This is to certify that the

thesis entitled

"Methods to Characterize Power Law Fluids Using a Brookfield Viscometer"

presented by

Jenni L. Briggs

has been accepted towards fulfillment
of the requirements for

M.S. degree in **Agricultural Engineering**

James F. Steffe
Major professor

Date **August 22, 1995**

Methods to Characterize Power Law Fluids Using a Brookfield Viscometer

By

Jenni L. Briggs

A THESIS

Submitted to
Michigan State University
in partial fulfillment of the requirements
for the degree of

MASTER OF SCIENCE

Department of Agricultural Engineering

1995

ABSTRACT

**Methods to Characterize Power Law Fluids
Using a Brookfield Viscometer**

By

Jenni L. Briggs

Using a Brookfield viscometer equipped with a small sample adapter and flag impeller, a technique to rheologically characterize power law fluid foods was developed. Mixer viscometry methodologies (slope and matching viscosity methods) were employed to determine average shear rates. Results provide a simple procedure to calculate apparent viscosity and shear rate from the Brookfield data consisting of percent torque and rotational speed. A quality control test protocol was devised using the small sample adapter and flag impeller.

Mitschka (1982, Rheo. Acta 21:207-209) proposed a method to evaluate rheological properties of power law fluids using a Brookfield viscometer and disc spindles. This method, and a simplified Mitschka approximation, developed in this study, was applied to fluid foods. Both methods predicted power law properties that were in good agreement with those determined using a controlled stress rheometer equipped with a cone and plate sensor.

Acknowledgments

I would like to extend my sincerest appreciation to Dr. James Steffe. Without his support, encouragement and advice, this thesis would not have been possible. Also, the support I received from my parents, Betty and Gale, was an integral part of my success. Lastly, thanks to my Agricultural Engineering family (faculty, staff, and friends) at Michigan State University for giving me a warm home.

Table of Contents

Table of Contents (Cont'd.)

List of Tables

List of Figures

List of Figures (Cont'd.)

List of Figures (Cont'd.)

Nomenclature

A	=	constant (dimensionless)
d	=	spindle diameter (m)
f	=	multiplication factor (Pa s)
k'	=	mixer viscometer constant
$k_{N\gamma}$	=	Mitschka average shear stress multiplication factor
$k_{\alpha\sigma}$	=	Mitschka (average) shear stress multiplication factor
K	=	consistency coefficient (Pa s^n)
M	=	torque (N m)
n	=	flow behavior index (dimensionless)
N	=	rotational speed (rpm)
P	=	power (N m s^{-1})
$\dot{\gamma}$	=	shear rate (s^{-1})
$\dot{\gamma}_a$	=	average shear rate (s^{-1})
η	=	apparent viscosity (Pa s)
μ	=	Newtonian viscosity (Pa s)
μ_{pl}	=	plastic viscosity of a Bingham fluid (Pa s)
σ	=	shear stress (Pa)
σ_a	=	average shear stress (Pa)
σ_o	=	yield stress (Pa)
Ω	=	rotational speed (rev/s)

Chapter 1

Introduction

1.1. Fluid Food Rheology

Rheology is the study of the flow of matter and how matter responds to applied stress or strain. Rheological applications in food processing are far reaching. Examples include process engineering calculations, quality control, and product development. In food process engineering knowledge of the rheological data of a fluid is used for proper equipment design. Rheological properties can also serve as the basis of quality control applications providing guidelines for product consistency. Flow behavior of various products can also be correlated to consumer acceptance when producing new products.

Several constitutive models have been developed to describe the relationship between shear stress and shear rate of fluids. A Newtonian model,

$$\sigma = \mu\dot{\gamma} \qquad [1.1]$$

specifies that shear rate is directly proportional to shear stress. Common examples include corn syrup, corn oil, and honey. A power law model

$$\sigma = K\dot{\gamma}^{n} \qquad [1.2]$$

is used to describe a fluid that is shear-thickening ($n > 1$) or shear-thinning ($n < 1$). When $n=1$ the power law model collapses into the

Newtonian model. The flow behavior index, n, indicates to what extent the fluid deviates from Newtonian behavior. Newtonian and power law fluids are only two of several types of fluids. Other well known constitutive models are Bingham plastic ($\sigma = \mu_{pl}(\dot{\gamma}) + \sigma_o$), Herschel-Bulkley ($\sigma = K(\dot{\gamma})^n + \sigma_o$), and Casson ($\sigma^{0.5} = K(\dot{\gamma})^{0.5} + \sigma_o^{0.5}$). In all three of the previously listed models, a presence of a yield stress, the minimum amount of stress to cause flow, is taken into account.

Definition of apparent viscosity is derived from the constitutive model. Apparent viscosity (η) is the shear stress divided by shear rate. For a Newtonian fluid, the apparent viscosity equals the Newtonian viscosity (μ):

$$\eta = \mu = \sigma/\dot{\gamma} \qquad [1.3]$$

showing that it is shear rate independent. For a power law fluid, however, apparent viscosity is shear rate dependent:

$$\eta = K(\dot{\gamma})^n / \dot{\gamma} = K(\dot{\gamma})^{n-1} \qquad [1.4]$$

When referring to apparent viscosity of a power law fluid, the shear rate used when performing the calculation must be stated to provide meaningful information.

Time-dependent behavior of a fluid will strongly influence rheological parameters. Not all fluids exhibit time-dependency but those which do are considered thixotropic (thinning with time) or rheopetic (thickening with time) upon the application of a constant shear field. Either characteristic may be reversible or irreversible depending on the fluid. Thixotropy is generally due to the breakdown of microstructure within the fluid.

Given the importance of flow behavior of fluids, methods of making rheological measurements have been developed. Two major categories of measuring devices are tube and rotational viscometers. Subcategories of tube viscometry include glass capillaries, high pressure capillaries and pipe viscometers. Rotational viscometry encompasses cone and plate, parallel plate, and concentric cylinder geometries, and mixer systems. In rotational viscometry, part of the apparatus is rotated. For example, the cone and plate and parallel plate viscometers require filling a small gap with sample fluid and rotating the cone or plate at a known speed. A shear rate may be calculated based on system geometry and rotational speed. Shear stress is calculated as a function of torque experienced by the cone or plate. In mixer viscometry, an impeller is immersed into a sample fluid and rotated. The torque caused by the fluid on the impeller is measured. Rheological properties may be determined as a function of rotational speed, torque, and system geometry. Mixer viscometer techniques are especially useful when evaluating fluids with large particles, particle settling, time-dependent behavior, or slip tendencies (Steffe,1992).

1.2. Literature Review

1.2.1. Mixer Viscometry

Evaluating non-Newtonian fluids using mixer viscometry was originally investigated by Metzner and Otto (1957). In particular, they proposed a quantitative method for classifying power law fluids using a mixing system and matching viscosity concept. This concept assumes that the average shear rate for a non-Newtonian fluid equals the average shear rate for a Newtonian fluid when the Newtonian viscosity equals the apparent viscosity of the non-Newtonian fluid. The shear rate was defined in terms of an experimentally determined mixer viscometer constant (k') multiplied by the rotational speed of the impeller.

Since the research performed by Metzner and Otto, many other studies have been conducted in mixer viscometry. The effects of impeller type (turbine, marine type, flag impeller, etc.), system geometry, and different fluids have been examined. Castell-Perez and Steffe (1992) have completed a thorough literature review on the historical development and recent activity in mixer viscometry.

Rao (1975) began applying mixer viscometry to fluid foods. In some cases, mixer techniques were superior to conventional concentric cylinder rotational viscometry due to the presence of particulates and suspensions. Unlike Metzner and Otto (1957), Rao applied the slope method proposed by Rieger and Novak (1973) to calculate a mixer viscometry constant. Rheological parameters of power law foods (apple sauce, tomato puree, apple pulp, and tomato pulp) were characterized. Later, a comparison study of the slope method and matching viscosity method to determine average shear rates in a mixing system was investigated by Rao and Cooley (1984). Both methods yielded similar mixer viscometry constants for the flag and star impellers tested.

Castell-Perez et al. (1991) proposed an alternative technique to the slope and matching viscosity method. For shear rate and shear stress calculations, the approach was analogous to the concentric cylinder system calculations found in Steffe (1992). Findings revealed that empirical equations relating system geometry (cup and paddle geometry, and immersion depth) estimated the shear stress and shear rate reasonably well. The primary advantage of this method is reduction of data collection because reference fluids for k' determination are not needed. Fluids used in this study included various concentrations of hydroxypropyl methylcellulose and salad dressing.

Castell-Perez and Steffe (1990) investigated the effects of rotational speed, fluid properties, and system geometry on the mixer viscometer constant (k') using paddle impellers. Two different approaches were used to determine k', slope and matching viscosity method. Larger k' values were found using the latter method. Results indicated that k' was constant above rotational speeds of 0.3 rev/s (20 rpm), k' was a function of fluid properties (K and n), and k' was affected by various ratios of impeller to cup diameter. As the impeller to cup diameter ratio increased, the value of k' decreased in most cases. Mackey et al. (1987) investigated the effects of shear-thinning fluids on k' using a flag impeller. They found that k' was larger for fluids with smaller flow behavior indexes (n). Also, findings suggested that k' is not constant at low rotational speeds.

Mixer viscometry has proven successful in the rheological evaluation of several foods. Steffe and Ford (1985), for example, investigated the shelf-stability of starch-thickened strained apricots. Using a rotational viscometer (Haake RV12) and a paddle for mixing, the consistency coefficient (K) and flow behavior index (n) were evaluated. Mixer viscometry techniques were also used to quantify the degree of thixotropy found in the strained apricots (Ford and Steffe, 1986). Dolan and Steffe (1990) used a mixer viscometry approach to model rheological behavior of gelatinizing starch (corn and bean) solutions. The viability of determining rheological characteristics of gelatinizing corn starch was investigated by Steffe et al. (1989). Instrumentation consisted of a Brookfield viscometer equipped with a small sample adapter and flag impeller. Results were described in terms of torque, not apparent viscosities and shear rates.

1.2.2. Brookfield Viscometers Using Disc Spindles

A rotational viscometer based on mixer viscometry theory that has found world wide usefulness is the Brookfield viscometer. Some of the major industries utilizing this instrument are food, cosmetic, pharmaceutical, paper, and chemical. The low cost and compact size make this viscometer attractive for measuring rheological behavior of many fluids. As a consequence of the Brookfield's popularity, several ASTM specifications are based on this viscometer (Table 1.1).

Standard sensors for viscosity measurements with the Brookfield viscometers (RV/HA/HB models) are disc spindles with varying diameters. Other optional, yet popular, supplemental equipment are T-bar impellers and small sample adapters. Flag impellers are also available through Brookfield. To date, however, flag impellers have been used for very few applications.

During testing using a standard disc, a spindle immersed in a fluid is rotated creating a torque upon the spindle and an ill defined shear field of axial and radial flow. For Newtonian fluids, viscosity is calculated from simply multiplying the torque scale response by a factor that was predetermined at Brookfield Engineering Laboratories, Inc.. This factor -- dependent on spindle, rotational speed, and viscometer model -- was calculated using Newtonian fluid standards. Factors may

Table 1.1. ASTM specifications relating to the Brookfield Viscometer.

