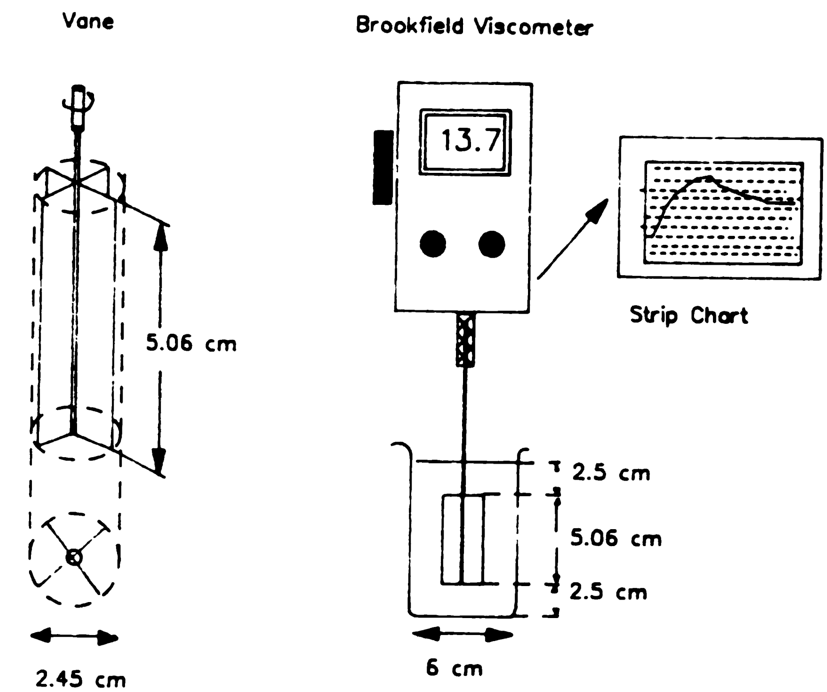

Figure 7. Schematic diagram of vane system.

4.3 Experimental Procedures

4.3.1 Operation of the Back Extruder

Two major problems in back extrusion testing are nonhomogeneous fluids and entrapped air bubbles during sample loading. Although the samples for each experimental series were taken from the same container, it was not uncommon to find that a sample taken from the top part was different from one taken from the bottom part of the can due to storage changes. To avoid inconsistency, the material in each container was stirred slowly and thoroughly before being loaded into sample holders. A container of sample might consist of several jars of baby food, a can of tomato paste, a bucket of peanut butter or chicken batter prepared at the same time.

To prevent entrapping air bubbles during sample loading, two techniques were applied. The first technique, applied to peanut butter, involved melting the sample at 50 $°C$ and pouring it into a graduate cylinder. With the other technique, applied to tomato paste and chicken batter, the sample was loaded using a stuffer (Fig. 8). Mixed cereal did not need a special treatment. It was just poured slowly into a graduate cylinder.

It is important to describe the essentials of sample loading using stuffer. The stirred sample was put into the cylinder and forced down. During this action, the entrapped air left through the gap between the plunger and the cylinder wall. The fluid forced through the tube was expected to have no entrapped air bubbles. It was, then, loaded into a graduate cylinder starting from the bottom. The stuffer tube was slowly removed during loading to ensure uniform fill.

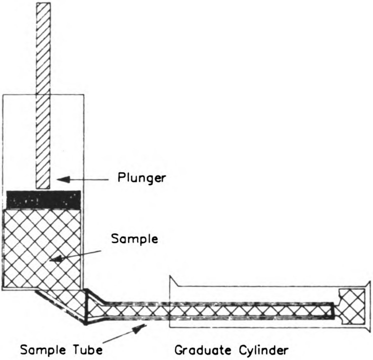

Figure 8. Stuffer as a loader for tomato paste and chicken batter.

The loaded graduate cylinders were allowed to rest for 24 hours before testing. Chicken batter was stored at 5 °C and the other fluids were stored at the room temperature, approximately 25 °C. During storage, samples were covered with aluminum foil to eliminate surface drying. By allowing a 24 hour rest period, it was expected that strain history due to loading would not influence subsequent tests. The density of a fluid was calculated from the weight per unit volume of the loaded samples.

Yield stress measurement procedures using back extrusion in this study was the same as procedures described by Steffe and Osorio (1987). The sample was subjected to a constant shear rate (constant plunger velocity) using the Instron Universal Testing Machine. Testing was conducted at room temperature and the force on the plunger versus time was recorded on a strip-chart recorder. In addition, every two minutes the magnitude of the force was printed out. After one minute of downward motion, the plunger was stopped and the fluid was allowed to relax. The force on the plunger was recorded until an equilibrium state was reached.

Before the yield stress may be calculated (Eq. 16) a number of variables must be evaluated. The equilibrium force F_{T_e} can be read from the recorder. Density of the fluid, gravitational acceleration and the radius of plunger are known. The length of immersed plunger must be determined.

The length of plunger (Fig. 9) that penetrated the fluid is $\overline{OB}$ and the volume of fluid displaced by the rod is equal to $\pi R_i^2 \overline{OB}$. Since the displaced fluid is forced up, around the annulus, the following relationship must be valid (Osorio and Steffe, 1985):

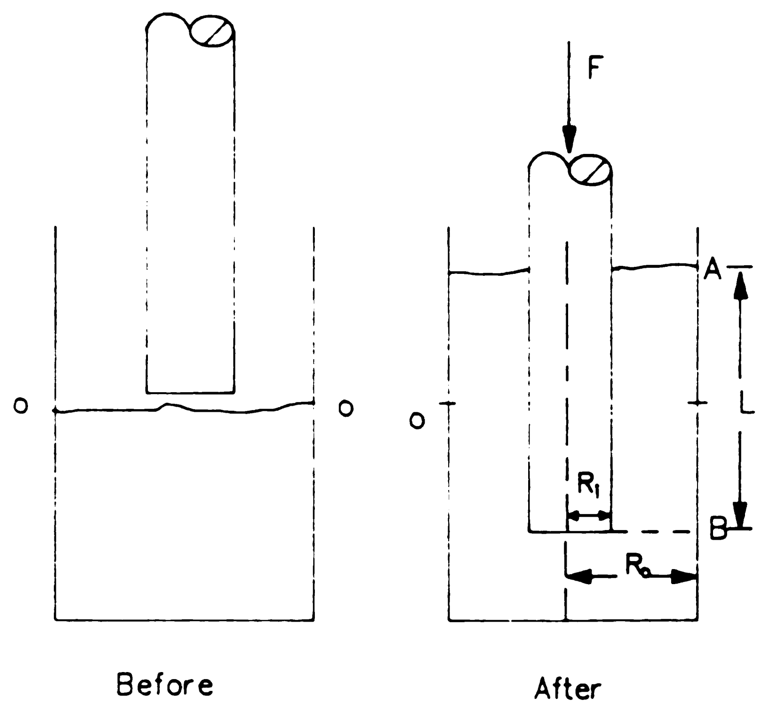

Figure 9. Position of plunger before and after testing.

$$\pi R_i^2 \overline{OB} = \pi R_o^2 \overline{AO} - \pi R_i^2 \overline{AO} \tag{17}$$

or

$$\overline{AO} = \frac{R_i^2}{R_o^2 - R_i^2} \overline{OB} \tag{18}$$

and

$$L = \overline{AO} + \overline{OB} \tag{19}$$

by putting Eq. (18) into Eq. (19), the value of L will be

$$L = \frac{\overline{OB}}{1 - K^2} \tag{20}$$

Where:

$K = R_i / R_o$

$\overline{OB}$ can be determined from chart length as

$$\overline{OB} = \frac{l_{ch}}{C_{sp}} v_p \tag{21}$$

where:

l_{ch} = chart length, from recorder, m

C_{sp} = chart speed of the recorder, m/s

v_p = velocity of the plunger, m/s

By knowing the value of L and the other relevant variables, the yield stress of fluid may be calculated using Eq. (16).

4.3.2 Operation of the Gun Rheometer

Samples for gun rheometer testing (Fig. 6) were prepared from the same material used in back extrusion testing. It was loaded into a sample tube using a Model 1142 grease gun made by Lincoln-St. Louis. The length of sample, as recommended, was more than ten times its diameter. The entrapped air in the loaded sample was removed with a syringe. As in back extrusion testing, the samples were covered with aluminum foil and allowed to rest for 24 hours before testing. Except for chicken batter that was stored at 5 °*C*, all samples were stored at room temperature.

The technique of excess pressure as described in Section 2.2.2.1 was used in this study. For satisfactory measurements, a variety of velocities had to be obtained; thus, a different level of excess pressure had to be applied to each sample.

Before a sample was tested with the gun rheometer, the air pressure in the vessel was set to a pressure higher than the pressure needed to initiate flow. By opening the air supply valve, the air pressure in the vessel was increased to line pressure. Then the valve was closed and if the air pressure in the vessel was too high a pressure adjustment was made using the release valve. The water manometer was only used if the air pressure in the vessel was below 5 kPa.

Excess pressure was applied to each sample for two minutes and the discharged sample was measured. The data collected from all samples were plotted as pressure versus mass average velocity (volumetric flow rate divided by cross-sectional area). By extrapolating to zero velocity, the minimum pressure to initiate flow was obtained. The yield stress, then, was calculated from this value using Eq. 9.

4.3.3 Operation of the Vane Device

Samples were prepared in the same manner as that described for back extrusion. The only important difference was the sample holders for the vane device: glass containers with diameters of 6 cm and heights of 10 cm.

The vane spindle (Fig. 7) connected to the Brookfield was introduced smoothly into a sample until the top of the vane was immersed to a depth approximately equal to the vane diameter (2.45 cm). After waiting for approximately 10 minutes, the vane was rotated as slowly as possible with a constant speed (0.5 rpm). The torque on the shaft was recorded as function of time. Maximum torque was determined from a maximum value on the digital window of Brookfield viscometer multiplied by full scale torque value of the device and divided by 100. The yield stress of the fluid was calculated from the maximum torque (Eq. 7).

4.4 Experimental Design

To fulfill the objectives of this study (as mention in the Introduction), yield stress of a fluid was measured using back extrusion with different plunger surface characteristics and geometrical factors. Each treatment was tested with three replications. The average yield stress of one treatment was compared statistically to that of another treatment using t-student distribution at 0.05 confidence level. If the two treatments did not have a significantly means difference, the variances of the yield stress were compared to analyze their uniformities. The following experimental design fulfilled the objectives.

4.4.1 Back Extrusion

4.4.1.1 Smooth Versus Grooved Plunger

Three smooth plunger rods made of aluminum, teflon and plexiglass respectively were used in this experiment. The selection of the three materials was to assess whether or not the yield stress measured with plungers made of different materials would alter the calculated value. These plungers had the same diameter

(28.56 mm) and all samples were held in 250 ml graduate cylinders. Thus, K was held at a constant value of 0.80. This follows the recommendation by Marte-Guzman (1987) for measuring rheological properties during back extrusion testing.

To examine the no slip condition required in the back extrusion method, as described by Osorio-Lira (1985), three ribbed plunger rods were tested. These grooved plunger rods were made of the same material and diameter as the smooth plunger rods. The plunger rods were ribbed horizontally with 1.25 mm width and 0.72 mm depth grooves, so that there were 8 grooves per cm of plunger length.

To measure yield stress of the fluids, a sample was subjected to a constant downward plunger velocity of 100 mm/min. After one minute, the plunger was stopped and the fluid was allowed to relax. The forces on the plunger surface during relaxation were recorded on the strip chart paper. The equilibrium force was determined one hour after the plunger was stopped. Testing was repeated three times for every plunger; thus (using 6 plungers) there were a total of 18 experiments. The yield stress values measured with different plungers were compared and examined.

4.4.1.2 Lubricated Versus Unlubricated Plunger

This experiment referred to the study done by Christianson et al. (1985), which revealed that friction factors on the plates during compression testing made a large contribution to nonreproducibility of results. The same problem may be found in back extrusion testing when measuring yield stresses with unlubricated plungers.

To evaluate whether a friction factor on the plunger surface had an effect on yield stress measurement, the results from a lubricated and an unlubricated plunger were compared. The friction factor on the plunger surface of a lubricated plunger was reduced by lubricating the plunger with vegetable oil (pan coating oil from Vegalene).

In the lubricated plunger experiment, the shear rate reduced to a small value. The total forces on the plunger when measuring fluids with yield stress, therefore, equal to the total of the force due to yield stress, buoyancy force and the force responsible for fluid motion. The Eq. 13 may be expressed as

$$F_T = 2\pi R_i L \sigma_0 + \pi R_i^2 \Delta P + \rho g L \pi R_i^2 \tag{22}$$

Tomato paste and peanut butter were tested using lubricated and unlubricated aluminum plungers with diameter of 28.56 mm, three replications for each plunger. The 250 ml graduate cylinders were used as sample holders and the yield stress was determined one hour after the plunger was stopped.

4.4.1.3 Ratio of Plunger to Tube Diameter

The distance between solid boundaries in back extrusion represented by annular gap may influence the yield stress calculation. A narrow annular gap may retard the relaxation. To observe the effect of solid boundaries on yield stress measurement, three different annular gaps, expressed as the ratio of plunger to graduate cylinder diameter, were used: K values of 0.78, 0.62 and 0.52. The equilibrium force measured one hour after the plunger was stopped and the curve of force reduction on the plunger versus time were recorded for a three hour period.

4.4.1.4 Different Plunger Velocities and Depths of Penetration

The yield stress was determined from the force at equilibrium. At that state, no plunger movement effect was expected. To observe whether plunger movement affected the equilibrium force, a fluid was tested using different plunger speeds and different the depths of penetration. The characteristics of the plunger and the ratio of plunger to tube diameter were chosen from earlier experiments.

4.4.1.5 Time-Dependent Yield Stress

Due to the fact that most yield stress value measured using stress relaxation depend upon the observation time, it was important to examine whether or not the yield stress measured at one hour after the plunger stopped was time-dependent. Therefore, data of force on the plunger versus time was plotted for three hour periods.

4.4.2 Comparison of Back Extrusion to Other Methods

The best experimental techniques for back extrusion were established from the result of the previous experiments. This technique, then, was used to measure yield stress of four fluids: chicken batter, tomato paste, peanut butter and mixed cereal baby food. To validate these results, they were compared to other yield stress measurement methods.

4.4.2.1 Comparison to Gun Rheometer Data

The same fluids considered in back extrusion testing were measured using a gun rheometer. A yield stress was determined using an excess pressure technique and linear regression. The yield stress of each fluid obtained from the regression method was compared to the average yield stress of each fluid obtained from the back extrusion technique.

4.4.2.2 Comparison to Vane Device Data

The lowest rotational speed of the Brookfield equipment was 0.5 rpm; however, the fluids were tested using three rotational speeds (0.5,1.0 and 2.5 rpm). If the yield stresses measured using these three speeds had different values, the yield stress at zero rpm (determined using extrapolation method) was taken as the actual value.