Standard No.	ASTM Specification Title
C 965-81	Practices for measuring viscosity of glass above the softening point
D 115-85	Testing varnishes used for electrical insulation
D 789-86	Test methods for determination of relative viscosity, melting point, and moisture content of polyamide
D 1076-80	Concentrated, ammonia preserved creamed and centrifuged natural rubber latex
D 1084-81	Tests for viscosity of adhesives
D 1417-83	Testing rubber latices - synthetic
D 1439-83	Testing sodium carboxymethylcellulose
D 1638-74	Testing urethane foam isocyanate raw materials
D1824-87	Test for apparent viscosity of plastisols and organosols at low shear rates by Brookfield Viscometer
D 2196-86	Test for rheological properties of non-Newtonian material by rotational (Brookfield) viscometer
D 2364-85	Testing hydroxyethylcellulose
D 2393-86	Testing for viscosity of epoxy resins and related components
D 2556-80	Test for apparent viscosity of adhesives having shear rate dependent flow properties
D 2669-82	Test for apparent viscosity of petroleum waxes compounded with additives (hot melts)
D 2849-80	Test for urethane foam polyol raw materials
D 2983-80	Test for low-temperature viscosity of automotive fluid lubricants measured by Brookfield Viscometer
D 2994-77	Testing rubberized tar
D 3232-83	Method for measurement of consistency of lubricating greases at high temperatures
D 3232-83	Apparent viscosity of hot melt adhesives and coating materials
D 3716-83	Testing emulsion polymers for use in floor polishes
D 4016-81	Test method for viscosity of chemical grouts by the Brookfield Viscometer
D 4300-83	Test method for effect of mold contamination of mold contamination on permanence of adhesive preparations and adhesive films
D 4402-84	Standard method for viscosity determinations of unfilled asphalts using the Brookfield Thermosel Apparatus
D 5133-90	Stand test method for low temperature, low shear rate, viscosity/temperature dependence of lubricating oils using a temperature-scanning technique

also be determined for other geometries such as the flag impeller and small sample adapter.

The same simplistic approach for determining viscosity does not apply to power law fluids. Instead, an apparent viscosity ($\eta = K(\dot{\gamma})^{n-1}$) at a specific shear rate must be calculated. Unlike Newtonian viscosity, apparent viscosity of a power law fluid is shear-dependent. Without a defined shear field, this calculation can not be accomplished.

Previous research has investigated methods to adapt the torque output of the Brookfield, while using disc spindles, into apparent viscosity readings for non-Newtonian fluids. Williams (1979) adapted a complicated mathematical model based on the equations of motion to calculate an average shear rate and determining power law fluid parameters. The spindles were assumed to be infinitely thin with a finite radius. Results from this research correlated well with experimental data but the method is cumbersome due to lengthy computational requirements.

Mitschka (1982) developed a simple procedure to calculate apparent viscosity using data from Brookfield discs. Conversion factors based on spindle number and the flow behavior index (n) are used to determine average shear rate and shear stress. The experimental procedure is not well documented in the English language literature but may be found in Czechoslovakian (Mitschka, 1982). This method was derived from the basic theoretical development and apparently does not use a conventional mixer viscometry approach. Savvas et al. (1994) calculated apparent viscosity measurements of polymer solutions based on the Mitschka method. Apparent viscosities of the solutions were not tested using other accepted rheological practices such as rotational viscometry.

Durgueil (1987) proposed a method of calculating shear stress and shear rate based on torque, rotational speed, and spindle geometry from a Brookfield viscometer. Shear rate was evaluated from the deformation tensor for the power law model. Shear stress was determined by multiplying the torque by the wetted area. Simplified equations were developed for cylindrical, T-bar, and disc spindles. The accuracy of these equations was not collaborated using other rheological means.

ASTM specification D 2196-86, Standard Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometers, describes a procedure for determining apparent viscosity, and determining the degree of shear-thinning and thixotropy using disc spindles. The apparent viscosity calculation does not take into account the effects of shear-dependency on power law fluids; hence, a true apparent viscosity cannot be found using this method. This method can be used for a crude quality control test but is not applicable for determining absolute rheological behavior.

1.3. Objectives

The objectives of this research are to 1) develop mixer viscometry techniques (slope and matching viscosity methods) to calculate apparent viscosity using a Brookfield viscometer with a flag impeller and small sample adapter, 2) evaluate the simple approach proposed by Mitschka (1982) to calculate apparent viscosities of power law fluid foods using a Brookfield viscometer and disc spindles.

Chapter 2

Theoretical Development

2.1. Mixer Viscometry

Mixer viscometry in laminar flow ($N_{Re,I} < 63$) is based on the following relationship developed through dimensionless analysis (Steffe, 1992):

$$N_{Po} = \frac{A}{N_{Re,I}} \qquad [2.1]$$

where A is a dimensionless constant that depends on the system geometry. The power number (N_{Po}) and impeller Reynolds number $(N_{Re,I})$ are defined as

$$N_{Po} = \frac{P}{\rho\Omega^3 d^5} \qquad [2.2]$$

and

$$N_{Re,I} = \frac{\rho\Omega d^2}{\mu} \qquad [2.3]$$

Substituting the definitions of the power number (Eq. [2.2]) and the impeller Reynolds number (Eq. [2.3]) into Eq. [2.1] yields

$$\frac{P}{d^5\Omega^3\rho} = \frac{A\mu}{d^2\Omega\rho} \qquad [2.4]$$

where $P = M\Omega$. Eq. [2.4] simplifies to

$$\frac{M}{d^3\Omega} = A\mu \qquad [2.5]$$

Eq. [2.5] is convenient because it eliminates the need for measuring fluid density. If apparent viscosity (η) is substituted for Newtonian viscosity (μ), Eq. [2.5] may be applied to non-Newtonian fluids:

$$\frac{M}{d^3\Omega} = A\eta \qquad [2.6]$$

Basing the definition of apparent viscosity on an average shear rate ($\dot{\gamma}_a$)

$$\eta = K(\dot{\gamma}_a)^{n-1} \qquad [2.7]$$

and assuming the average shear rate is

$$\dot{\gamma}_a = k'\Omega \qquad [2.8]$$

Eq. [2.6] becomes

$$\frac{M}{d^3\Omega} = AK(\dot{\gamma}_a)^{n-1} = AK(k'\Omega)^{n-1} \qquad [2.9]$$

This equation provides a basis for calculating numerical values of k'. In a mixing system, the maximum shear rate will be found at the impeller tip. k', the mixer viscometer constant, is a function of mixer geometry and may be influenced by rotational speed (Ω) and flow behavior index (n).

2.1.1. Evaluation of Mixer Viscometry Constant (k')

2.1.1.1. Slope Method

k' may be determined using the slope method. Taking the logarithm of Eq. [2.9] and rearranging the results gives

$$\log_{10}\left(\frac{M}{Kd^3\Omega^n}\right) = \log_{10}(A) - (1-n)\log_{10}(k') \qquad [2.10]$$

Hence, k' may be determined from a plot of $\log_{10}\left(M/\left(Kd^3\Omega^n\right)\right)$ versus (1-n). If this relationship is non-linear, the assumption $\dot{\gamma}_a = k'\Omega$ is not valid (Steffe, 1992). Eq. [2.10] implicitly assumes that k' is not a function of angular velocity or the flow behavior index.

2.1.1.2. Matching Viscosity Method

Matching viscosity method is an alternative to the slope method for determining k'. However, both methods stem from the Reynolds number and power number relationship described by Eq. [2.1]. This technique assumes that the Newtonian viscosity is equal to the apparent viscosity of a non-Newtonian fluid when the average shear rates of both fluids are equal. Matching viscosity method involves the following steps:

1. Establish power number and Reynolds number relationship for Newtonian fluids defined by Eq. [2.1]. A, a constant resulting from the mixing system geometry, is the slope of the power number versus the inverse of the Reynolds number plot.

2. Calculate apparent viscosity given by Eq. [2.6] using power law reference fluids:

   $$\eta = \frac{M}{Ad^3\Omega} \qquad [2.11]$$

   A power law reference fluid is one in which the values of K and n have been established using standard rheological methods.

3. Recalling that $\eta = K(\dot{\gamma}_a)^{n-1}$, solve for the average shear rate:

$$\dot{\gamma}_a = \left[\frac{\eta}{K}\right]^{\frac{1}{(n-1)}} \qquad [2.12]$$

   η is found from Eq. [2.11].

4. Repeating steps 1-3 using numerous impeller speeds leads to the determination of k'. Remembering $\dot{\gamma}_a = k'\Omega$, k' is determined from a plot of $\dot{\gamma}_a$ versus Ω or a single point value of k' may be calculated as

$$k' = \dot{\gamma}_a / \Omega \qquad [2.13]$$

The matching viscosity method does not have the implicit limitations of Eq. [2.10] and allows k' to be evaluated as a function of both the angular velocity and the flow behavior index. Reference fluids with different values of the flow behavior index are required to determine the influence of n on k'.

2.1.2. Determining Non-Newtonian Fluid Properties

2.1.2.1. Power Law Fluids

Power law fluid parameters (K and n) are easily determined if k' known and assumed to be constant. From Eq. [2.9] K is found as a function of y-intercept and n is equal to the slope of a plot of $\log_{10}(M)$ versus $\log_{10}(\Omega)$:

$$\log_{10}(M) = \log_{10}\left(d^3 A K k'^{(n-1)}\right) + n\log_{10}(\Omega) \qquad [2.14]$$

This definition of n is valid regardless of the assumption made on k'.

If A is unknown, properties of an unknown fluid may be determined provided a reference fluid with known rheological properties is available. The procedure for evaluating the flow behavior index of the unknown fluid remains the same; n equals the slope of a plot of $\log_{10}(M)$ versus $\log_{10}(\Omega)$. To determine K, begin with Eq. [2.9] and solve for the torque for two fluids:

$$M_x = d^3 A K_x (k')^{n_x - 1} \Omega^{n_x} \qquad [2.15]$$

and

$$M_y = d^3 A K_y (k')^{n_y - 1} \Omega^{n_y} \qquad [2.16]$$

where subscripts x and y refer to the unknown fluid and known fluid, respectively. Taking the ratio of the Eq. [2.15] and Eq. [2.16], and solving for Kx yields

$$K_x = K_y \left(\frac{M_x}{M_y} \right) \left(\frac{\Omega_y^{n_y} (k')^{n_y}}{\Omega_x^{n_x} (k')^{n_x}} \right) \qquad [2.17]$$

Eq. [2.17] eliminates A from the calculation. The methods presented in this section for determining K are not applicable if k' is considered to be a function of n or Ω.

2.1.2.2. Time-independent Fluids

Rheological parameters of time-independent fluids may be determined from a rheogram of shear stress versus shear rate. Assuming k' is constnt, average shear rate is calculated from Eq. [2.8]. Average shear stress equals

$$\sigma_a = \eta \dot{\gamma}_a \qquad [2.18]$$

where η is calculated from Eq. [2.6]. Curve fitting of shear stress versus shear rate data leads to a constitutive model (Bingham plastic, Casson, power law, etc.) characterizing the fluid. Note that this method is a good alternative for the special case of power law fluid calculations discussed in the previous section.

2.2. Mitschka Method

Mitschka (1982) offers a method to determine average shear rates, shear stresses, and apparent viscosities of power law fluids using Brookfield disc spindles. Mitschka's procedure is based on previous theoretical research (published in Czechoslovakian) and cited in Mitschka (1982). The flow behavior index (n) is found as the slope of the logarithm of shear stress versus logarithm of rotation speed (N in rpm):

$$n = \frac{d(\log_{10} \sigma_a)}{d(\log_{10} N)} = \frac{d(\log_{10} M)}{d(\log_{10} N)} \qquad [2.19]$$

This calculation is essentially the same as finding n as the slope of Eq. [2.14]. The average shear stress (in Pa) is

$$\sigma_a = k_{\alpha\sigma}(\text{dial reading}) \qquad [2.20]$$

where "dial reading" represents percent torque displayed on the Brookfield viscometer. An average shear rate (in s-1) is calculated as

$$\dot{\gamma}_a = k_{N\gamma}(N) \qquad [2.21]$$

Values of both $k_{\alpha\sigma}$ and $k_{N\gamma}$ are found in Table 2.1. $k_{\alpha\sigma}$ is a function of the spindle number and $k_{N\gamma}$ is a function of both the spindle number and the flow behavior index. Apparent viscosity (in Pa s), based on average shear rate, is determined by dividing Eq. [2.20] by Eq. [2.21]:

$$\eta = \frac{\sigma_a}{\dot{\gamma}_a} \qquad [2.22]$$

A simplified method to calculate average shear rate has been developed in this research. From a plot of $k_{N\gamma}$ values versus n (Fig. 2.1), a power law relationship was established as

$$k_{N\gamma} = 0.263(n)^{-0.771} \qquad [2.23]$$

with r2 > 0.97. Using the Mitschka approximation (Eq. [2.23]) to calculate average shear rate combines $k_{N\gamma}$ values found in Table 2.1 into one mathematical expression and eliminates linear interpolation between n values. The average shear stress calculation remains unchanged -- Eq. [2.20] is used. Apparent viscosity is determined from Eq. [2.22] where the average shear rate is

$$\dot{\gamma}_a = \left(0.263(n)^{-0.771}\right)N \qquad [2.24]$$

Table 2.1. Conversion factors for the method described by Mitschka (1982).

Brookfield spindles			1	2	3	4	5	6	7
$k_{\alpha\sigma}$			0.035	0.119	0.279	0.539	1.05	2.35	8.40
	n =	0.1	1.728	1.431	1.457	1.492	1.544	1.366	1.936
		0.2	0.967	0.875	0.882	0.892	0.907	0.851	1.007
		0.3	0.705	0.656	0.656	0.658	0.663	0.629	0.681
		0.4	0.576	0.535	0.530	0.529	0.528	0.503	0.515
$k_{N\gamma}$		0.5	0.499	0.458	0.449	0.445	0.442	0.421	0.413
		0.6	0.449	0.404	0.392	0.387	0.382	0.363	0.346
		0.7	0.414	0.365	0.350	0.343	0.338	0.320	0.297
		0.8	0.387	0.334	0.317	0.310	0.304	0.286	0.261
		0.9	0.367	0.310	0.291	0.283	0.276	0.260	0.232
		1.0	0.351	0.291	0.270	0.262	0.254	0.238	0.209

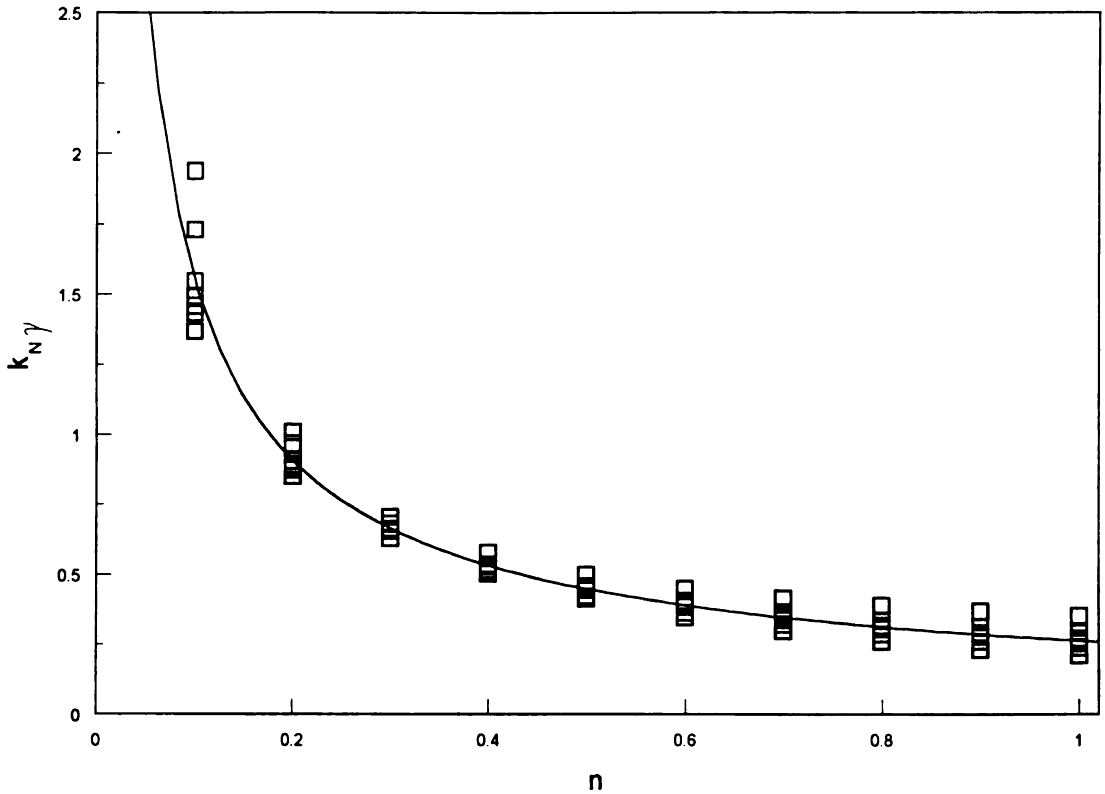

Figure 2.1. $k_{N\gamma}$ versus n to determine the Mitschka approximation for average shear rate.

Chapter 3

Materials and Methods

3.1. Equipment

A Haake RS-100 controlled stress rheometer equipped with a cone and plate sensor was used to determine the rheological properties of all fluids considered in this study. Temperature control was obtained using a water bath with a variation ± 0.1 °C. Calibration of the RS-100 was completed using standard silicon oils.

Two Brookfield viscometers with different torque capacities were used. The 1/2 RVDT and RVDT have a maximum torque capacity of 0.0003594 N m (3594 dyne cm) and 0.0007187 N m (7187 dyne cm), respectively. Between the two instruments, a variety of fluids with a large range of viscosities and apparent viscosities could be measured. Calibration of the Brookfield viscometers was completed using standard silicone oils (Brookfield Engineering Laboratories Inc., Stoughton, MA).

To facilitate temperature control and the testing of a small quantity of fluid, a small sample adapter, available from Brookfield Laboratories, Inc., is ideal. The cylindrical vessel (model SC4-13R) is 6.48 cm in height and 1.9 cm in diameter with a volumetric capacity of 18 ml. A thermistor fitted into the bottom of the sample chamber allows temperature monitoring. Temperature control was achieved with a water jacket used in conjunction with the small sample adapter. A flag impeller with two staggered blades with a diameter of 1.5 cm was used (Fig. 3.1).

Only disc spindles are used for the Mitschka method. Fig. 3.2 and 3.3 and Table 3.1 illustrate the geometry of spindles # 1 - 7 and lists the dimensions, respectively. Following Brookfield Laboratories, Inc. guidelines, a 600 ml low form Griffin beaker was used as the sample vessel for various fluid foods. During all experiments, the guard leg was not used. A mercury thermometer provided temperature monitoring.

3.2. Experimental Materials

Silicon oil, glycerin, and honey were the Newtonian fluids used to determine the value of A from the power number and Reynolds number relationship given by Eq. [2.1]. Testing these fluids at numerous temperatures resulted in Newtonian behavior with varying viscosities.

Methylcellulose, hydropropyl methylcellulose (Methocel cellulose ethers products, Dow Chemical Co.) and guar gum (Sigma Co.) solutions provided power law reference fluids for determining k' for the small sample adapter with the flag impeller. A 1.5 % (by weight) A4M methylcellulose solution and a 1.0 % K100M hydroxypropyl methylcellulose solution were prepared by dispersion in hot water as directed by the manufacturer. Approximately 180 ml of dionized water (1/3 of total volume) was heated to 95 °C. The appropriate amount of Methocel was gradually added while agitating the fluid. Agitation continued until all particles were thoroughly wetted. Then, cold water was added to achieve the desired volume of approximately 550 ml.

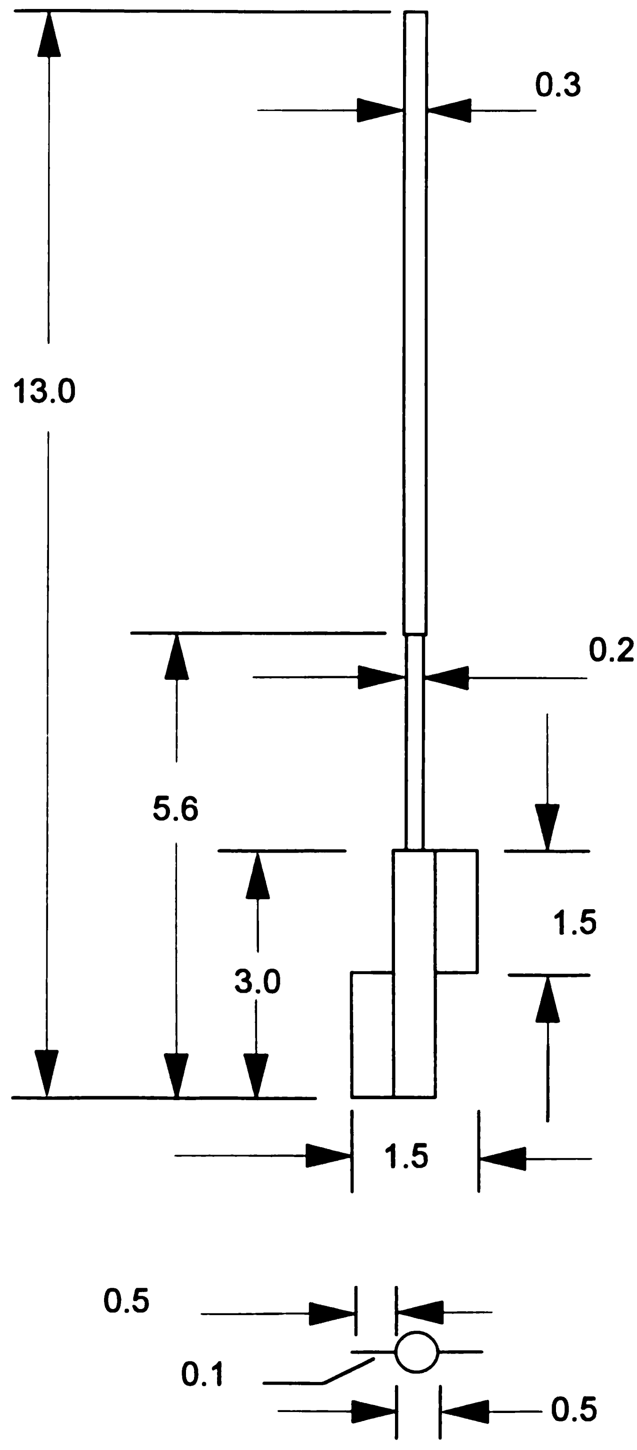

Figure 3.1. Flag impeller used with the small sample adapter (all dimensions in cm).