Chapter 5

Results and Discussion

5.1 Back Extrusion

5.1.1 Smooth Versus Grooved Plunger

Table 3 shows the average yield stress values and standard deviations obtained from back extrusion using smooth and grooved plungers. The values of the yield stress are very high in deviation. Thus, it is impossible to determine the true value of yield stress from those data, no single plunger produced consistent results. For example, the average yield stress of tomato paste measured with smooth plexiglass is 138.1 Pa and standard deviation is equal to 121.9 Pa. It means as Chebyshev's rule guarantees (Bhattacharyya and Johnson, 1977), at least 75 percent (2 std dev.) of the yield stress measured with the same condition will be varied from minus 105.7 Pa to 381.9 Pa.

The smallest standard deviation is for the yield stress of chicken batter when measured with smooth plunger made of teflon, but this plunger yielded very high standard deviations when measuring other foods.

Therefore, the method used by Steffe and Osorio (1987) can not be applied to measure a yield stress of semi-solid food which have a high yield value. The reason why the method did not perform well with these materials is difficult to explain.

Table 3. Yield stress of semi-solid foods measured using different unlubricated plunger surfaces with K=0.80.

	Tomato paste		Peanut butter		Chicken batter	
Plunger surface	Average (Pa)	Std.dev.	Average (Pa)	Std.dev	Average (Pa)	Std.dev.
Plexiglass						
smooth	138.1	121.9	55.8	25.8	556.7	54.7
grooved	204.9	53.9	190.0	53.9	747.3	49.9
Teflon						
smooth	202.1	143.1	88.8	131.9	759.7	38.0
grooved	126.3	25.7	130.8	121.7	732.7	44.0
Aluminum						
smooth	132.6	80.1	111.1	48.7	570.7	144.9
grooved	109.6	96.9	137.1	68.9	842.4	102.2

The possibility of friction on the surface of plunger or entrapped air bubbles at grooved plunger resulted in nonuniform distribution of forces along the plunger.

The poor reproducibility was also found by Dzuy and Boger (1983) when measuring red mud suspensions using stress relaxation method. However, their conclusion, that the problem was due to a slip effect, can not be proved in this experiment.

5.1.2 Lubricated Versus Unlubricated Plunger

A lubricated plunger was used to reduce errors due to surface friction has been applied in back extrusion. The results of yield stress measurement can be seen in Table 4. It shows that yield stress values obtained using a lubricated plunger are more consistent than those found using an unlubricated plunger.

Although the average yield stresses are different when measured using a lubricated or an unlubricated plunger, the difference is not statistically significant at 0.05 confidence level. However, the variance between the lubricated and the unlubricated plungers are significantly different at 0.05 confidence level.

Thus, the values of yield stress measured using a lubricated plunger are more uniform than using an unlubricated plunger. It may be concluded that surface friction has a strong influence on yield stress measurement. The best reproducibility in experimental data will be obtained using a lubricated plunger when measuring yield stress using the back extrusion technique.

Table 4. Yield stress measured using lubricated and unlubricated aluminum plunger.

Plunger surface	Yield stress (Pa)	
K=0.80	Peanut butter	Tomato paste
Lubricated	61.9 58.8 71.9	145.5 152.0 175.1
Average Std. dev.	64.1 6.9	157.6 15.6
Unlubricated	58.4 154.6 120.4	224.1 98.1 230.7
Average Std. dev.	111.1 48.8	184.3 74.7

Typical shape of fluid after the completion of a back extrusion test is illustrated in Fig. 10. For the lubricated plunger, the fluid filled the annular space at the same level but for the unlubricated plunger it did not. It is possible that the friction factor on the plunger surface may inhibit upward fluid movement and the development of the velocity profile with zero slip.

The magnitude of buoyancy force may also be altered by this shape. In addition, it may create a nonuniform distribution of force along the immersed plunger surface bringing about an error in the calculation of the yield stress. This response is similar to the result found by Christianson et al. (1985) in uniaxial compression experiments where an unlubricated plate altered the distribution of force (Fig. 4).

5.1.3 Ratio of Plunger to Tube Diameter

The effects of annular gap during stress relaxation can be seen in Table 5 and Fig. 11. Yield stress determined after one hour observation reveals that higher K values produced smaller calculated yield stresses. The differences are statistically significant at 0.05 confidence level compared to either yield stress measured with K=0.67 or K=0.52. Yield stresses measured with K= 0.67 had lower values than yield stresses measured with K= 0.52. However, the differences are not statistically significant at a confidence level of 0.05. For longer observation time (until three hours), yield stress decreased faster for the higher K value.

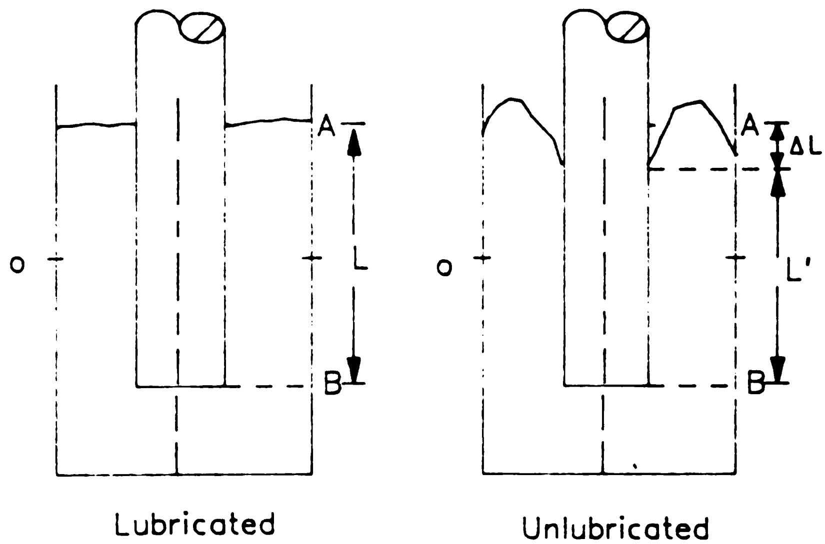

Figure 10. Typical shape of fluid in relaxed state for smooth, lubricated and unlubricated plungers.

The consistency of result are also affected by annular gap. The smaller K value, the more uniform the yield stresses obtained. Good reproducibility of experimental data is very important in yield stress measurement because most methods have failed to produce uniform results. Thus, it may be concluded that reproducible data will be obtain in back extrusion testing to determine yield stress when using a lubricated plunger and K = 0.52.

The effect of solid boundaries may be explained on the basis of the force balance along the immersed plunger during stress relaxation. Two possibilities may affect the distribution: shear rate history and solid boundaries, because a narrower annular gap also produce higher shear rate. However, due to the purpose of this experiment only to investigate solid boundaries effect, it is assumed no strain history effect was present at equilibrium.

The experiments show that a narrower annular gap (higher K value) results in more rapid relaxation and sometimes a force with opposite direction to buoyancy force occurs on the plunger (negative balance at three hour period). The negative yield stress may not be interpreted in physical meaning, but it may due to a mistake of mathematical expression. This phenomenon, which can not be understood, was also found when polyisobutylene in primol was subjected to a high shear rate (Bird et al., 1987). The stress, then, recovered to the same equilibrium as found with lower shear rates for longer observation times. It also appeared in back extrusion tests with high K values when observed for 24 hours, but the data is not reliable due to dehydration during the observation period.

Table 5. Yield stress of tomato paste measured using lubricated aluminum plungers, with different ratios (K values) of plunger to inner graduate cylinder radius after various relaxation period.

Ratio plunger to tube radius	σ_0 (Pa) 1 hour	σ_0 (Pa) 2 hours	σ_0 (Pa) 3 hours
K=0.78	62.3 73.7 11.4	47.7 8.2 -4.4	-14.1 -14.1 -11.4
Average Std.dev.	49.1 33.2	17.2 27.2	-13.2 1.5
K=0.67	69.2 100.0 106.0	57.4 66.6 86.7	40.7 37.6 72.5
Average Std dev.	91.7 19.7	70.2 15.0	50.3 19.3
K=0.52	94.6 105.4 110.1	73.0 101.6 105.4	63.2 87.2 91.8
Average Std.dev.	103.4 7.9	93.3 17.7	80.7 15.4

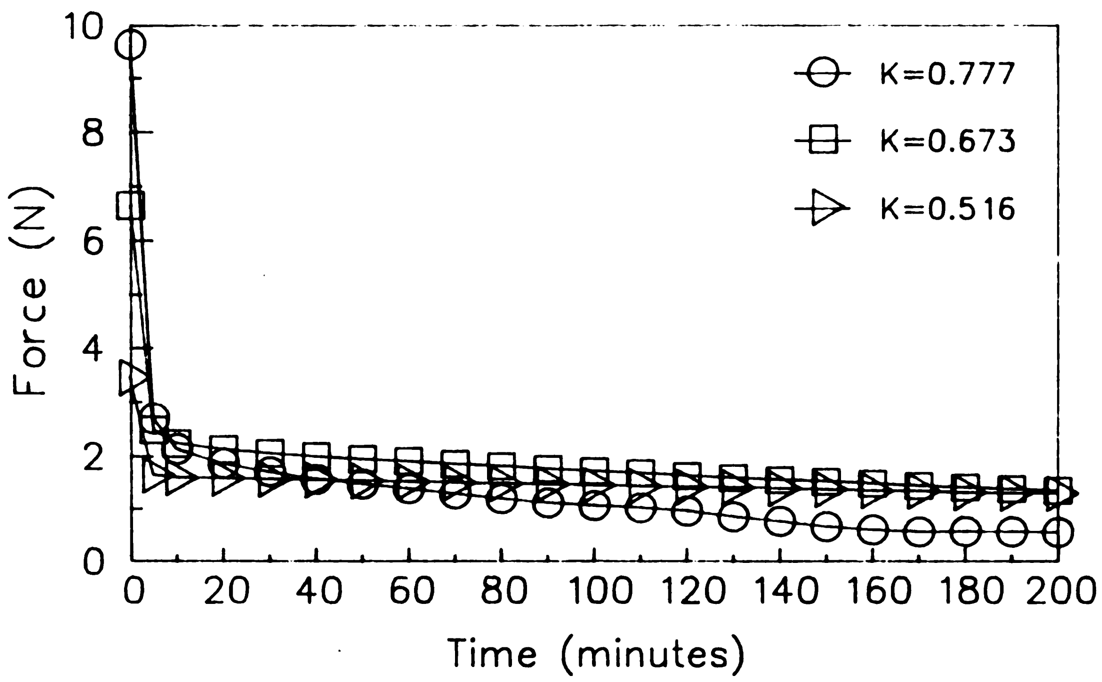

Figure 11. Stress relaxation curve of tomato paste in back extrusion using a lubricated plunger and different annular gap.

5.1.4 Different Plunger Velocities and Depths of Penetration

The early observations revealed that friction on the plunger surface and a narrow annular gap had strong effects on yield stress evaluation of semi-solid foods. To investigate whether plunger movement affected the measurement of yield stress, other effects should be eliminated or constant, so a lubricated aluminum plunger and a wide annular gap (K=0.52) were used.

There are two ways to obtained different plunger movement in back extrusion at the same annular gap with the lubricated plunger. First, the foods are measured with the same depth of penetrations but different plunger velocities. Second, the foods are measured with the same plunger velocities but different depths of penetration as in Fig. 12 and 13, respectively.

Fig. 12 demonstrates that different plunger velocities do not affect yield stress measurement even though they result in different shear stresses. In three hours of observation, the yield stress of chicken frankfurter batter is still time-dependent (Fig. 12 or Table 6). The yield stresses measured every hour do not have significant differences between 50 mm per minute velocity and 100 mm per minute velocity.

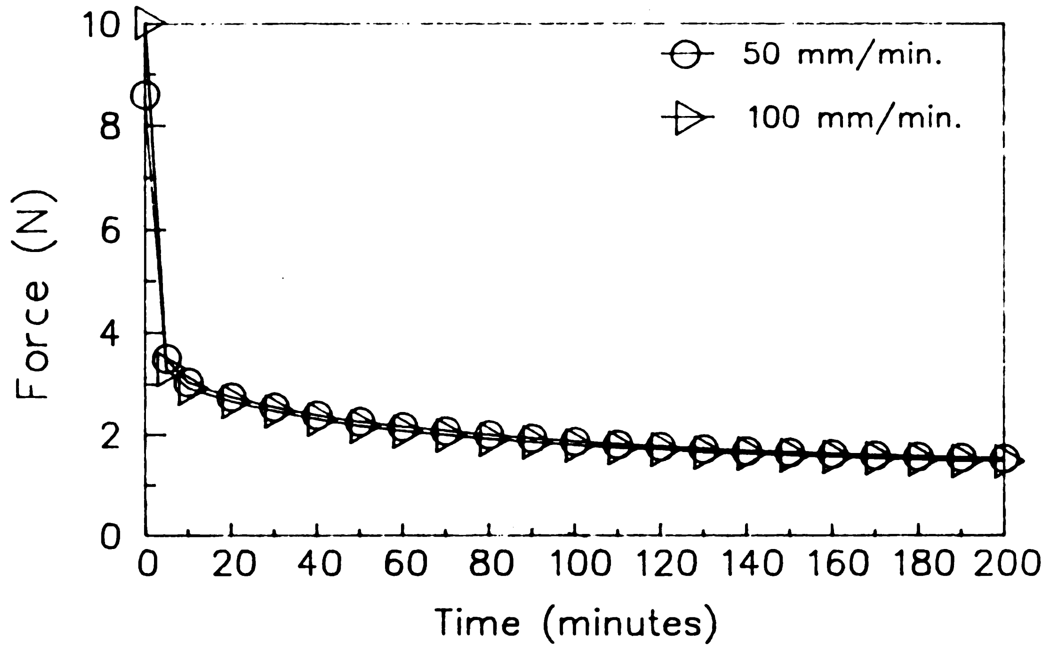

Figure 12. Stress relaxation curve of chicken batter in back extrusion using a lubricated plunger and different plunger velocities.

Table 6. **Yield stress of chicken batter measured using different plunger velocities with a lubricated aluminum plunger and K=0.52.**

Plunger velocity	σ_0 (Pa) 1 hour	σ_0 (Pa) 2 hours	σ_0 (Pa) 3 hours
50 mm/minute	177.5	140.0	119.2
	190.8	144.3	119.5
	188.8	138.2	115.0
Average	185.7	140.8	117.9
Std. dev.	7.2	3.1	2.5
100 mm/minute	171.0	139.2	119.8
	202.7	150.9	122.8
	178.5	133.5	111.2
Average	184.1	141.2	117.9
Std. dev.	16.6	8.9	6.0

On the other hand, Fig. 13 shows that different depths of penetration have produced different equilibrium forces. These forces were observed only for one hour period. Since yield stress is calculated from equilibrium forces, with consideration of the depth of penetration or the length of immersed plunger, we are not able to see the value of those yield stresses in Fig. 13. However, Table 7, shows that the yield stresses calculated from Fig. 13 have almost the same magnitude. The equilibrium forces per unit length of immersed plunger also can be seen in Table 7.