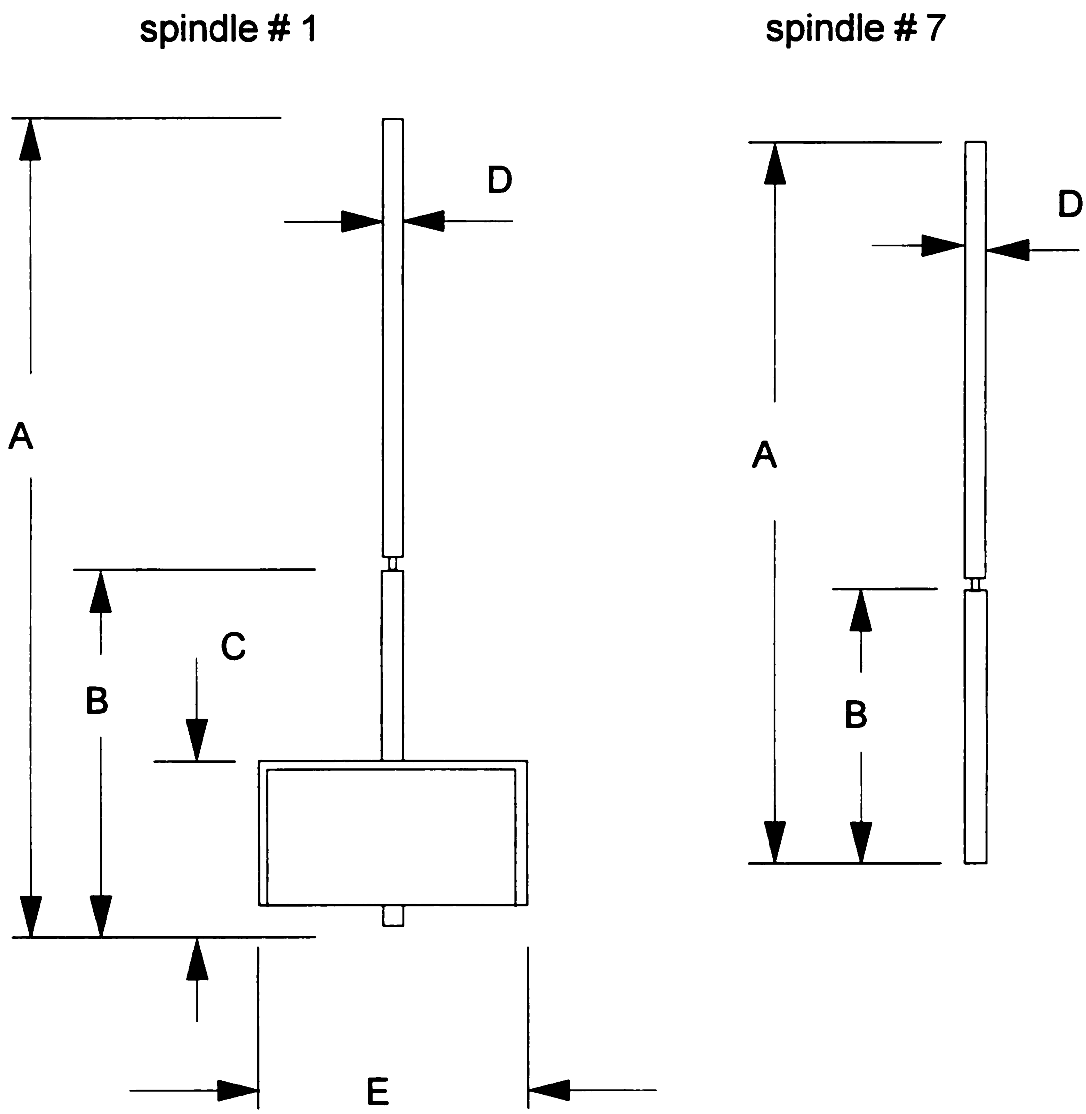

Figure 3.2. Brookfield disc spindles, numbered 1 and 7.

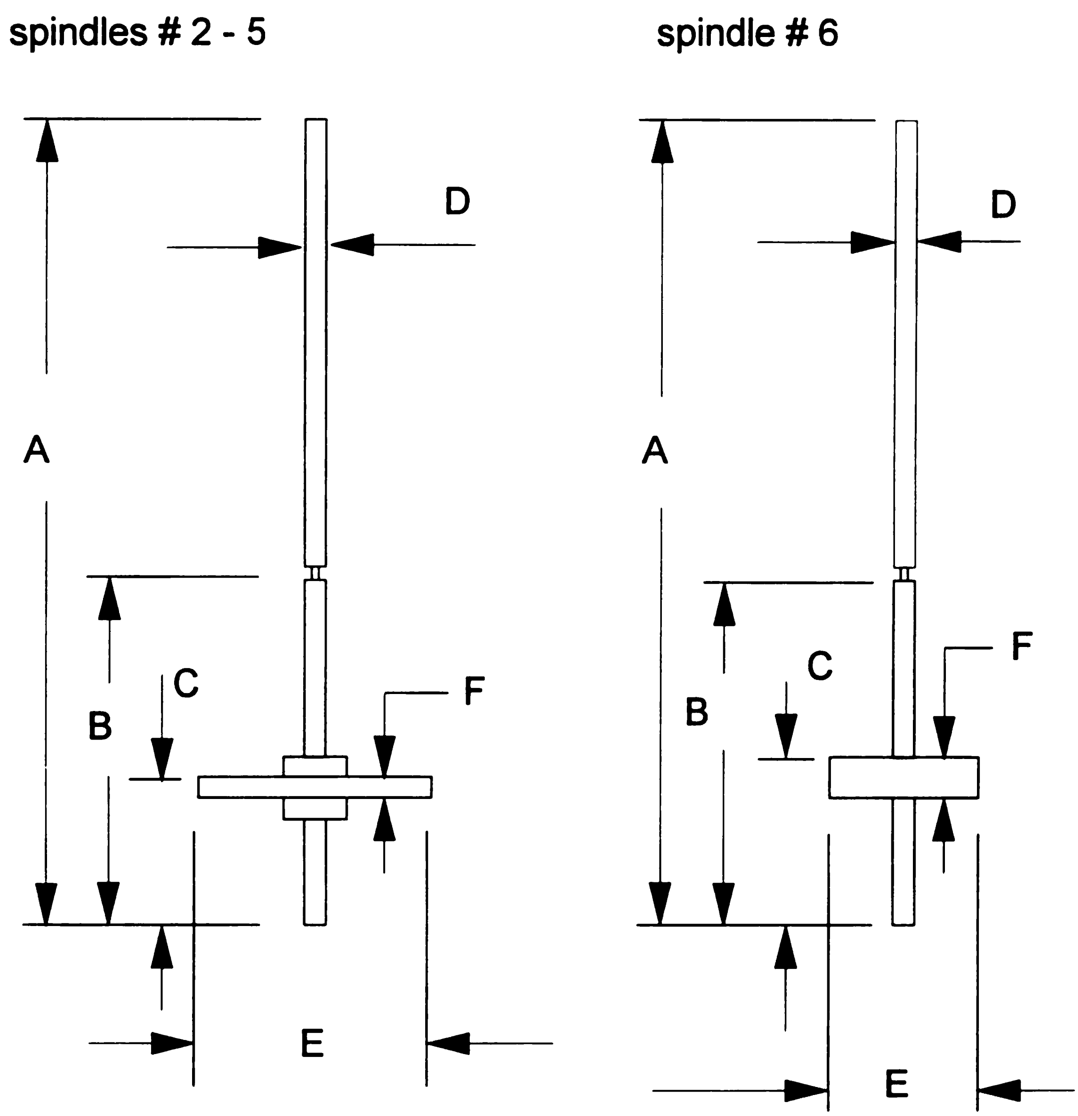

Figure 3.4. Brookfield disc spindles, numbered 2 - 5 and 6.

Table 3.1. Dimensions of Brookfield disc spindles (dimensions in cm).

spindle	A	B	C	D	E	F
1	13.30	6.11	2.70	0.32	5.63	N/A
2	13.30	4.92	2.70	0.32	4.69	0.16
3	13.30	4.92	2.70	0.32	3.47	0.16
4	13.30	4.92	2.70	0.32	2.73	0.16
5	13.30	4.92	2.70	0.32	2.11	0.16
6	13.30	4.92	3.02	0.32	1.46	0.16
7	13.30	5.99	N/A	0.32	N/A	N/A

Once the solution was at room temperature, stirring continued for an additional 30 minutes.

Hydroxypropyl methylcellulose is a surface treated powder. The mixing procedure did not require hot water dispersion. Instead, the powder is added directly to water. Upon raising the pH to between 8.5 and 9.0, the powder is able to hydrate and viscosity increases. 0.8 % and 1.5 % J40M hydroxypropyl methylcellulose solutions were made by mixing the appropriate amount of powder with water. The pH was raised to 8.5 - 9.0 with sodium hydroxide. Agitation continued for 30 minutes after the pH was adjusted.

Guar gum solution was prepared by a hot water dispersion technique. The guar gum powder was added to room temperature water. Once the particles were thoroughly wetted, the solution (approximately 550 ml) was heated to 90 °C while stirring. After reaching 90 °C, the solution was cooled. Agitation continued until the solution came to room temperature.

Commercially available fluid foods were purchased locally. The variety of foods tested are listed in Table 3.2. These materials were used directly from the original containers and were not altered in any way.

3.3 Data Collection Method

3.3.1. Reference Fluids

Newtonian fluids (silicon oil, glycerin, and honey) for determining A, defined by (Eq. [2.1]), and power law fluids (Methocel and guar gum solutions) for determining k' comprised the reference fluids for the small sample adapter research. During testing, a reference fluid was

Table 3.2. Fluid foods evaluated using the flag impeller in conjunction with the small sample adapter and disc spindles.

Flag impeller with small sample adapter	Disc spindles (Mitschka method)
banana baby food (Gerber)	banana puree (Chiquita)
chocolate syrup (Hershey)	Catalina salad dressing (Kraft)
enchilada sauce (Old El Paso)	chocolate syrup (Hershey)
mustard (French)	enchilada sauce (Old El Paso)
strawberry syrup (Smucker)	pancake syrup lite (Mrs. Butterworth)
turkey gravy (Heinz)	

placed into the small sample adapter. Then, the adapter was raised to immerse the flag impeller to a depth of 5.6 cm (Fig. 3.4). To eliminate time-dependent behavior the impeller was rotated at the highest speed possible, between 0.5 to 100 rpm, while avoiding torque overload (greater than 90 % on the dial reading). Preshearing continued until thixotropic behavior, if present, was eliminated. Once steady state conditions were met, experimental data were collected at various impeller speeds and the corresponding torque and temperature were recorded for each sample. Only percent torque readings between 5 % to 90 % were accepted for all Brookfield data taken. From the calibration procedure, this was determined to be the linear range. Not all samples were subjected to the full range of rotational speeds due to torque limitations of the instrument. This procedure was repeated twice for each fluid at each temperature.