However, different depths of penetration (Fig. 14) have different results. When plunger penetration is close to the bottom of sample tube. The end effects produce erratic curves even to a negative force when operated 1.5 cm from the bottom of sample tube. Therefore, to eliminate this effect, the plunger has to be operated far enough (at least 3 cm for the current study or 1.5 of plunger diameter) from the bottom of the sample tube.

Table 7. Yield stress of tomato paste measured with different depth of penetrations using lubricated aluminum plunger with K=0.62.

Length of immersed plunger (m)	F_{T_e} (N)	σ_0 (Pa)	F_{T_e}/L (N/m)
0.110	2.4	166.3	22.1
0.129	2.7	152.3	20.9
0.173	3.5	145.4	20.3
0.194	4.0	152.0	20.8
0.227	5.2	175.1	22.9
0.270	5.6	149.2	20.6
Average		156.7	
Std. dev.		11.4	

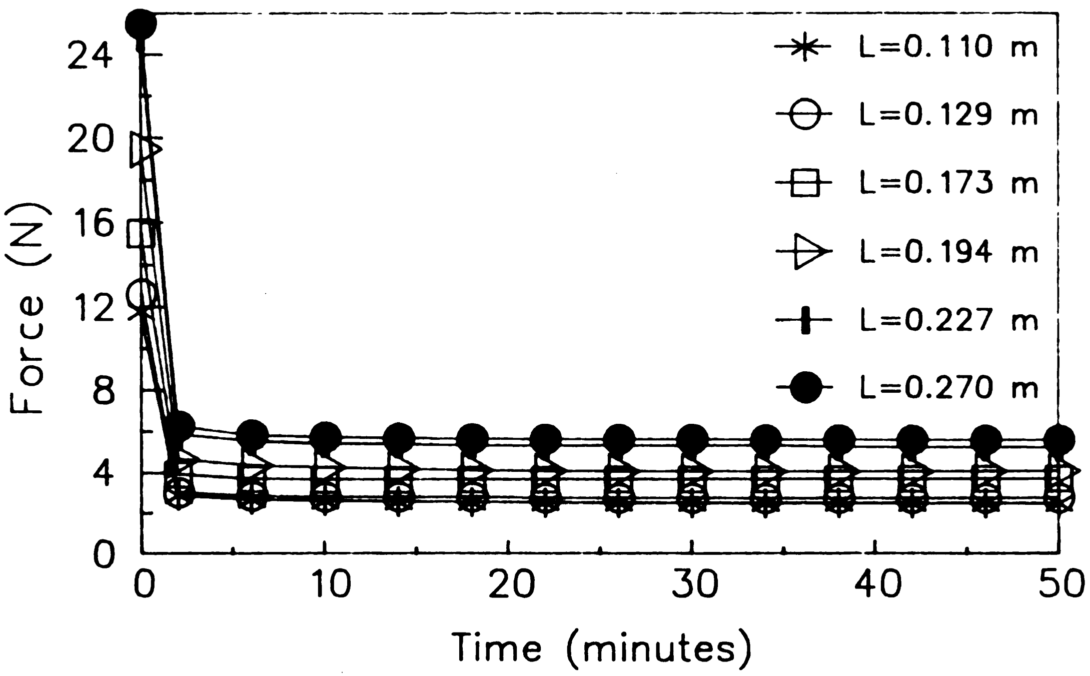

Figure 13. Stress relaxation curve of tomato paste in back extrusion using a lubricated plunger and different depths of penetration.

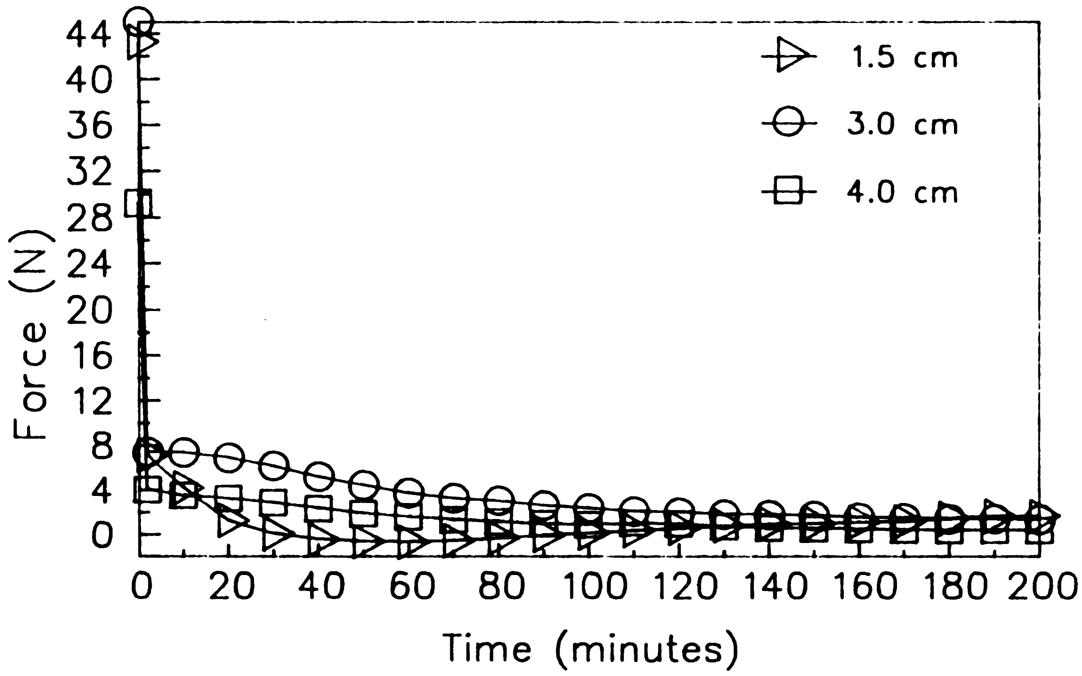

Figure 14. Stress relaxation curve of tomato paste in back extrusion a lubricated plunger and different end distances.

5.1.5 Time-Dependent Yield Stress

The magnitudes of equilibrium forces and yield stress calculated from them were determined for data one hour after the plunger was stopped. At that time it is expected that the force has already decayed to equilibrium state and strain history has no affect on the force on the plunger surface.

However, in a longer observation time (Table 8), the value of yield stress calculated during stress relaxation is time-dependent, even after three hours. The longer the observation time, the smaller the calculated yield stress. For a lubricated plunger, the residual force is reduced at a constant rate; however, the residual force does not follow a specific pattern with the an unlubricated plunger. For example, a higher yield stress could be calculated from the data (Fig. 15) when determined at 180 minutes than determined at 160 minutes after plunger was stopped

The average yield stress measured using a lubricated plunger seems higher than those found using an unlubricated plunger but the differences are not statistically significant (Table 8). Thus, this experiment shows that lubricated plunger (with K=0.52) increases uniformity, but does not alter the determined yield stress.

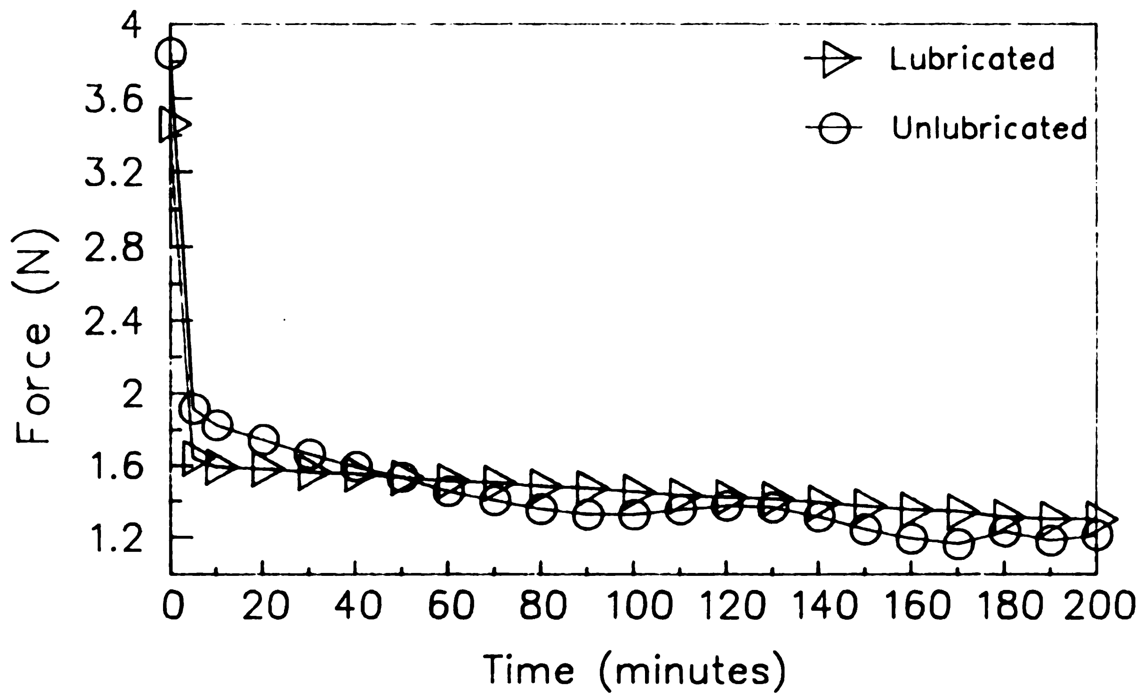

Figure 15. Stress relaxation curve of tomato paste in back extrusion using a lubricated and an unlubricated plunger with K=0.52.

Table 8. Yield stress of tomato paste measured using lubricated and unlubricated plungers with K=0.52.

Plunger surface	σ_0 (Pa) 1 hour	σ_0 (Pa) 2 hours	σ_0 (Pa) 3 hours
Lubricated	86.0	65.6	56.3
	96.2	92.7	79.0
	100.7	96.3	83.4
Average	94.3	84.8	72.9
Std. dev.	3.2	5.8	5.3
Unlubricated	86.9	112.7	87.5
	105.0	70.3	48.4
	76.6	55.6	34.6
Average	89.5	79.5	56.8
Std. dev.	14.2	12.1	11.2

5.2 Comparison of Back Extrusion to Other Methods

5.2.1 Comparison to Gun Rheometer Data

The gun rheometer was used for comparison because it is a reliable device, not relying on a spring like the rotary viscometer method and able to measure any type of fluid.

Table 9 shows the results of yield stress measurement using back extrusion and gun rheometry for peanut butter, tomato paste and chicken batter. Before comparing the two methods, it is important to explain why the gun rheometer failed to measure the yield stress of the chicken batter. As described in a previous section, the way the gun rheometer measures yield stress is to determine a minimum pressure required to initiate flow. In the case of chicken batter, the sample did not flow, but simply slid out of the sample tube. A combination of high yield stress and high fat content may have caused this problem. The minimum pressure needed to create sliding varied from 3.5 kPa to 30 kPa for the same 20 cm sample length. It is impossible to accept those values for yield stress calculations.

The yield stress of peanut butter obtained from the gun rheometer is higher than that determined from back extrusion after three hours relaxation time. In contrast, the yield stress of tomato paste is lower. There are many possible reasons why those values are not comparable.

Table 9. Yield stress of peanut butter, tomato paste and chicken batter measured using gun rheometer and back extrusion (lubricated plunger and K=0.52).

Method	σ_0 (Pa) 1 hour	σ_0 (Pa) 2 hours	σ_0 (Pa) 3 hours
Back extrusion			
Peanut butter	103.2 124.6 136.7	45.2 66.8 68.8	21.7 29.7 30.0
Average Std. dev.	121.5 17.0	80.2 13.1	27.2 4.7
Chicken batter	177.5 190.8 188.8	139.9 144.3 138.2	119.2 119.5 115.0
Average Std. dev.	185.7 7.2	140.8 3.1	117.9 2.5
Tomato paste	94.6 105.4 110.1	73.0 101.6 105.4	63.2 87.2 91.8
Average Std. dev.	103.4 7.9	93.3 17.7	80.7 15.4
Gun rheometer	Yield stress	Coeff. corr.	
Peanut butter	40.2	0.703	
Tomato paste	30.6	0.984	
Chicken batter	failed	failed	

First, the relaxation for samples in back extrusion may require a longer time than three hours, as seen in Fig. 18. It is true, for all three samples, that their curves of reducing force have not reached equilibrium states after three hours relaxation time. The curve for chicken frankfurter batter and tomato paste exhibit constant decreasing force with time. But the curve of peanut butter reduces with an inconsistent pattern. The pattern is similar to the curve of tomato paste (Fig. 11) when it measured in the narrower annular gap (K=0.78), but in the wider gap the erratic curve disappears and a higher yield stress is obtained after three hours relaxation. In conclusion, peanut butter may need a wider annular gap, greater than K=0.52, to obtain the best curve.

It may be true that each fluid measured using back extrusion needs a specific condition to obtain a true yield stress. For example, measuring the yield stress of Mixed Cereal can be satisfactory accomplished with K=0.67 and a relaxation time less than one hour. However, chicken frankfurter batter, tomato paste and peanut butter require a relaxation time longer than three hours. Also a K value less than 0.52 is needed for peanut butter to achieve optimum results.

In the gun rheometer method, the difficult aspect of measuring yield stress is to determine the minimum pressure (to cause flow) due to the fact that the fluid may not flow but only slide out (debond from solid surface). A low coefficient for correlation of 0.703 for peanut butter, when the data of pressure drop were extrapolated to zero velocity with linear regression method, may be caused by slip (Fig. 16 and 17). To prevent slip, Goodrich et al. (1989) placed internal "fins" in the sample tube when measured yield stress of gels. The same technique may be applied to the gun rheometer to eliminate the slip.

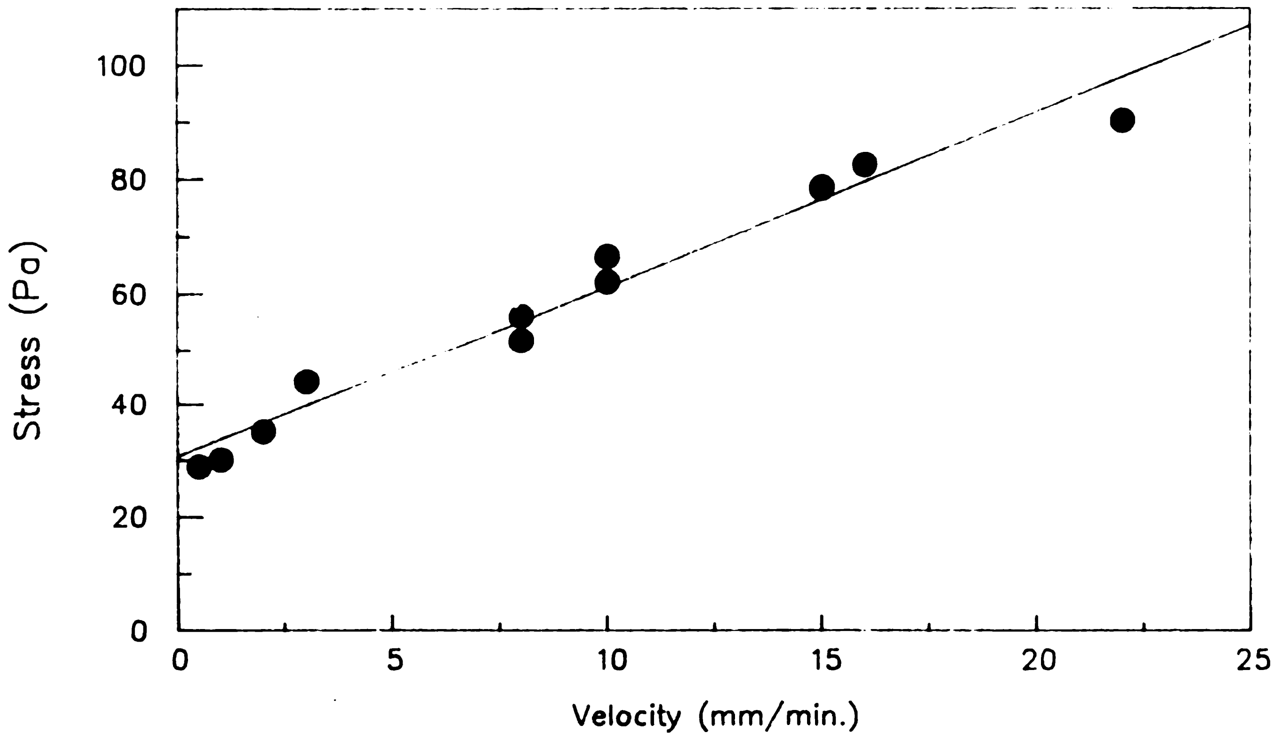

Figure 16. Plot of gun rheometer data for tomato paste.

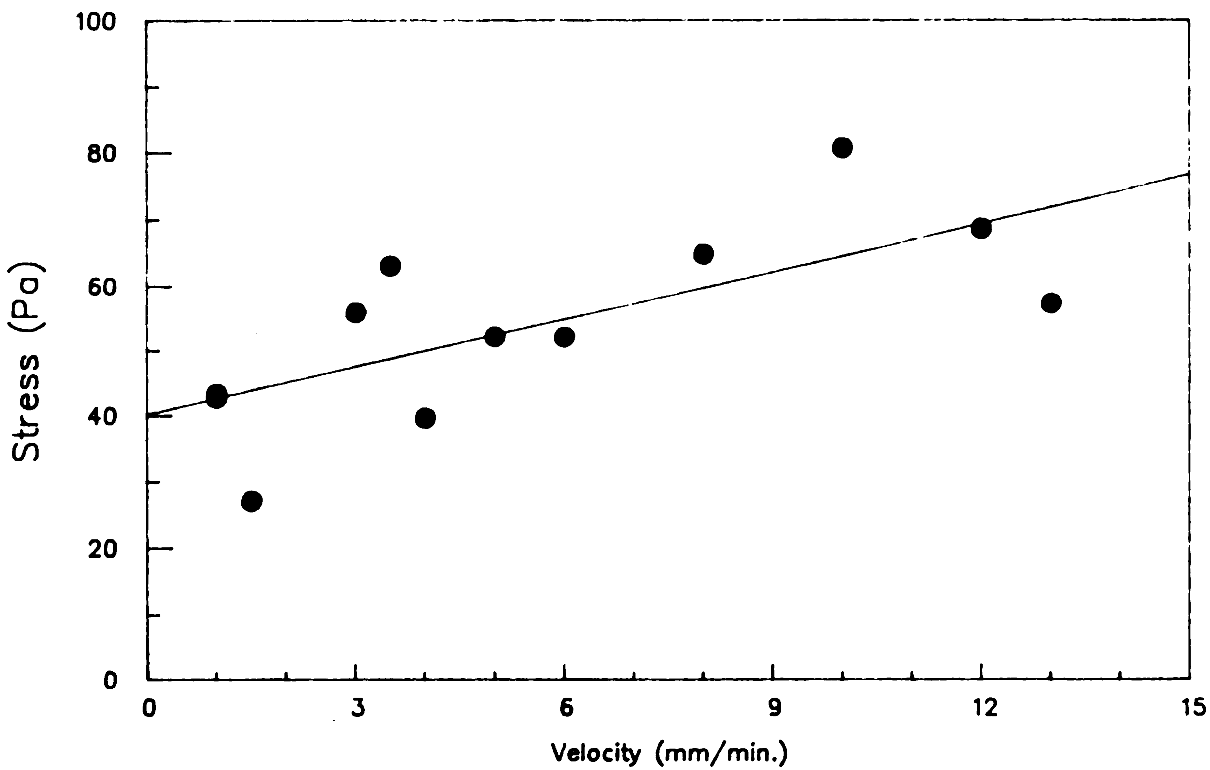

Figure 17. Plot of gun rheometer data for peanut butter.

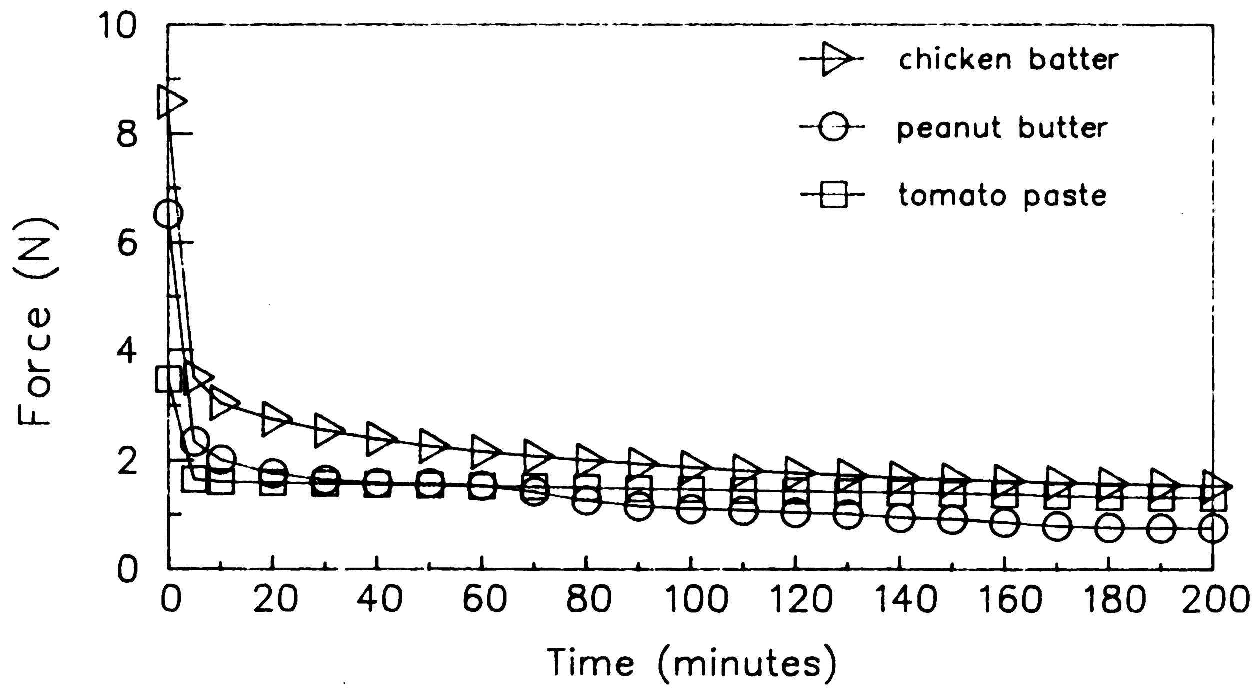

Figure 18. Stress relaxation curve of chicken batter, peanut butter and tomato paste using a lubricated aluminum plunger with K=0.52.

5.2.2 Comparison to Vane Device Data

It was very difficult to compare back extrusion results to the results of the vane method because of the high yield stress samples. A popular method, that is believed by some researcher as the most reliable method, the vane device could not be applied to peanut butter, tomato paste and chicken frankfurter batter using the HBTD Brookfield viscometer due to the yield stress of the samples being too high for the torque capability of the viscometer. Since back extrusion has capability of measuring yield stress of any type of fluid, from fluid like to solid like, Mixed Cereal baby food was used as a sample in comparing the vane rheometer to back extrusion method.

Mixed Cereal from Gerber was measured using three methods: back extrusion, vane device and gun rheometer. The results of these methods can be seen at Table 10. Yield stresses obtained by the vane device have small standard deviations, but depend upon the rotational velocity of the vane. Although Dzuy and Boger (1983) found that rotational velocity from 0.1 rpm to 8 rpm produced consistent results when they measured 66 percent of Red mud, yield stresses of mixed cereal measured using 0.5 to 2.5 rpm gave different results.

The theory of the vane device described by Dzuy and Boger (1983) requires a very low rotation of the vane. Unfortunately, the Brookfield viscometer used in this experiment did not have a rotational speed lower than 0.5 rpm. The effect of rotational speeds was not expected for high yield stress materials (Dzuy and Boger, 1983), but for low yield stress materials, the viscous resistance together with instrument inertia as described by Dzuy and Boger (1985) has already occured at 0.5 rpm vane speeds. If the value of yield stress at zero rpm was obtained using the extrapolation method, the yield stress of mixed cereal measured by vane device was 12.58 Pa with correlation coefficient is 0.978.

This magnitude is still higher when compares to the result of that measured by either the gun rheometer or the back extruder. These results agreed with previous experiments indicating that stress relaxation methods give results lower than those of the stress to initiate flow methods (Dzuy and Boger, 1985).

Back extrusion with a lubricated plunger has a higher yield stress than that found with an unlubricated plunger even though is not statistically significant at 0.05 confidence level. This clearly shows, once again, that the lubricated plunger will produce more uniform result than the unlubricated plunger.

The gun rheometer has the lowest yield stress value: 4.47 Pa. The distinct results among three methods make it difficult to conclude which value of them is the actual yield stress. The results of the vane device used in this experiment are not perfect due to the lack of low rotational speed even though the data show small standard deviations. The result of the gun rheometer is also obtained from extrapolation to zero velocity with correlation coefficient is 0.903. The back extrusion method also has a weakness because the fluids may have a time-dependent yield stress when measured using stress relaxation methods.

Table 10. Yield stress of Mixed cereal measured with back extrusion, vane device and gun rheometer.

Methods	Specific treatment	σ_0 (Pa)	Std. dev. (Pa)
Vane device	2.5 rpm	22.9	0.3
	1.0 rpm	17.6	0.4
	0.5 rpm	13.8	0.8
Back extrusion	Lubricated	7.8	1.1
(K=0.52)	Unlubricated	4.4	1.7
Gun rheometer		4.5	r=0.903

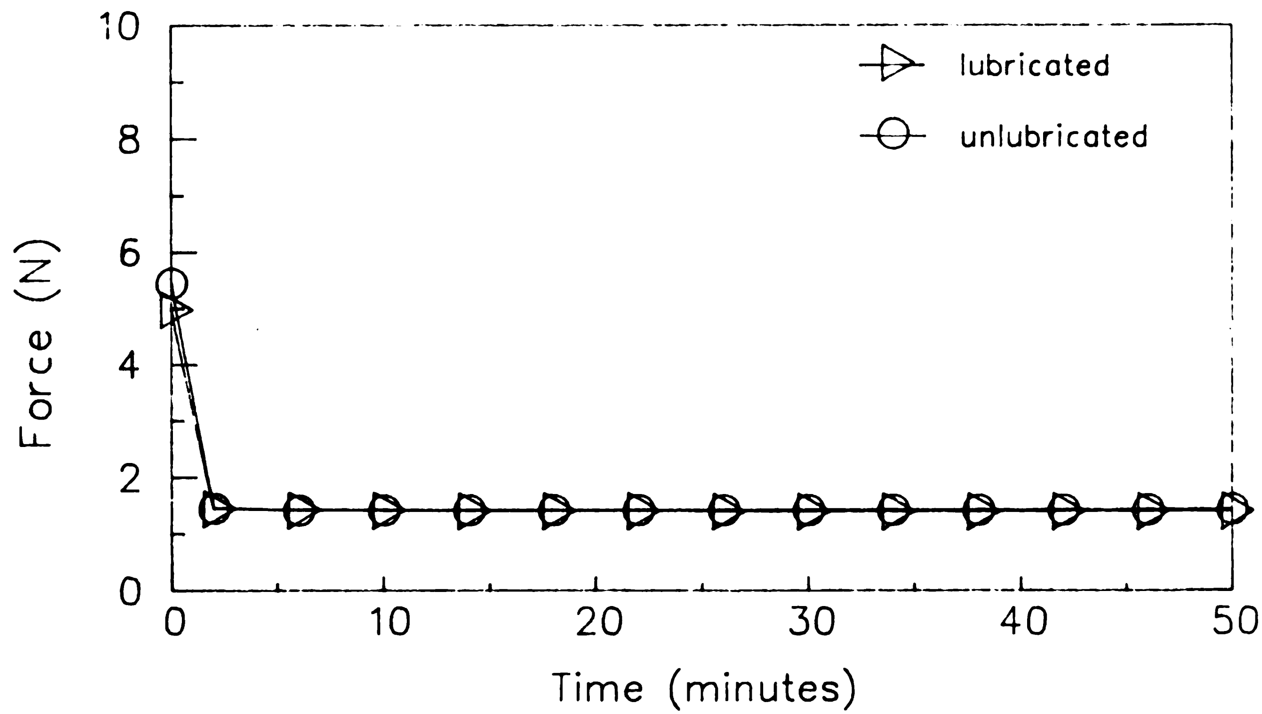

Figure 19. Stress relaxation curve of mixed cereal in back extrusion using a lubricated and an unlubricated plunger.

Chapter 6

Conclusion

The experiments showed that the back extrusion method can be used to measure yield stress of semi-solid foods. However, some specific conditions may be required to obtain reproducible and reliable values. The conditions are related to selection of plunger surface, annular gap and observation time.

Back extrusion using grooved and unlubricated smooth plungers revealed that the assumption of no slip on the annular wall for measuring other rheological parameters will be a critical factor which result in non-reproducible values in yield stress measurement.

A lubricated plunger applied in yield stress measurement to avoid an uncontrollable friction factor produce more consistent yield stress value than using an unlubricated plunger.

In principle, a wide annular gap is needed to measure yield stress. Every fluid has a wide limit for producing uniform curve, below that limit the curve will be inconsistent K=0.52 for chicken frankfurter batter and tomato paste, and smaller K is needed for peanut butter).