All reference fluids were evaluated with a Haake RS-100 controlled stress rheometer using cone and plate geometry (60 mm plate, 4° cone). Temperature was controlled to ± 0.1 °C and duplicated the testing temperature of the Brookfield experiments. Each fluid was subjected to preshearing followed by a steady state shear rate protocol. Shear rate ramps from 0.15 to 30 s^{-1}, then from 30 to 0.15 s^{-1}, eliminated time-dependent behavior in the sample. Immediately following the shear rate ramps, torque measurements were taken after steady state conditions were obtained at periodic shear rates (1 s^{-1} increments) over the range of 0.15 s^{-1} to 30 s^{-1}. Two replications were completed. Preliminary data analysis indicated the shear rate range achieved by the flag impeller used in conjunction with the small sample adapter was less than 30 s^{-1}.

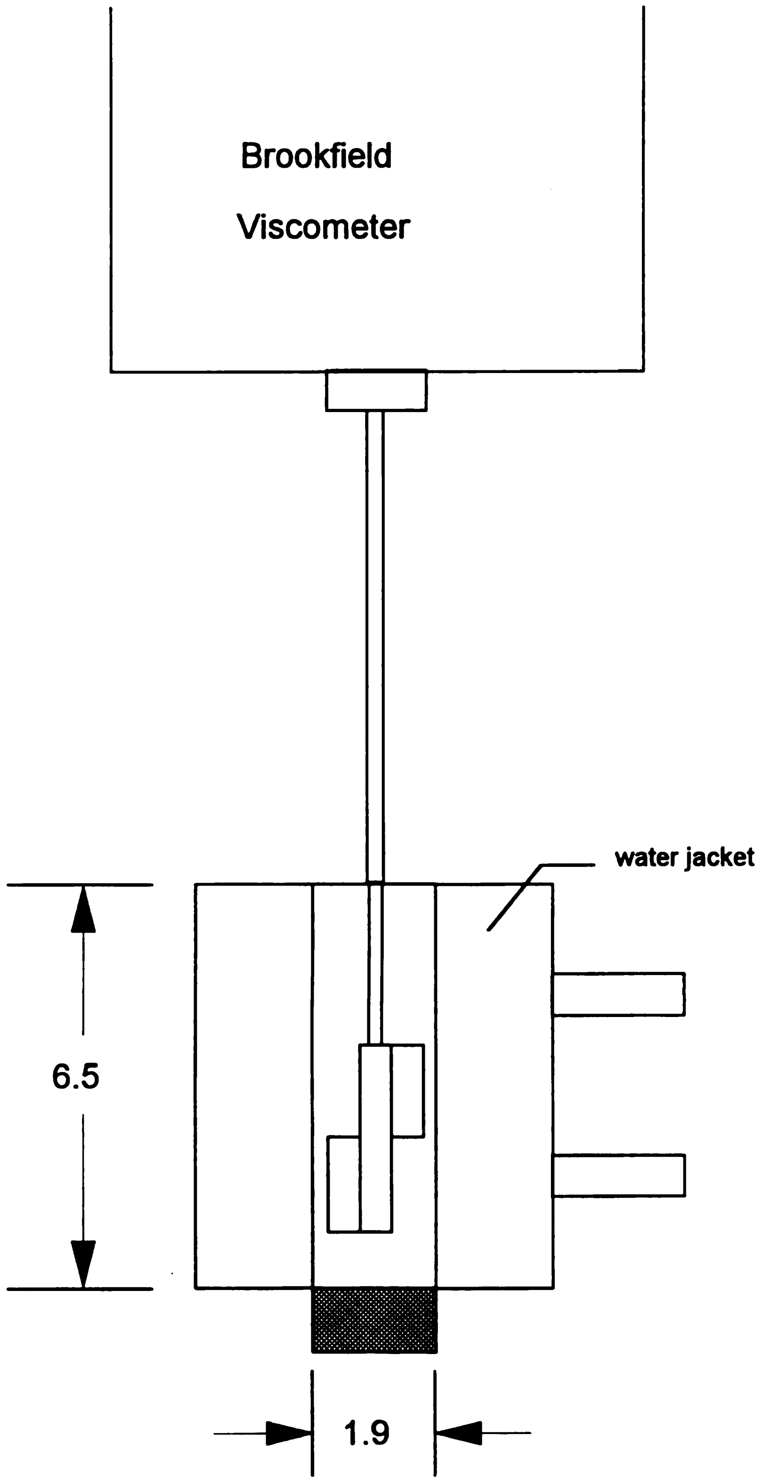

Figure 3.4. Schematic diagram of a Brookfield viscometer equipped with a small sample adapter and flag impeller (all dimensions in cm).

3.3.2. Fluid Foods - Mixer Viscometry

Fluid foods were subjected to the same testing procedure as the reference fluids. Using the small sample adapter, each sample was presheared prior to experimental data collection. Brookfield torque values and temperatures were recorded at various impeller speeds. This procedure was repeated twice with new samples. Due to the torque limitations with the 1/2 RV viscometer when testing some foods, the RV viscometer was used when a higher torque capacity was needed.

Fluid foods were also tested using the Haake RS-100. The 3-step protocol for these experiments remained the same:

1. Shear rate ramp from 0.15 to 30 s^{-1}.
2. Shear rate ramp from 30 to 0.15 s^{-1}.
3. Steady state shear rate ramp from 0.15 to 30 s^{-1}.

Two replications were completed for each sample.

3.3.3. Fluid Foods - Mitschka Method

A 600 ml low form Griffin beaker served as the sample vessel for fluid foods when collecting data to evaluate the Mitschka method. The food was a uniform temperature throughout the sample. Both the Brookfield 1/2 RV and RV viscometers were used during testing. Together, they provided a large torque range allowing several spindles to be used for each sample.

The disc was immersed into the sample at an angle to avoid air pocket formation on the underside to the disc. Before rotation, the disc was centered in the beaker. For disc spindles # 2 - 6 the immersion depth was 4.9 cm. The depth for spindle # 1 and # 7 was 6.1 cm and 6.0 cm, respectively. Immersion depths follow Brookfield recommendations. No guard leg was used for any test. Prior to collecting data, samples were subjected to preshearing by rotating the spindle at the highest speed possible until steady state conditions were met. Formal data collection immediately followed. Torque was recorded at various rotational speeds (0.5 rpm to 100 rpm) for each spindle. A temperature reading was taken at the beginning and end of each test.

Fluid foods were also tested using the Haake RS-100. Due to the shear rates predicted by Mitschka (1982) caused by the disc spindles, the shear rate range covered by the Haake RS-100 was increased from the range covered in the mixer viscometry research. The 3-step protocol was:

1. Shear rate ramp from 0.15 to 40 s^{-1}.
2. Shear rate ramp from 40 to 0.15 s^{-1}.
3. Steady state shear rate ramp from 0.15 to 40 s^{-1}.

Two replications were completed for each sample.

3.4. Data Analysis

3.4.1. Reference Fluids to Determine Mixer Viscometry Constant (k')

Reference fluid data obtained from the Brookfield viscometer using a small sample adapter with the flag impeller and Haake RS-100 (to determine absolute rheological properties) were used to determine k' for both the slope and matching viscosity methods. Raw data gathered from the Brookfield viscometer included percent torque and impeller rotational speed. The torque value was calculated as

$$M = \text{maximum torque capacity (dial reading)}/100 \qquad [3.1]$$

where the maximum torque capacity depends on the viscometer model. Rotational speed (N) in rpm was converted into angular velocity (Ω) in rad/s by the following formula:

$$\Omega = 2\pi(N)/60 \qquad [3.2]$$

Rheograms of shear stress versus shear rate were obtained from steady shear rate data using the Haake RS-100. Rheological parameters, K and n for power law fluids and μ for Newtonian fluids, were determined by linear regression analysis of the data over a shear rate range of 0.15 to 30 s^{-1}.

3.4.1.2. Slope Method

Eq. [2.10] established the relationship between the experimental data and k'. Plotting $\log_{10}\left(M/\left(Kd^3\Omega^n\right)\right)$ versus (1-n) of the several reference fluids gives a slope equal to $-\log_{10}(k')$. Linear regression analysis of the Brookfield data was performed to determine the slope. K and n values were determined from the Haake RS-100 data, and the M and Ω values were obtained from the Brookfield viscometer. Reference fluids provided a wide range of n values.

3.4.1.3. Matching Viscosity Method

Prior to determining k', the matching viscosity method requires that A be determined. Simplifying the power number and Reynolds number relationship gave

$$\frac{M}{d^3\Omega} = A\mu \qquad [2.5]$$

Plotting Newtonian reference fluid data as $\left(M/d^3\Omega\right)$ versus μ results in a slope equal to A. Once A is known, the matching viscosity assumption ca

d

can be applied and apparent viscosity is calculated from the Brookfield data as

$$\eta = \frac{M}{Ad^3\Omega} \qquad [2.11]$$

Shear rate is a function of η, K, and n:

$$\dot{\gamma}_a = \left[\frac{\eta}{K}\right]^{\frac{1}{n-1}} \qquad [2.12]$$

where K and n are determined from the Haake RS-100 data. k', found for each power law reference fluids is simply

$$k' = \dot{\gamma}_a / \Omega \qquad [2.13]$$

Plotting k' versus Ω reveals any dependence, if present, of flow behavior index on k'. Through linear regression analysis k' may be characterized as a function of Ω.

3.4.3. Fluid Foods - Mixer Viscometry

Fluid food behavior was characterized as a rheogram of apparent viscosity versus shear rate. Separate curves were given to represent results from the slope method, matching viscosity method, and Haake RS-100 data. Apparent viscosities for both the slope and matching viscosity methods were calculated from Eq. [2.11]. An average shear rate was calculated as

$$\dot{\gamma}_a = k'\Omega \qquad [2.8]$$

where k' was determined separately for the slope and matching viscosity methods. Apparent viscosity results at several shear rates from the slope method, matching viscosity method, and Haake RS-100 were compared. Flow behavior indexes calculated from torque versus angular velocity daa were also compared.

3.4.3. Fluid Foods - Mitschka Method

Rheograms of apparent viscosity versus shear rate were made to evaluate the accuracy of the Mitschka method for determining fluid food properties. The flow behavior index (n) of sample food is the slope of a plot of $\log_{10}(\sigma_a)$ versus $\log_{10}(N)$ where σ_a (in Pa s) is

$$\sigma_a = k_{\alpha\sigma}(\text{dial reading}) \quad [2.20]$$

$k_{\alpha\sigma}$,a function of the spindle used, is found in Table 1.1. Once n is known, shear rate may be calculated as

$$\dot{\gamma}_a = k_{N\gamma}(N) \quad [2.21]$$

$k_{N\gamma}$, a function of spindle and n, is found in Table 1.1. Linear interpolation was performed to calculate $k_{N\gamma}$ numbers that fell between given values of n (0.1, 0.2, 0.3 ... 1.0). Apparent viscosity is simply

$$\eta = \sigma_a/\dot{\gamma}_a \quad [2.22]$$

All values of apparent viscosity and shear rate data pairs per spindle were plotted together and one power law curve was fit. The resulting curve from the Mitschka method and the Haake RS-100 were compared.