Each fluid also needs a different relaxation time to reach an equilibrium state. Some fluid may achieve an equilibrium state within one hour (Mixed Cereal baby food), but other fluid may need more than three hours observation time. Otherwise the measured yield stress is still time-dependent (chicken frankfurter batter, tomato paste and peanut butter).

Yield stress measurement should be established separately from measuring other rheological parameters in the case of back extrusion testing. The value of yield stress obtained using a stress relaxation method, such as back extrusion, is lower than the vane device method. The true value of yield stress can not be determined during this study due to equipment difficulties and material time-dependency.

Chapter 7

Future Research

A major disadvantage found in measuring the yield stress of semi-solid foods using back extrusion was a very long observation time. The equilibrium state was not reached even over three hours of observation and the yield stress determined during that observation time was still time-dependent. Furthermore, to keep the sample from environmental effects was very difficult and dehydration or temperature changes may be a problem. Therefore, future research is needed to find a method of estimating the yield stress from shorter observation times which minimize environmental effects.

The stress relaxation method produced different yield stress values compared to the stress to initiate flow methods. It is important to observe, in more detail, the behavior of the fluid during stress relaxation. However, the yield stress determined with back extrusion may be preferable if compared to other stress relaxation methods, involving other instruments such as a concentric cylinder viscometer.

The experimental evaluation of the strain history effect is also important because the negative value of yield stress encountered in testing with K=0.78 (after three hours observation) may be caused by a high preceding shear rate rather than narrow solid boundaries.

Chapter 8

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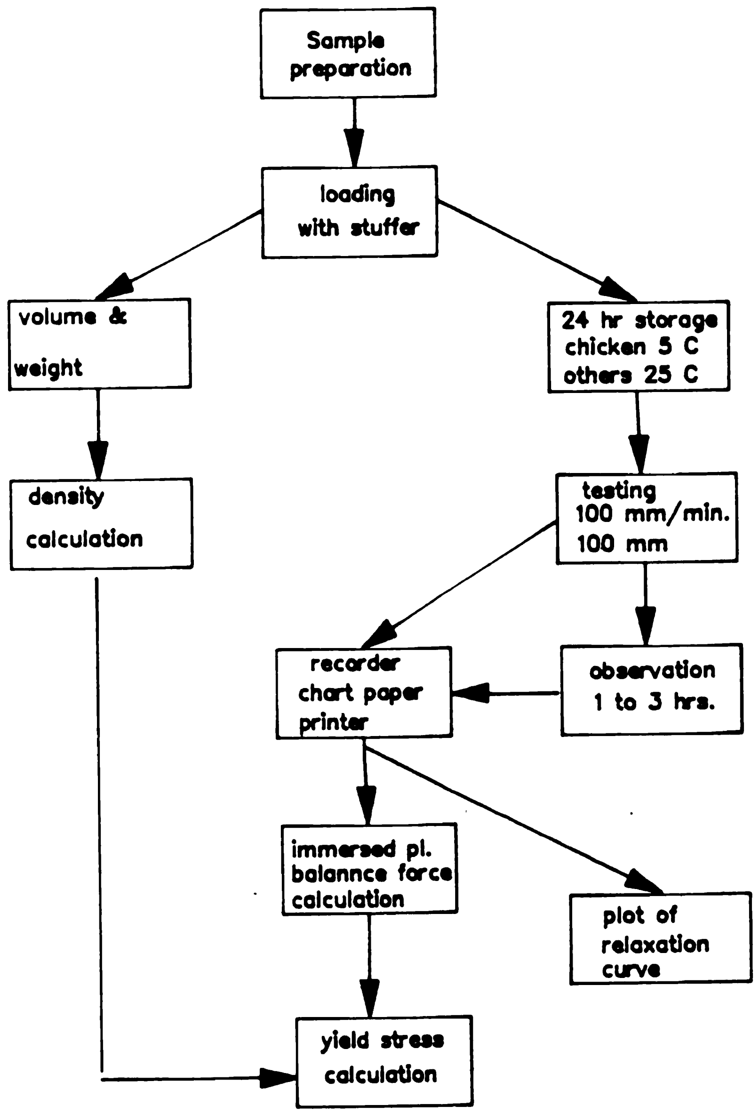
Appendix A

Schematic diagram of back extrusion experiments



Appendix B

Results of yield stress measurement using back extrusion method.

Table B1. Data of chicken batter obtained from back extrusion testing using plunger radius of 0.0142 m, graduate cylinder radius of 0.0178 m and plunger speed of 100 mm/min.

Plunger	F_T (N)	F_{T_e} (N)	L_{ch} (m)	F_b (N)	L (m)	σ_0 (Pa)
Plexiglass						
smooth	35.69	10.49	0.116	1.1	0.17	615.3
	32.52	8.98	0.118	1.1	0.17	506.8
	34.97	9.14	0.112	1.1	0.16	548.4
grooved	40.74	11.15	0.111	1.0	0.16	691.4
	44.39	13.34	0.118	1.1	0.17	787.0
	36.97	11.99	0.109	1.0	0.16	763.9
Teflon						
smooth	36.75	12.28	0.116	1.1	0.17	732.4
	41.46	12.90	0.112	1.1	0.16	803.2
	37.40	11.27	0.105	1.0	0.15	743.6
grooved	34.91	11.93	0.113	1.1	0.17	730.3
	40.28	11.97	0.107	1.0	0.16	778.1
	36.75	10.63	0.106	1.0	0.15	690.1
Aluminum						
smooth	30.66	11.53	0.113	1.1	0.17	703.4
	37.93	9.79	0.112	1.1	0.16	593.0
	35.93	7.19	0.112	1.1	0.16	416.1
grooved	40.63	14.44	0.108	1.0	0.16	943.8
	45.05	14.11	0.117	1.1	0.17	844.3
	45.26	11.96	0.112	1.1	0.16	739.5

Table B2. Data of peanut butter obtained from back extrusion testing using plunger radius of 0.0142 m, graduate cylinder radius of 0.0178 m and plunger speed of 100 mm/min.

Plunger	F_T (N)	F_{T_e} (N)	L_{ch} (m)	F_b (N)	L (m)	σ_0 (Pa)
Plexiglass						
smooth	36.51	1.98	0.102	1.0	0.15	66.8
	44.54	2.04	0.104	1.1	0.15	71.4
	38.31	1.38	0.103	1.0	0.15	24.5
grooved	45.02	3.10	0.102	1.0	0.15	153.5
	45.14	2.70	0.085	0.9	0.13	161.2
	45.15	2.55	0.059	0.6	0.09	250.5
Teflon						
smooth	45.11	0.81	0.102	1.0	0.15	-17.3
	45.05	1.11	0.071	0.7	0.10	43.4
	45.10	3.79	0.092	0.9	0.13	235.4
grooved	33.14	2.87	0.103	1.0	0.15	134.1
	45.02	4.29	0.100	1.0	0.15	248.4
	41.56	1.14	0.105	1.1	0.15	5.2
Aluminum						
smooth	44.58	1.82	0.103	1.0	0.15	56.8
	45.01	2.61	0.086	0.9	0.13	153.0
	45.15	2.64	0.102	1.0	0.15	118.8
grooved	33.81	3.74	0.104	1.6	0.15	195.4
	36.83	1.65	0.091	0.9	0.13	60.2
	33.25	2.71	0.090	0.9	0.13	150.9

Table B3. Data of tomato paste obtained from back extrusion testing using plunger radius of 0.0142 m, graduate cylinder radius of 0.0178 m and plunger speed of 100 mm/min.

Plunger	F_T (N)	F_{T_e} (N)	L_{ch} (m)	F_b (N)	L (m)	σ_0 (Pa)
Plexiglass						
smooth	19.23	1.18	0.112	1.2	0.16	-1.1
	21.32	4.84	0.139	1.5	0.20	183.1
	21.54	5.85	0.143	1.5	0.21	229.3
grooved	18.45	3.69	0.106	1.3	0.15	183.7
	19.15	5.93	0.130	1.4	0.19	265.0
	20.85	4.50	0.140	1.5	0.20	163.0
Teflon						
smooth	19.76	1.89	0.113	1.2	0.17	45.8
	16.89	4.50	0.110	1.2	0.16	229.6
	18.96	6.62	0.123	1.3	0.18	327.7
grooved	20.17	3.03	0.107	1.1	0.16	133.7
	16.56	3.94	0.132	1.4	0.19	145.7
	16.03	3.02	0.129	1.4	0.19	96.4
Aluminum						
smooth	17.32	4.89	0.122	1.3	0.18	224.1
	21.35	3.26	0.139	1.5	0.20	98.1
	26.17	6.75	0.165	1.7	0.24	230.7
grooved	20.88	1.99	0.124	1.3	0.18	40.8
	20.26	5.15	0.130	1.4	0.19	219.6
	23.05	2.57	0.133	1.4	0.19	65.5

Table B4. Data obtained from back extrusion testing using a lubricated aluminum plunger with radius of 0.0142 m, graduate cylinder radius of 0.0178 m and plunger speed of 100 mm/min.

Plunger	F_T (N)	F_{T_e} (N)	L_{ch} (m)	F_b (N)	L (m)	σ_0 (Pa)
Chicken batter						
	23.99	6.88	0.110	1.1	0.16	403.8
	24.30	5.57	0.112	1.1	0.16	306.5
	21.97	5.40	0.109	1.0	0.16	304.9
Peanut butter						
	33.81	1.90	0.105	1.1	0.15	60.2
	36.83	1.81	0.102	1.0	0.15	57.2
	33.25	2.00	0.103	1.0	0.15	70.3
Tomato paste						
	15.48	3.51	0.118	1.2	0.17	145.5
	19.44	4.04	0.132	1.4	0.19	152.0
	24.81	5.21	0.155	1.6	0.23	175.1

Table B5. Data of tomato paste obtained from back extrusion testing using a lubricated aluminum plunger with different ratios of plunger to graduate cylinder diameter (Vp = 100 mm/min.)

Plunger	F_T (N)	F_{T_e} (N)	L_{ch} (m)	F_b (N)	L (m)	σ_0 (Pa)
1 hour						
K = 0.78	9.04	1.58	0.120	0.7	0.20	68.9
(R_o = 0.0128 m)	9.53	1.71	0.119	0.7	0.20	81.0
(R_i = 0.01 m)	10.36	0.86	0.114	0.7	0.19	15.2
K = 0.672	6.23	1.68	0.128	0.8	0.15	76.8
(R_o = 0.0178 m)	6.84	1.93	0.120	0.7	0.14	109.3
(R_i = 0.012 m)	6.87	2.08	0.125	0.8	0.15	115.6
K = 0.52	3.37	1.44	0.131	0.6	0.12	94.7
(R_o = 0.0232 m)	3.55	1.54	0.131	0.6	0.12	105.4
(R_i = 0.012 m)	3.45	1.55	0.129	0.6	0.12	110.4
2 hours						
K = 0.78	9.04	1.38	0.120	0.7	0.20	53.5
	9.53	0.85	0.119	0.7	0.20	11.8
	10.36	0.66	0.114	0.7	0.19	-1.5
K = 0.67	6.23	1.54	0.128	0.8	0.15	64.3
	6.84	1.55	0.120	0.7	0.14	74.1
	6.87	1.85	0.125	0.8	0.15	95.3
K = 0.52	3.37	1.25	0.131	0.6	0.12	74.0
	3.55	1.50	0.131	0.6	0.12	101.6
	3.45	1.51	0.129	0.6	0.12	105.4
3 hours						
K = 0.78	9.04	0.56	0.120	0.7	0.20	-11.7
	9.53	0.56	0.119	0.7	0.20	-11.7
	10.36	0.57	0.114	0.7	0.19	-8.9
K = 0.67	6.23	1.33	0.128	0.8	0.15	46.7
	6.84	1.21	0.120	0.7	0.14	43.4
	6.87	1.68	0.125	0.8	0.15	80.3
K = 0.52	3.37	1.16	0.131	0.6	0.12	63.2
	3.55	1.38	0.131	0.6	0.12	87.2
	3.45	1.40	0.129	0.6	0.12	91.8

Table B6. Data of chicken batter obtained from back extrusion testing using a lubricated aluminum plunger with different plunger speeds (K = 0.52).

Plunger	F_T (N)	F_{T_e} (N)	L_{ch} (m)	F_b (N)	L (m)	σ_0 (Pa)
1 hour						
50 mm/min.	8.36	2.20	0.137	0.6	0.12	177.5
	8.75	2.15	0.127	0.5	0.11	190.8
	8.66	2.10	0.125	0.5	0.11	188.8
100 mm/min.	9.70	1.94	0.062	0.5	0.11	171.0
	9.90	2.20	0.062	0.5	0.11	202.7
	10.45	2.03	0.062	0.5	0.11	178.5
2 hours						
50 mm/min.	8.36	1.85	0.137	0.6	0.12	140.0
	8.75	1.75	0.127	0.5	0.11	144.3
	8.66	1.67	0.125	0.5	0.11	138.2
100 mm/min.	9.70	1.67	0.062	0.5	0.11	139.2
	9.90	1.77	0.062	0.5	0.11	150.9
	10.45	1.65	0.062	0.5	0.11	133.5
3 hours						
50 mm/min.	8.36	1.66	0.137	0.6	0.12	119.2
	8.75	1.54	0.127	0.5	0.11	119.5
	8.66	1.48	0.125	0.5	0.11	115.0
100 mm/min.	9.70	1.51	0.062	0.5	0.11	119.8
	9.90	1.53	0.062	0.5	0.11	122.8
	10.45	1.46	0.062	0.5	0.11	111.2

Table B7. Data of tomato paste obtained from back extrusion testing using a lubricated and an unlubricated aluminum plunger with different the depth of penetrations (Vp = 100 mm/min. and K = 0.67).

L_{ch} (m)	F_T (N)	F_{T_e} (N)	F_b (N)	L (m)	σ_0 (Pa)
Lubricated					
0.075	11.73	2.43	0.8	0.11	166.3
0.088	12.58	2.69	0.9	0.13	152.3
0.118	15.48	3.51	1.2	0.17	145.5
0.132	19.44	4.04	1.4	0.19	152.0
0.155	24.81	5.21	1.6	0.23	175.1
0.184	25.46	5.58	1.9	0.27	149.2
Unlubricated					
0.088	14.41	2.79	0.9	0.13	160.7
0.104	16.70	1.80	1.1	0.15	51.1
0.122	17.32	4.89	1.3	0.18	224.1
0.139	21.35	3.26	1.5	0.20	98.1
0.165	26.17	6.75	1.7	0.24	230.7
0.180	29.92	6.88	1.9	0.26	210.2

Table B8. Data of tomato paste obtained from back extrusion testing using a unlubricated aluminum plunger (Vp = 100 mm/min. and K = 0.52).

Plunger	F_T (N)	F_{T_e} (N)	L_{ch} (m)	F_b (N)	L (m)	σ_0 (Pa)
1 hour	4.30	1.49	0.135	0.6	0.12	95.5
	3.56	1.52	0.123	0.6	0.11	114.6
	3.65	1.33	0.129	0.6	0.12	84.7
2 hours	4.30	1.74	0.135	0.6	0.12	122.8
	3.56	1.22	0.123	0.6	0.11	78.0
	3.65	1.14	0.129	0.6	0.12	62.5
3 hours	4.30	1.50	0.135	0.6	0.12	96.2
	3.56	1.02	0.123	0.6	0.11	54.9
	3.65	0.95	0.129	0.6	0.12	40.3

Table B9. Data of chicken batter, peanut butter and tomato paste obtained from back extrusion testing using a lubricated aluminum plunger (Vp = 50 mm/min. and K = 0.52).

Plunger	F_T (N)	F_{T_e} (N)	L_{ch} (m)	F_b (N)	L (m)	σ_0 (Pa)
1 hour						
Chicken batter	8.36	2.20	0.137	0.6	0.12	177.5
	8.75	2.15	0.127	0.5	0.11	190.8
	8.66	2.10	0.125	0.5	0.11	188.8
Peanut butter	8.98	1.33	0.117	0.5	0.10	103.2
	4.47	1.46	0.114	0.5	0.10	124.6
	6.09	1.76	0.129	0.6	0.12	136.7
Tomato paste	3.38	1.44	0.131	0.6	0.12	94.6
	3.55	1.54	0.131	0.6	0.12	105.4
	3.46	1.56	0.129	0.6	0.12	110.1
2 hours						
Chicken batter	8.36	1.85	0.137	0.6	0.12	139.9
	8.75	1.75	0.127	0.5	0.11	144.3
	8.66	1.67	0.125	0.5	0.11	138.2
Peanut butter	8.98	0.90	0.117	0.5	0.10	48.9
	4.47	1.00	0.114	0.5	0.10	64.5
	6.09	1.17	0.129	0.6	0.12	68.8
Tomato paste	3.38	1.25	0.131	0.6	0.12	73.0
	3.55	1.50	0.131	0.6	0.12	101.6
	3.46	1.51	0.129	0.6	0.12	105.4
3 hours						
Chicken batter	8.36	1.66	0.137	0.6	0.12	119.2
	8.75	1.54	0.127	0.5	0.11	119.5
	8.66	1.48	0.125	0.5	0.11	115.0
Peanut butter	8.98	0.71	0.117	0.5	0.10	24.7
	4.47	0.72	0.114	0.5	0.10	28.1
	6.09	0.83	0.129	0.6	0.12	30.0
Tomato paste	3.38	1.16	0.131	0.6	0.12	63.2
	3.55	1.38	0.131	0.6	0.12	87.2
	3.46	1.20	0.129	0.6	0.12	91.8

Table B10. Data of mixed cereal baby food obtained from back extrusion testing using an aluminum plunger with radius of 0.0142 m, graduate cylinder radius of 0.0178 m and plunger speed of 100 mm/min.

Plunger	F_T (N)	F_{T_e} (N)	L_{ch} (m)	F_b (N)	L (m)	σ_0 (Pa)
Lubricated	5.31	1.42	0.126	1.3	0.18	8.6
	5.09	1.43	0.128	1.3	0.19	8.3
	4.50	1.33	0.121	1.2	0.18	6.6
Unlubricated	5.77	1.47	0.136	1.4	0.20	5.3
	4.99	1.42	0.131	1.3	0.19	5.5
	5.55	1.36	0.130	1.3	0.19	2.4

Appendix C
Stress relaxation curves

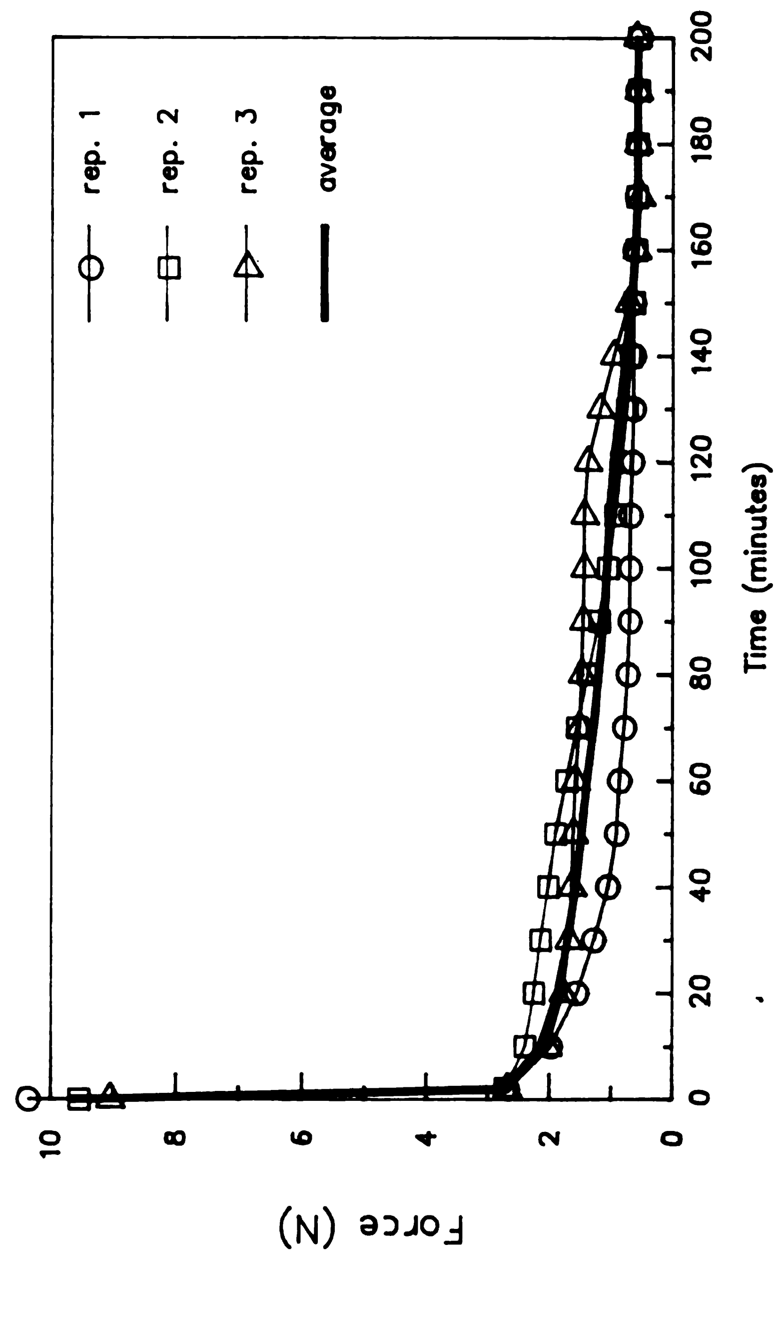


Figure C1. Stress relaxation curve of tomato paste in back extrusion using a aluminum lubricated plunger with K=0.78.

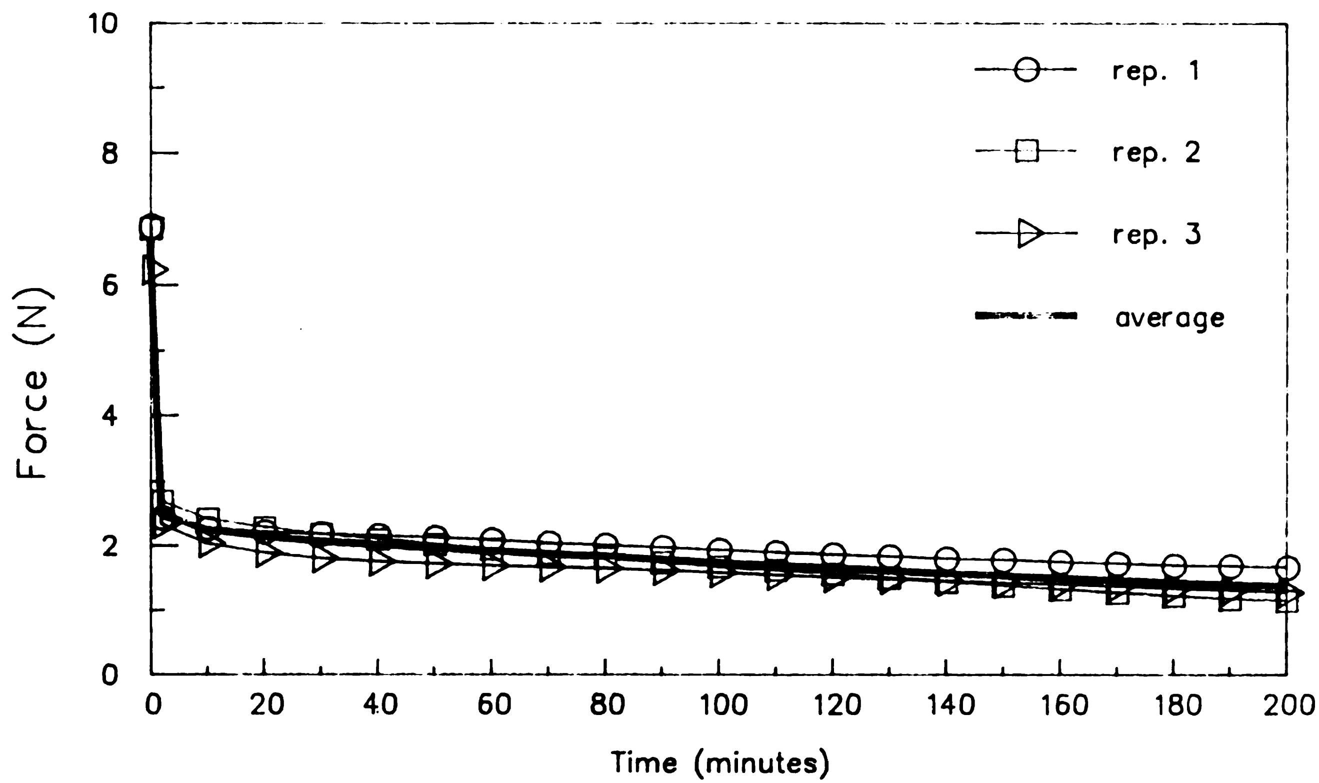


Figure C2. Stress relaxation curve of tomato paste in back extrusion using a aluminum lubricated plunger with K=0.67.

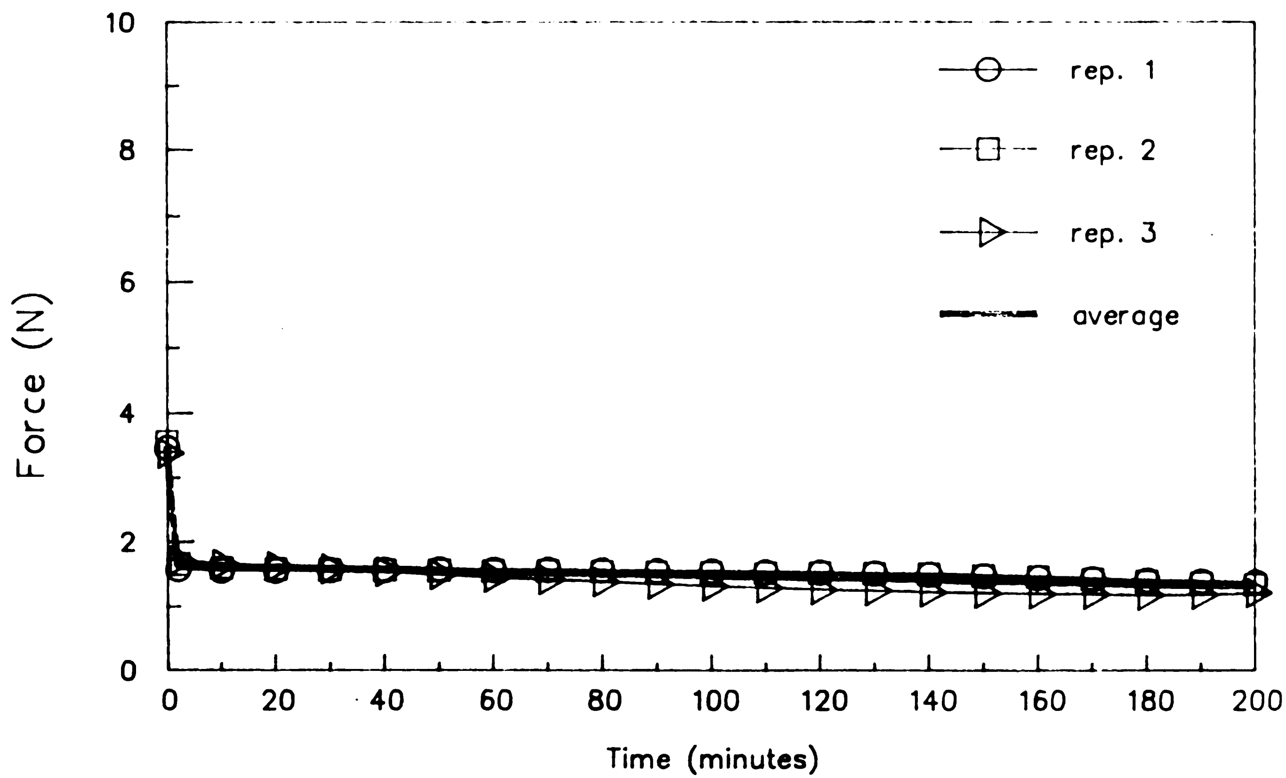


Figure C3. Stress relaxation curve of tomato paste in back extrusion using a aluminum lubricated plunger with K=0.52.