Chapter 4

Results and Discussion

4.1. Mixer Viscometry Constant (k')

4.1.1. Slope Method

To determine the value of k' by the slope method, Eq. [2.10] was employed and a plot of $\log_{10}\left(M/\left(Kd^3\Omega^n\right)\right)$ versus (1-n) was constructed (Fig. 4.1). k' is determined from the slope:

$$k' = 2.92 \text{ rad-1}$$

The data showed considerable scattering. This trend was consistent with previous research by Rao and Cooley (1984) and Rieger and Novak (1973). From Fig. 4.1, it can be seen that the spread was in part due to the various rotational speeds. By linearly regressing the curves for each rotational speed, the slopes for speeds 1 to 100 rpm were similar in magnitude.

4.1.2. Matching Viscosity Method

A, the constant resulting from system geometry, was determined from Eq. [2.6] as the slope of $(M/(\Omega d^3))$ versus μ of the Newtonian reference fluids (Fig. 4.2). Linear regression revealed that A equaled 4.84 (dimensionless) with r2 > 0.99.

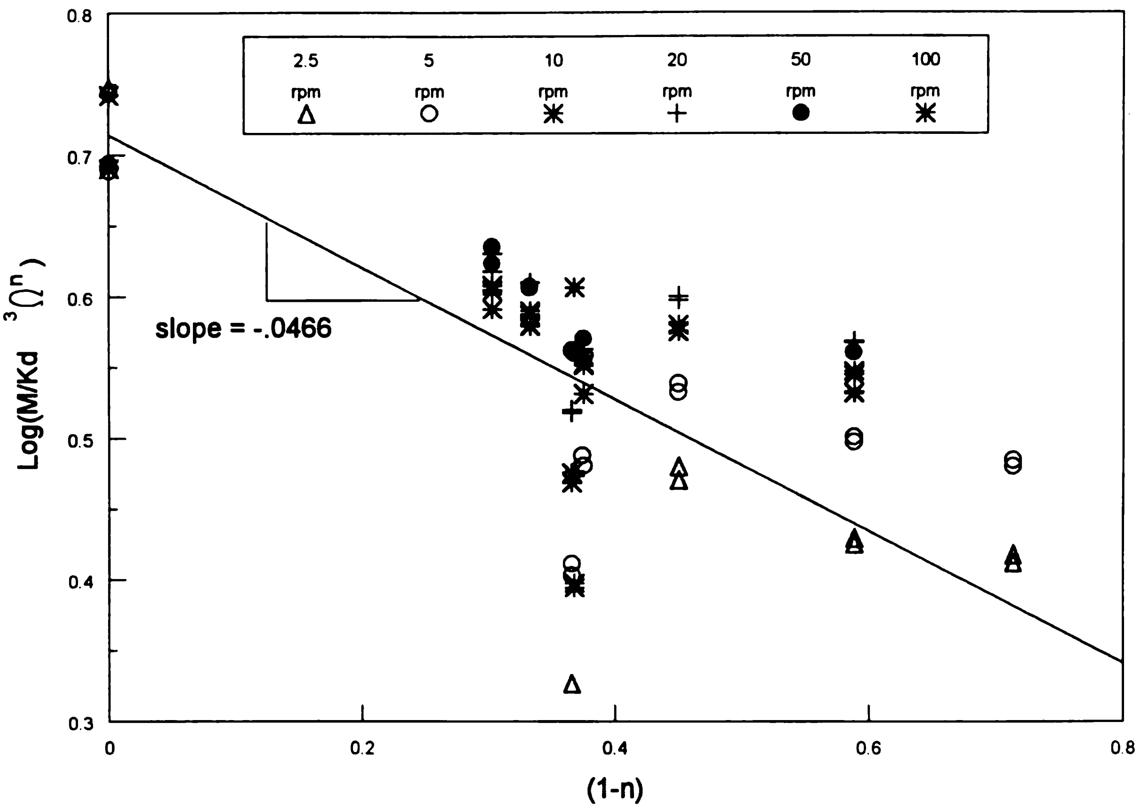

Figure 4.1. $\mathrm{Log}_{10}\left(M/\left(Kd^3\Omega^n\right)\right)$ versus (1-n) for the determination of k' by the slope method.

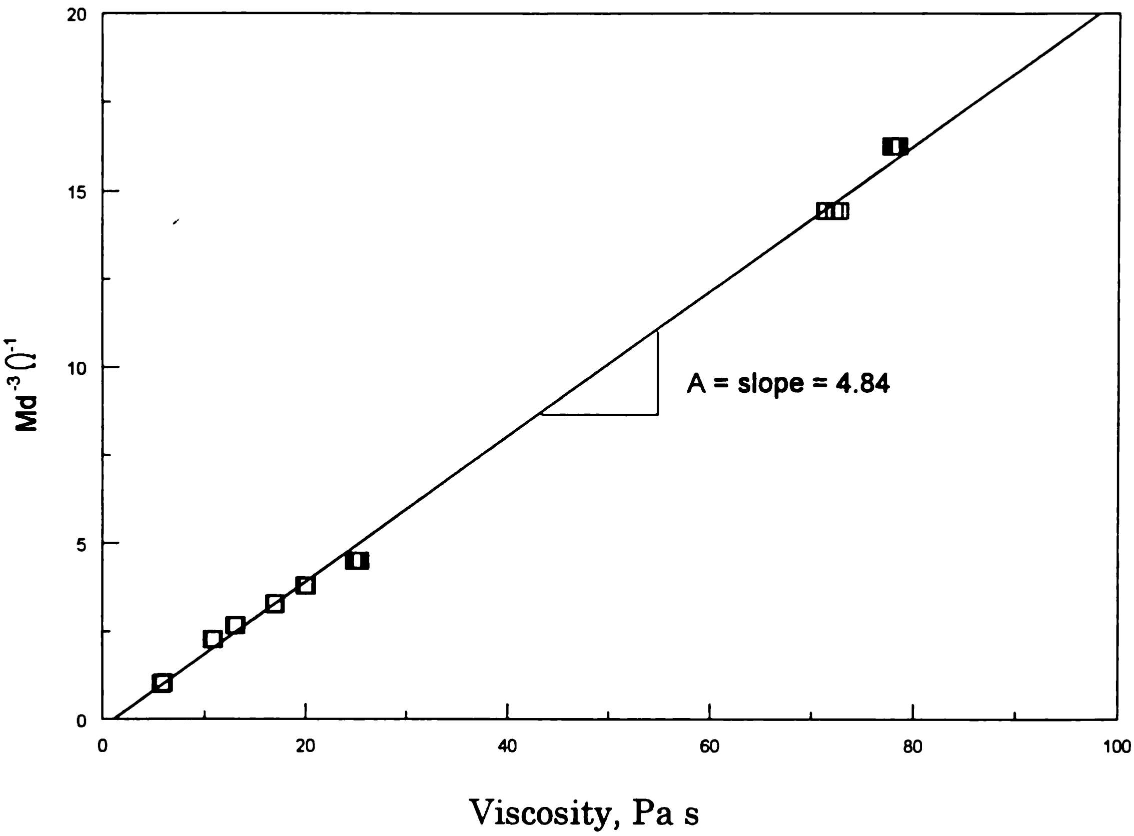

Figure 4.2. $M/(\Omega d^3)$ versus μ for the determination of A.

The matching viscosity method allows k' to be evaluated as a function of rotational speed (Ω) and flow behavior index (n). Fig. 4.3 showed that k' was not a strong function of n but highly influenced by Ω. At rotational speeds of 5.23 and 10.47 rad/s (50 and 100 rpm), k' was nearly constant; however, at speed less than 5.23 rad/s, k' increased rapidly and more data scattering occurred. Castell-Perez and Steffe (1990) observed similar trends.

To characterize k' as a function of omega, linear regression was performed. Several curve fits (exponential, logarithmic, and power law) were attempted. An exponential fit did not sufficiently account for the increasing k' values at low rotational speeds. The logarithmic and power law fits better reflected the increasing k' trend at low rotational speeds. Both curves underestimated k' at 10.47 rad/s (100 rpm); however, the power law curve was more accurate. Hence, k' was determined from a power law fit of the data:

$$k' = 2.54(\Omega)^{-0.176} \qquad [4.1]$$

The curve given by Eq. [4.1] is plotted in Fig. 4.3.

4.2 Fluid Foods - Mixer Viscometry

Apparent viscosities were plotted against average shear rates for each fluid food (Fig. 4.4 - 4.9). Visually, the resulting curve from the slope and matching viscosity method compared well with the curve (considered the true flow curve) from the Haake RS-100. Table 4.1 compares the mixer viscometry apparent viscosities with the Haake apparent viscosities. The apparent viscosities from the matching viscosity method were closer to the Haake results than the slope

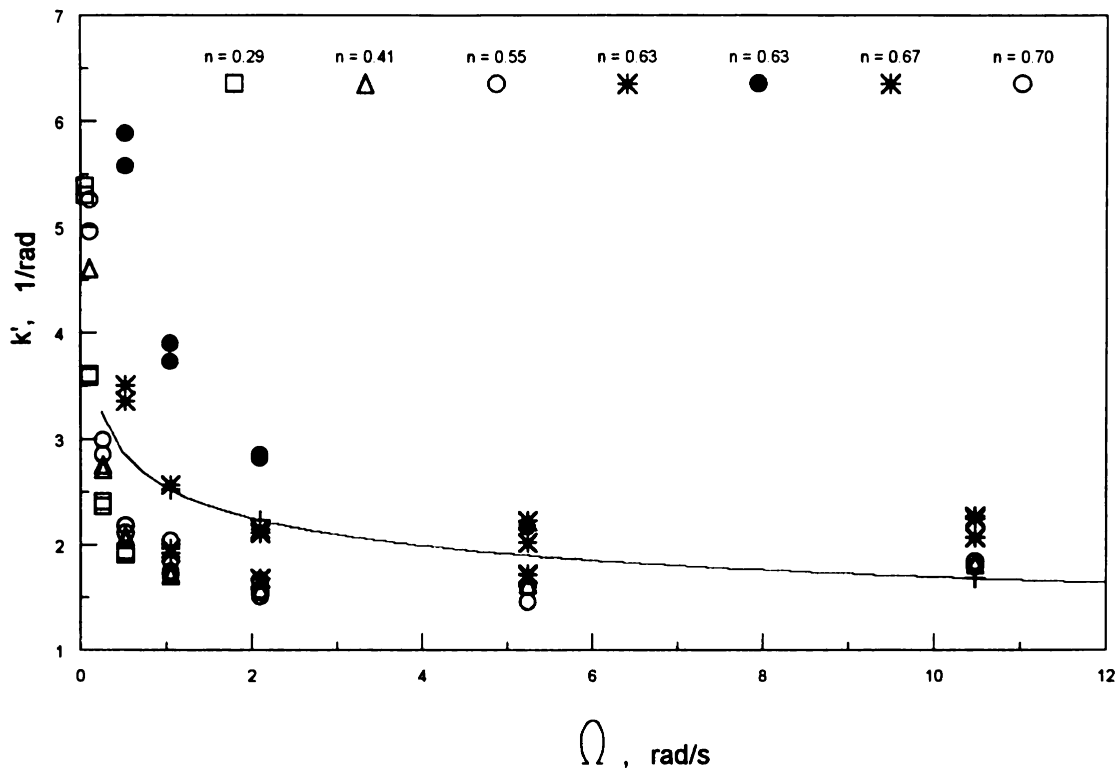

Figure 4.3. k' from the matching viscosity method versus Ω for determining the influence of Ω on k'.