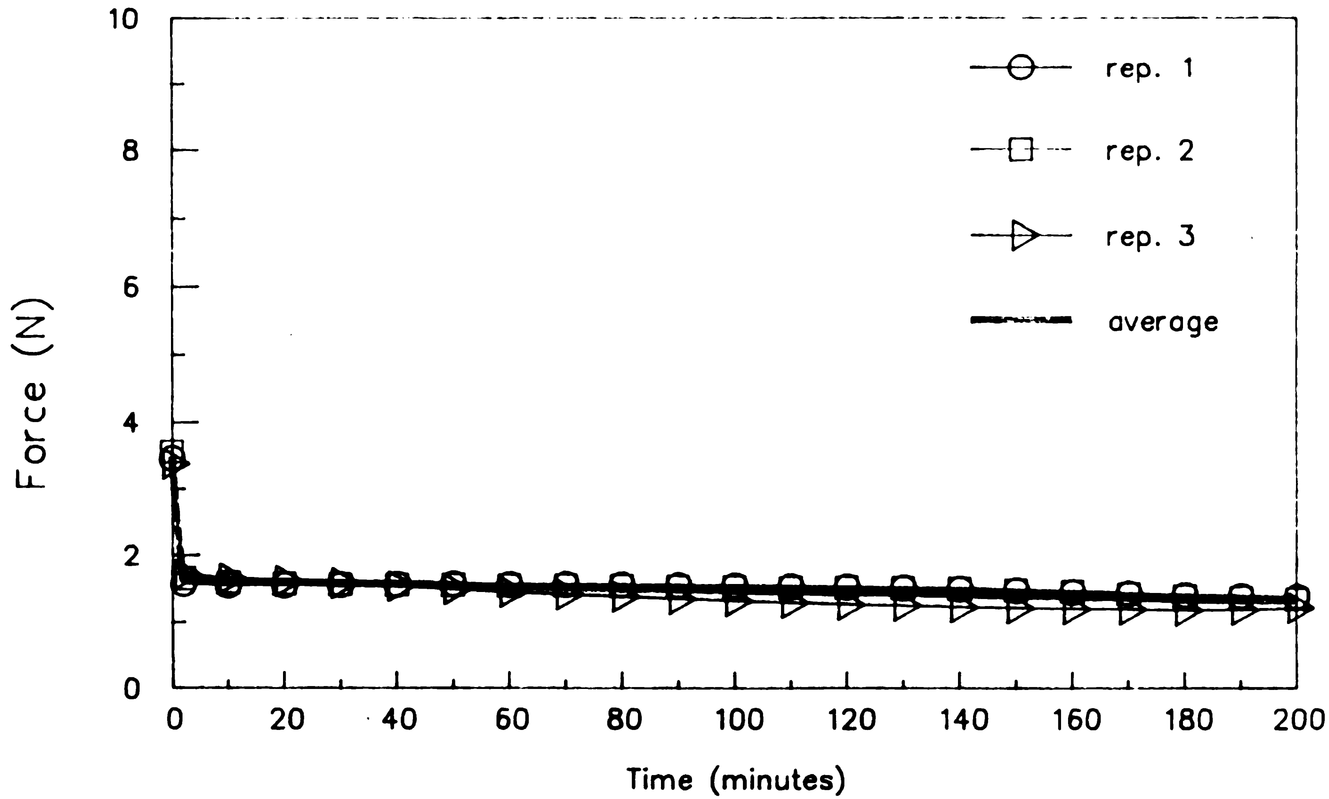


Figure C3. Stress relaxation curve of tomato paste in back extrusion using a aluminum lubricated plunger with K=0.52.

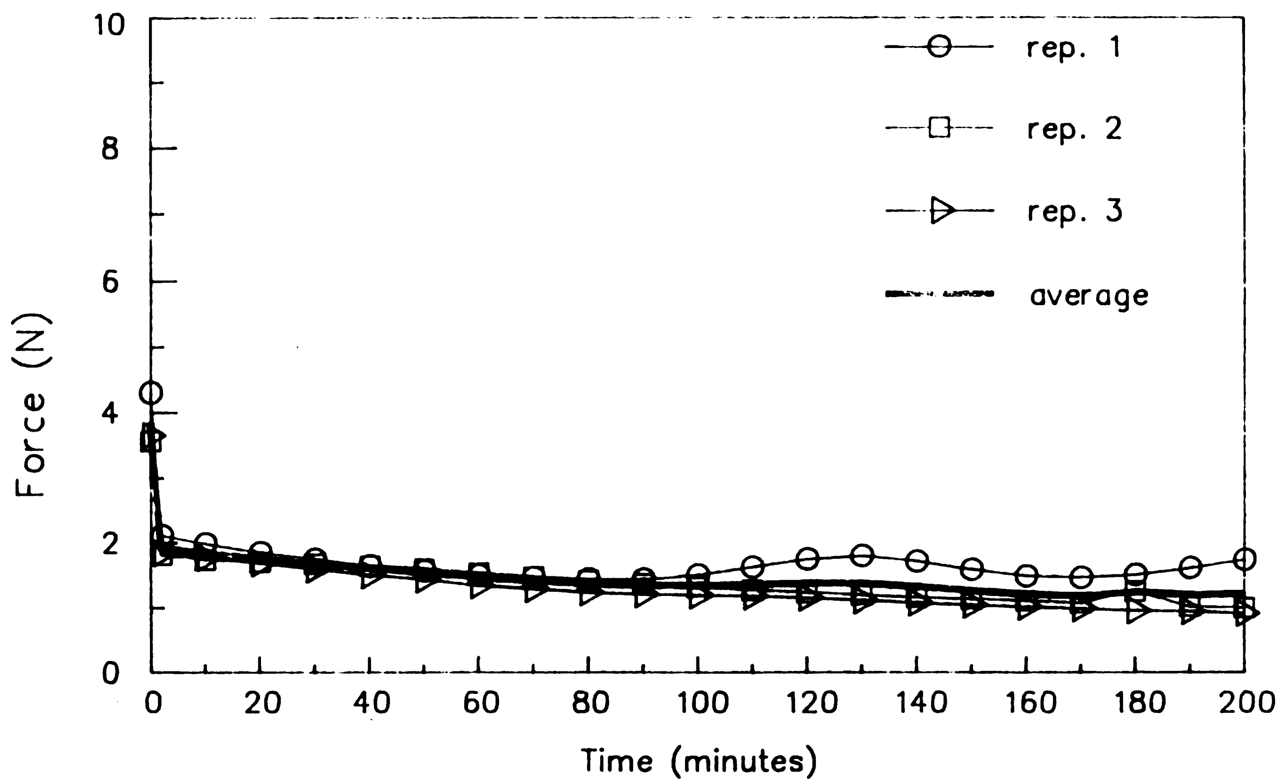


Figure C4. Stress relaxation curve of tomato paste in back extrusion using a aluminum unlubricated plunger with K=0.52.

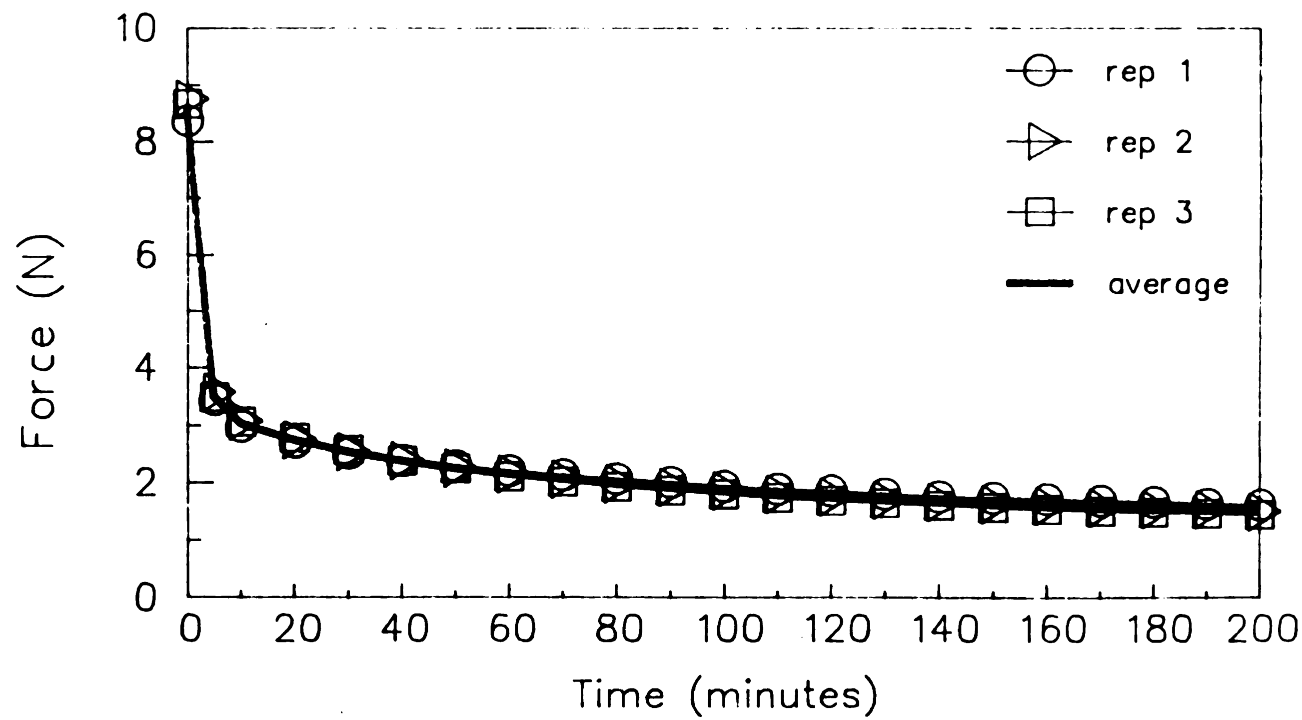


Figure C5. Stress relaxation curve of chicken batter in back extrusion using a lubricated aluminum plunger with 50 mm/min velocity.

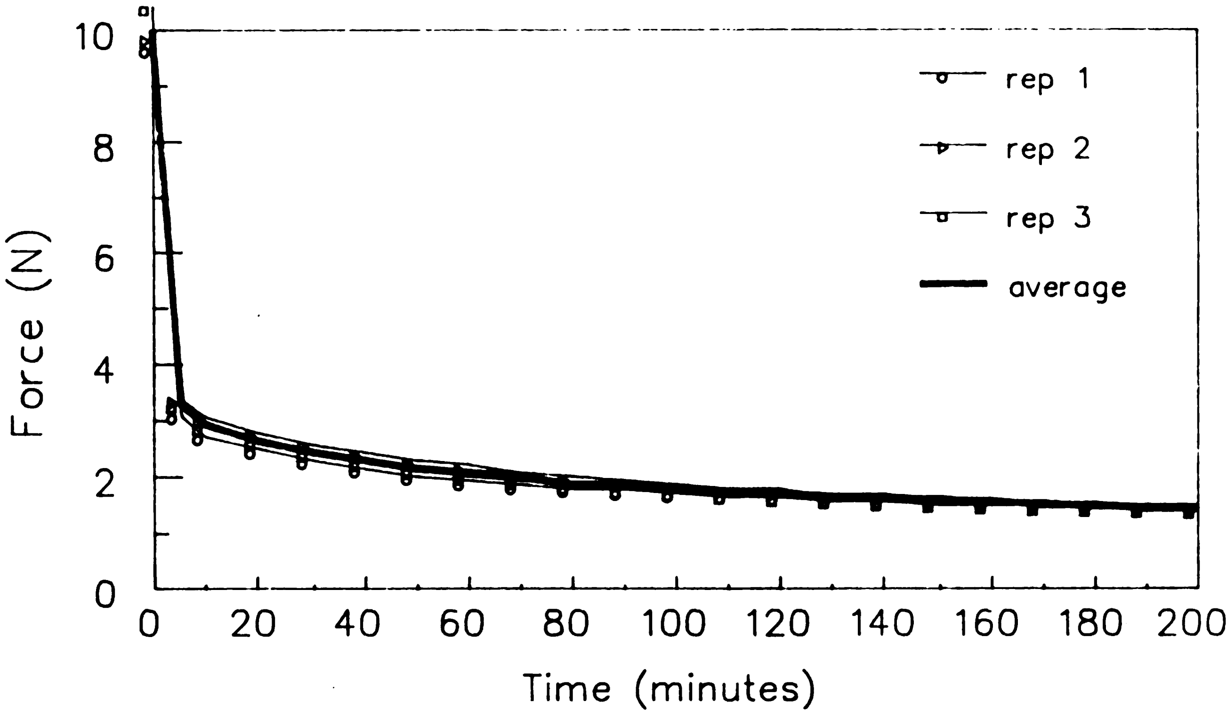


Figure C6. Stress relaxation curve of chicken batter in back extrusion using a lubricated aluminum plunger with 100 mm/min. velocity.

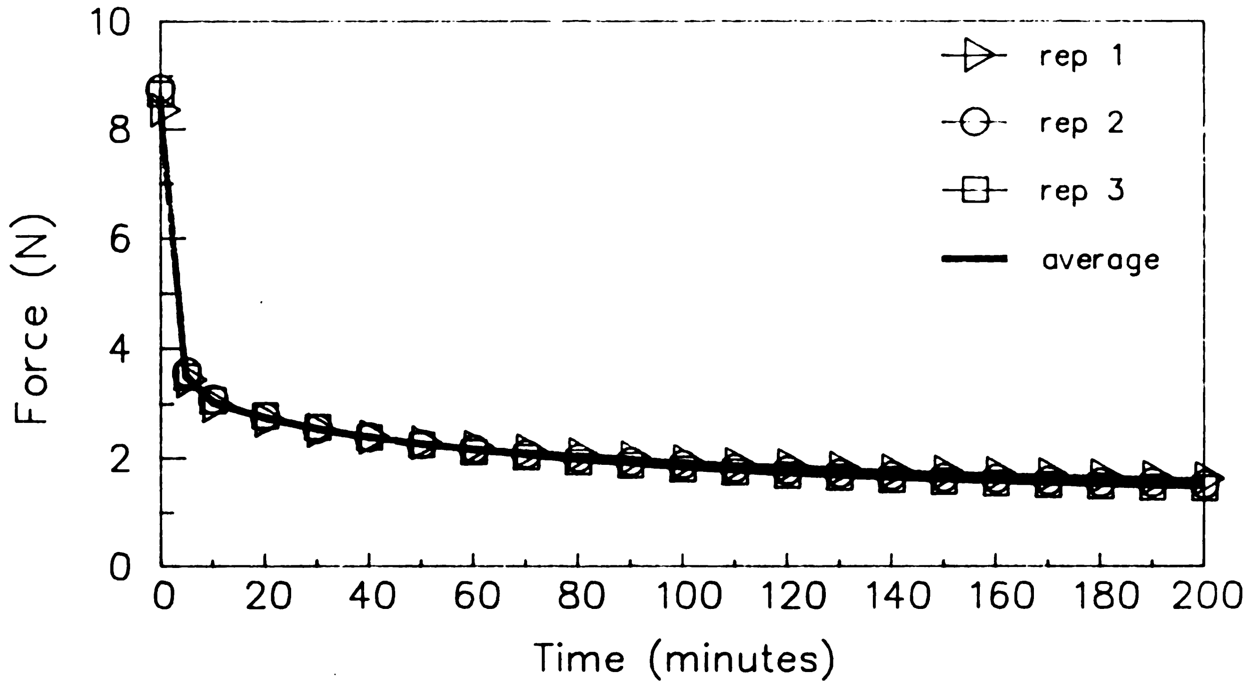


Figure C7. Stress relaxation curve of chicken batter in back extrusion using a lubricated aluminum plunger with K=0.52.

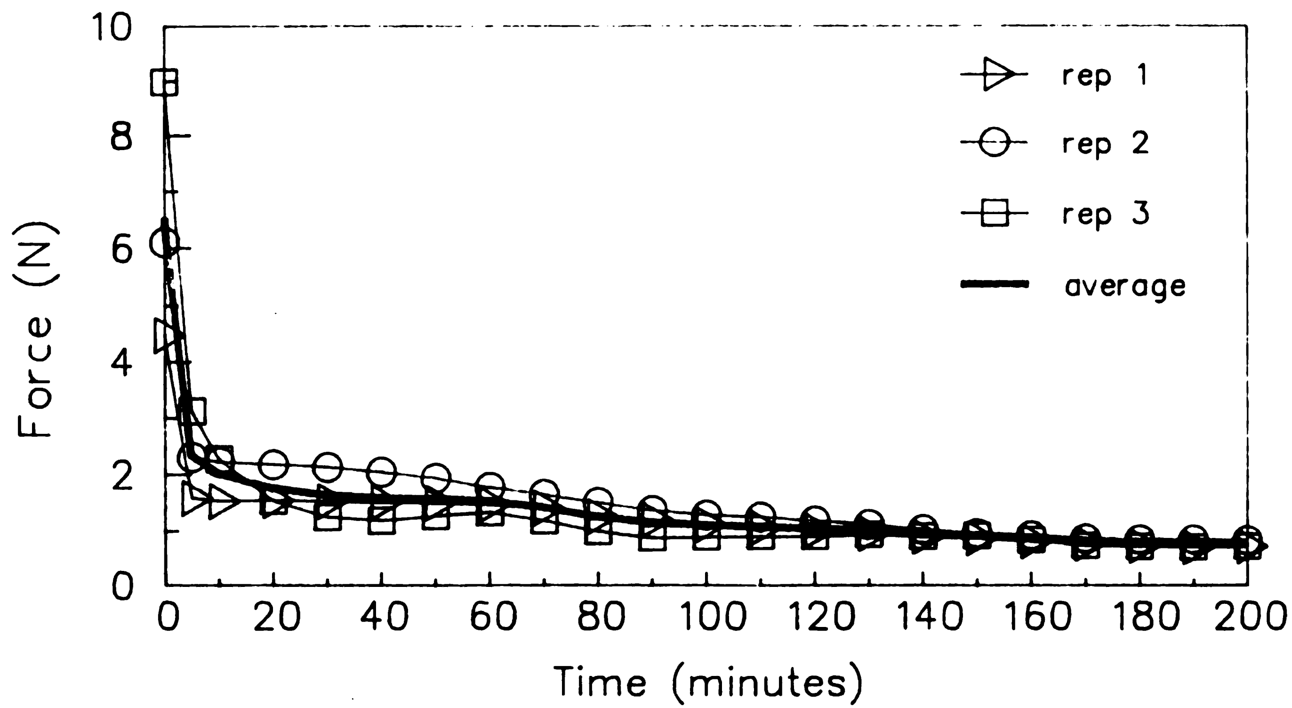


Figure C8. Stress relaxation curve of peanut butter in back extrusion using a lubricated aluminum plunger with K=0.52.

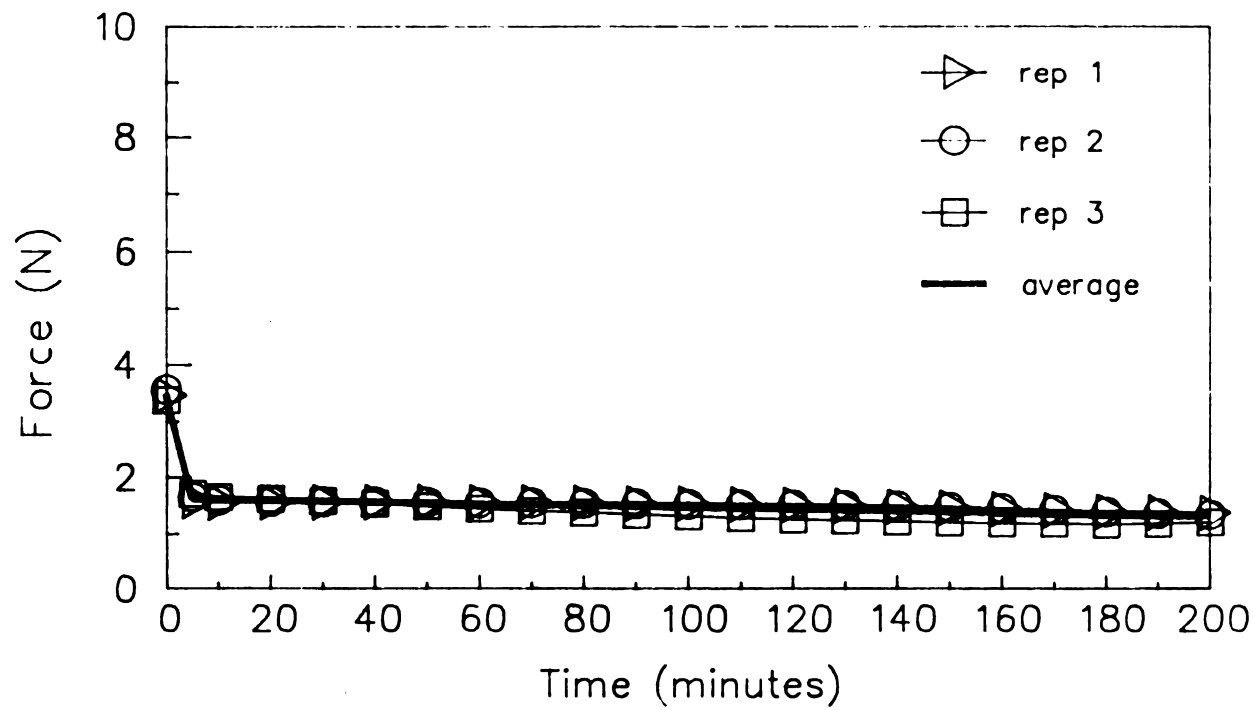


Figure C9. Stress relaxation curve of tomato paste in back extrusion using a lubricated aluminum plunger with K=0.52.

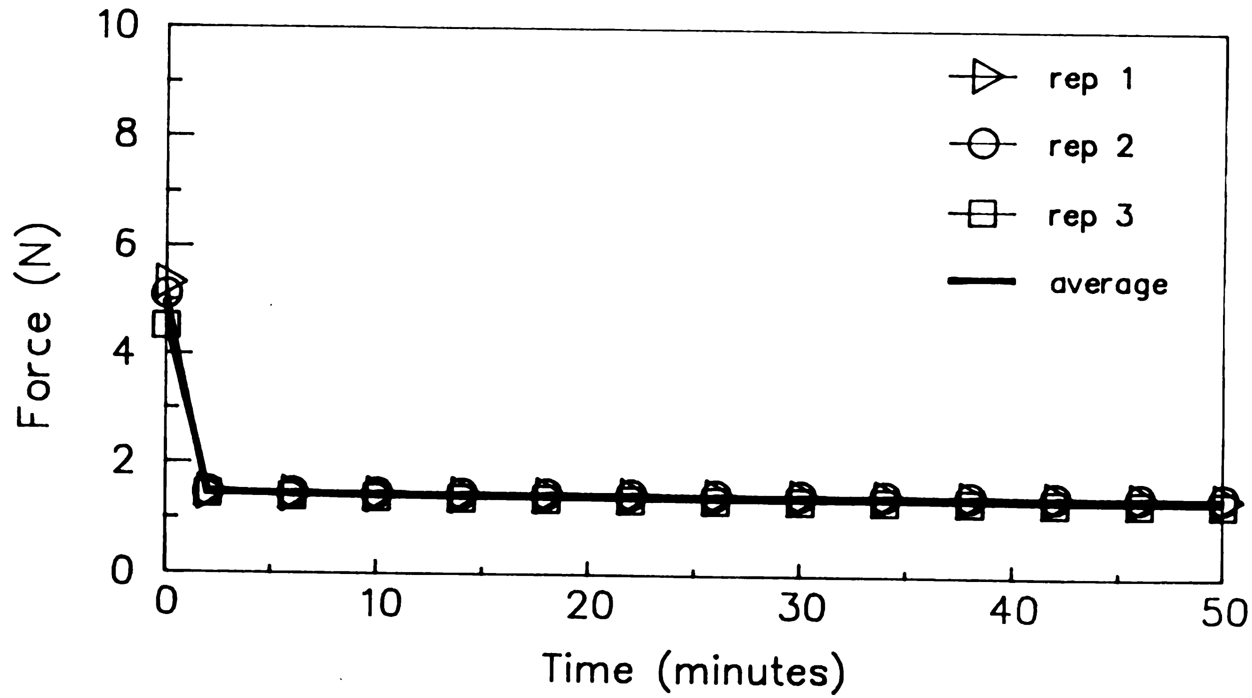


Figure C10. Stress relaxation curve of mixed cereal in back extrusion using a lubricated aluminum plunger with K=0.52.

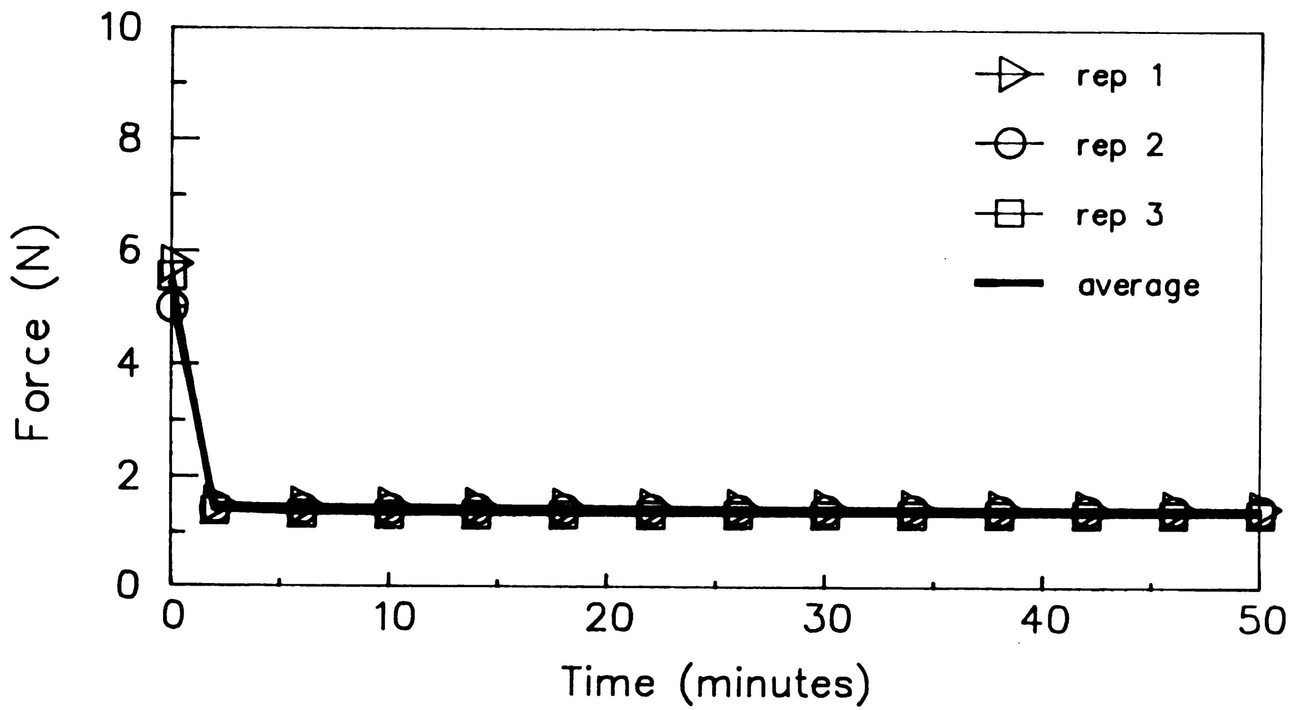


Figure C11. Stress relaxation curve of mixed cereal in back extrusion using an unlubricated aluminum plunger with K=0.52.

Appendix D

Results of yield stress measurement using a gun rheometer

Table D1. Data of tomato paste measured using gun rheometer.

Ratio L/D	Pressure (mmH_2O)	Pressure (Pa)	Velocity (mm/min.)	Stress (Pa)
13.0	153	1500.376	0.5	28.853
12.7	156	1529.795	1.0	30.114
12.7	182	1784.761	2.0	35.133
12.5	225	2206.435	3.0	44.129
13.5	285	2794.818	8.0	51.756
13.5	309	3030.171	8.0	56.114
13.5	342	3353.782	10.0	62.107
14.0	380	3726.424	10.0	66.543
15.0	480	4707.062	15.0	78.451
15.0	481	4716.869	15.0	78.614
15.6	525	5148.395	16.0	82.506
15.7	578	5668.088	22.0	90.256

Sample tubes had diameter of 10 mm.
Yield stress or stress at zero velocity = 30.621 Pa.
Coefficient correlation = 0.984

Table D2. Data of peanut butter measured using gun rheometer.

Ratio L/D	Pressure (mmH_2O)	Pressure (Pa)	Velocity (mm/min.)	Stress (Pa)
15.0	265	2598.69	1.0	43.31
17.5	305	2990.95	1.0	42.73
14.8	163	1598.44	1.5	27.00
15.0	342	3353.78	3.0	55.90
18.0	463	4540.35	3.5	63.06
17.2	278	2726.17	4.0	39.62
15.8	336	3294.94	5.0	52.13
17.8	378	3706.81	6.0	52.06
18.6	492	4824.74	8.0	64.85
20.0	658	6452.60	10.0	80.66
17.0	476	4667.84	12.0	68.64
17.0	397	3893.13	13.0	57.25

Sample tubes had diameter of 10 mm.
Yield stress or stress at zero velocity = 40.152 Pa.
Coefficient correlation = 0.703

Table D3. Data of mixed cereal measured using gun rheometer.

Ratio L/D	Pressure (mmH_2O)	Pressure (Pa)	Velocity (mm/min.)	Stress (Pa)
13.4	27	264.8	2.0	4.94
19.0	43	421.7	4.0	5.55
13.9	37	362.8	5.0	6.53
19.5	55	539.4	5.0	6.91
13.8	32	313.8	6.0	5.68
14.3	31	304.0	6.0	5.31
19.9	43	421.7	6.0	5.30
14.5	53	519.7	8.0	8.96
20.9	67	657.0	10.0	7.86
18.6	74	725.7	12.0	9.75
15.7	53	519.7	14.0	8.28
13.2	53	519.7	15.0	9.84
21.0	89	872.9	15.0	10.39
22.9	93	912.0	20.0	9.96
16.0	75	735.5	21.0	11.49

Sample tubes had diameter of 10 mm.
Yield stress or stress at zero velocity = 4.471 Pa.
Coefficient correlation = 0.904

Appendix E

Results of yield stress measurement using a vane device

Table E1. Data of mixed cereal measured using vane device.

RPM	Windows	T_m (dyne cm)	σ_0 (dyne cm)	σ_0 (Pa)
0.5	13.0	7474	142.9	14.3
	13.0	7474	142.9	14.3
	11.8	6785	129.7	13.0
1.0	16.0	9429	180.2	18.0
	16.0	9084	173.6	17.4
	16.0	9141	174.7	17.5
2.5	21.0	12189	233.0	23.3
	20.0	11729	224.2	22.4
	21.0	11959	228.6	22.9