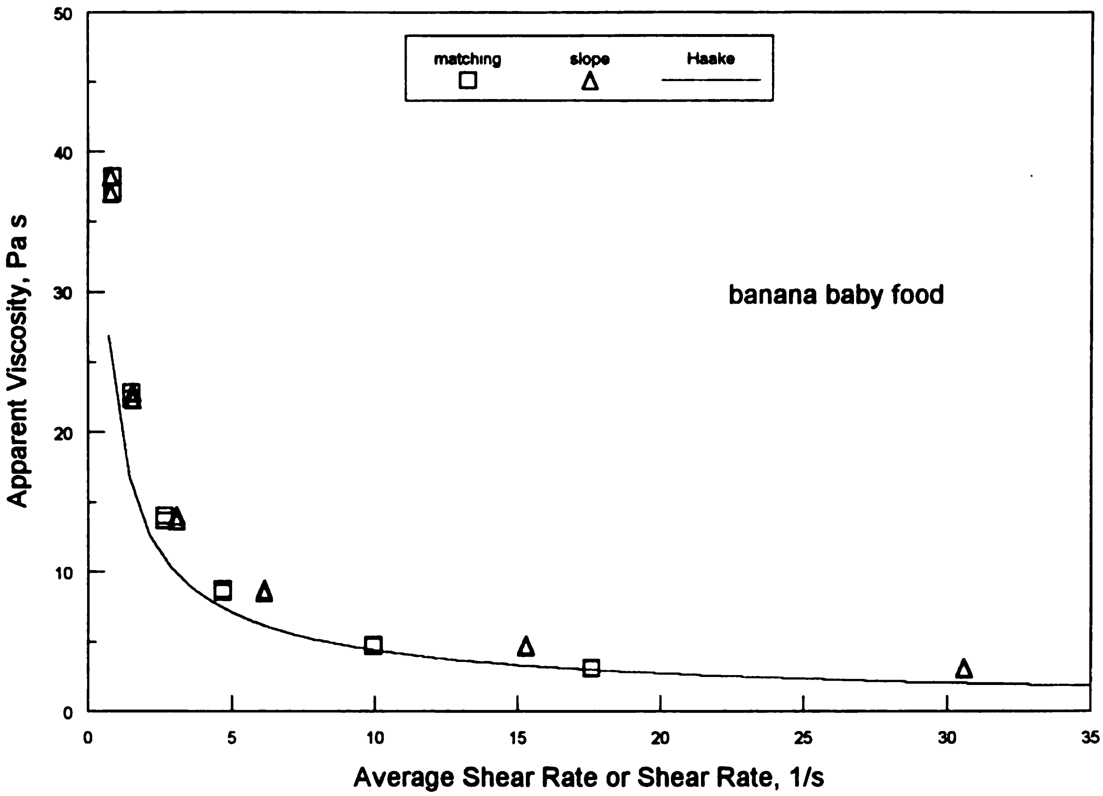

Figure 4.4. Apparent viscosity versus average shear rate (slope and matching methods) or shear rate (Haake) for banana baby food at 29.2 °C.

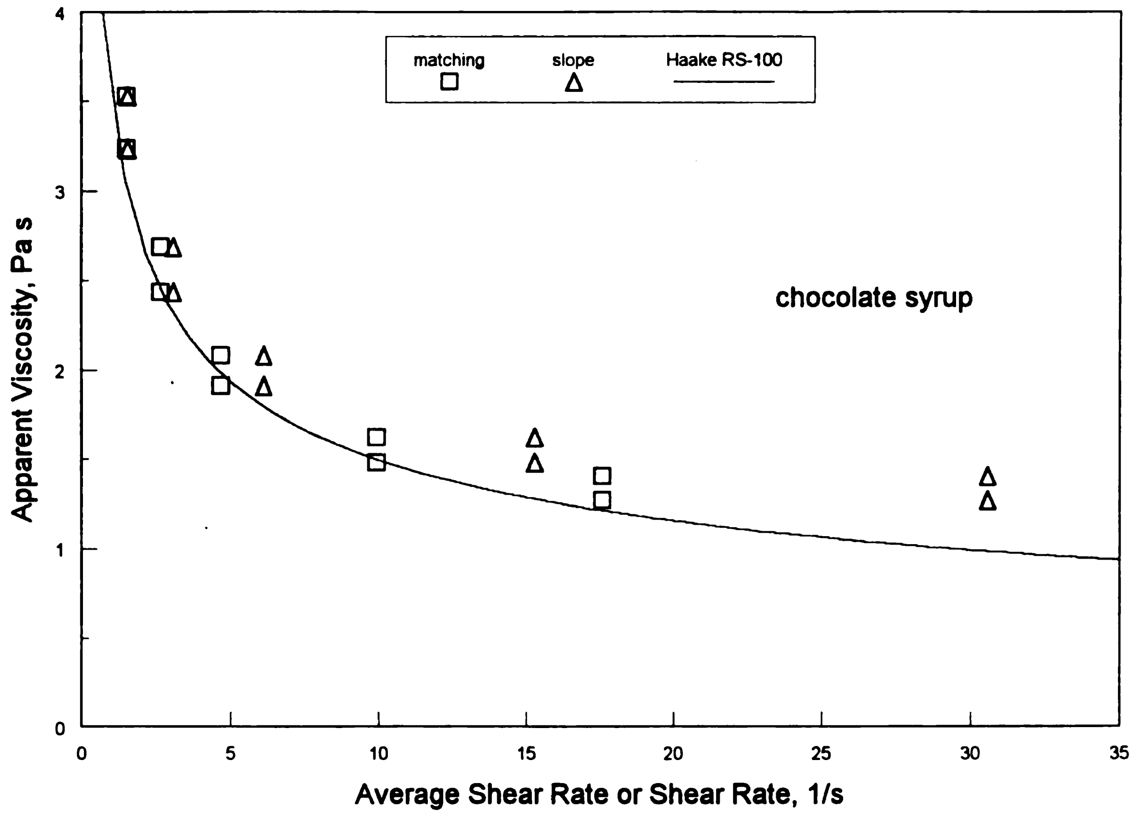

Figure 4.5. Apparent viscosity versus average shear rate (slope and matching methods) or shear rate (Haake) for chocolate syrup at 20.8 °C.

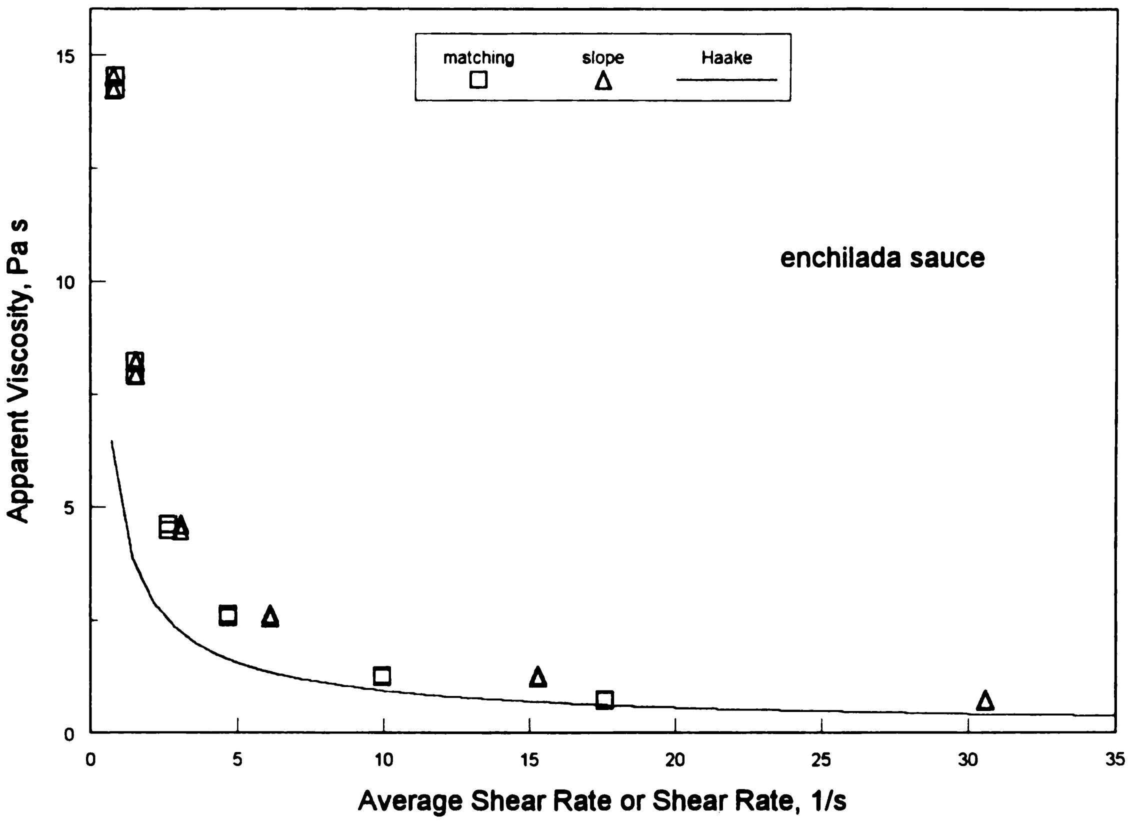

Figure 4.6. Apparent viscosity versus average shear rate (slope and matching methods) or shear rate (Haake) for enchilada sauce at 24.7 °C.

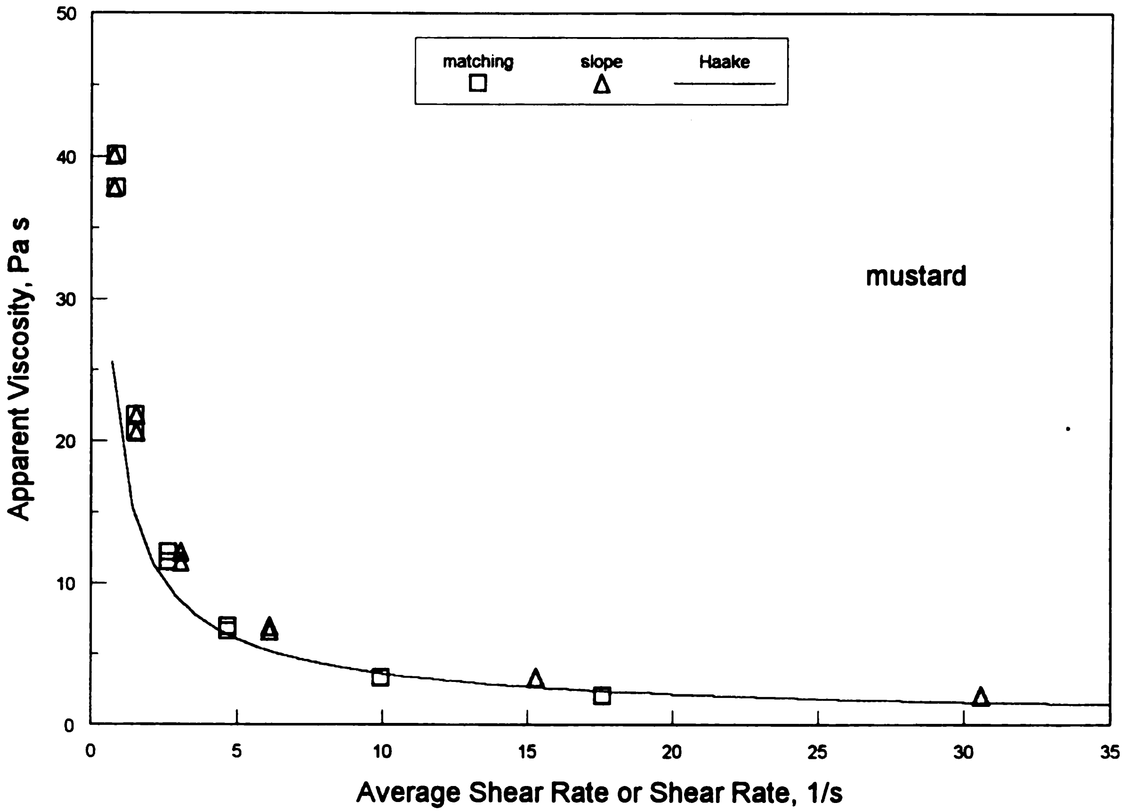

Figure 4.7. Apparent viscosity versus average shear rate (slope and matching methods) or shear rate (Haake) for mustard at 23.4 °C.

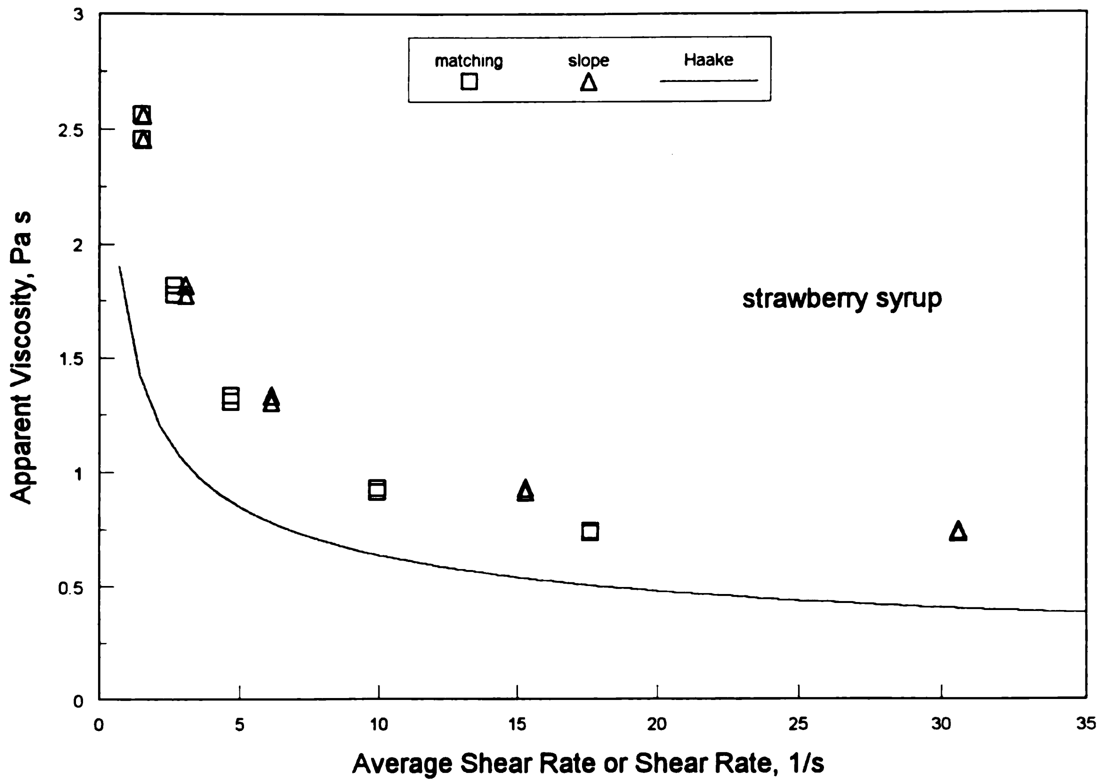

Figure 4.8. Apparent viscosity versus average shear rate (slope and matching methods) or shear rate (Haake) for strawberry syrup at 23.4 °C.

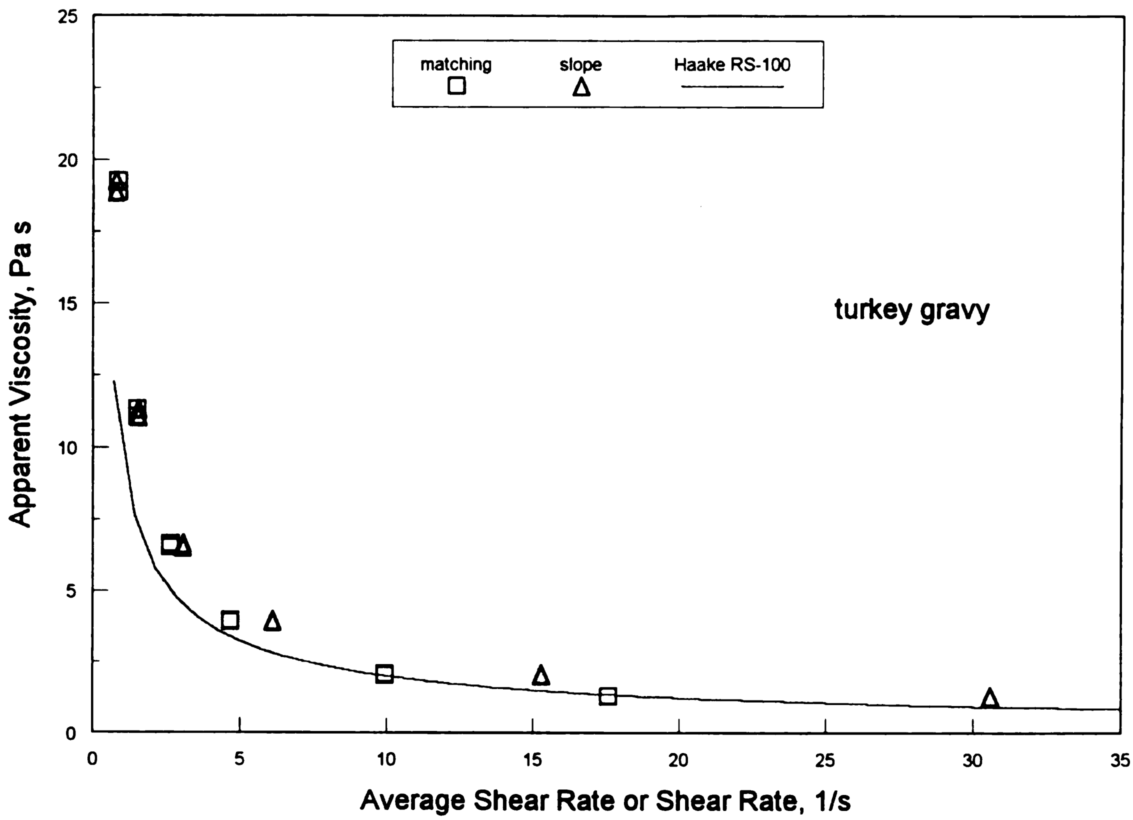

Figure 4.9. Apparent viscosity versus average shear rate (slope and matching methods) or shear rate (Haake) for turkey gravy at 26.5 °C.

Table 4.1. Comparing apparent viscosities at various shear rates obtained using the small sample adapter and flag impeller, or the Haake rheometer.

Fluid Food	Method	Apparent Viscosity, Pa s			
		2.5*	5*	10*	15*
banana baby food	slope	16.30	10.19	6.4	4.84
	matching	14.87	8.41	4.76	3.41
	Haake	11.39	7.08	4.40	3.33
chocolate syrup	slope	2.77	2.23	1.80	1.59
	matching	2.66	2.05	1.58	1.35
	Haake	2.28	1.82	1.46	1.28
enchilada sauce	slope	5.43	3.11	1.78	1.28
	matching	4.87	2.47	1.25	0.84
	Haake	2.59	1.56	0.94	0.70
mustard	slope	14.41	8.30	4.78	3.46
	matching	12.94	6.62	3.38	2.29
	Haake	10.11	6.05	3.62	2.68
strawberry syrup	slope	1.98	1.49	1.12	0.95
	matching	1.87	1.33	0.94	0.77
	Haake	1.13	0.85	0.64	0.54
turkey gravy	slope	7.81	4.71	2.85	2.12
	matching	7.08	3.84	2.08	1.45
	Haake	5.20	3.23	2.01	1.52

* Shear Rate, 1/s

Table 4.2. Flow behavior indexes for fluid foods using the Brookfield small sample adapter and flag impeller, or Haake data.

Fluid Food	Method	n* (-)	r2
banana baby food	mixing	0.32	1.00
	Haake	0.31	0.99
chocolate syrup	mixing	0.69	0.97
	Haake	0.68	0.97
enchilada sauce	mixing	0.19	1.00
	Haake	0.27	1.00
mustard	mixing	0.21	1.00
	Haake	0.26	0.99
strawberry syrup	mixing	0.59	0.99
	Haake	0.59	0.99
turkey gravy	mixing	0.27	0.99
	Haake	0.32	0.99

$* n = d(\log_{10} M)/d(\log_{10} \Omega)$

method. Although, both methods produced acceptable results from a practical stand point.

4.3. Recommendation for Quality Control Tests on Food

Using the small sample adapter in conjunction with the flag impeller possess four major advantages over the traditional Brookfield spindles. 1) A small sample (18 ml) may be evaluated; 2) Sample temperature is easily controlled; 3) Problem associated with slip can be eliminated; 4) Problems associated with particle settling can be greatly reduced. Considering the research conducted in this study, both the slope method and matching viscosity method may be successfully used to determine apparent viscosities for fluid foods. In addition, the flow behavior index may be accurately determined from regression of torque versus angular velocity curve plotted on log-log scales. Due to the simplicity of the slope method (constant k' value), it is recommended for calculating average shear rates in simple quality control tests.

To establish a quality control program for a power law food product, an acceptable product must first be analyzed using the small sample adapter and flag impeller. Torque readings are taken at two impeller speeds. Apparent viscosity is calculated as $\eta = M/\left(Ad^3\Omega\right)$ and average shear rate is calculated as $\dot{\gamma}_a = 2.92(\Omega)$. From this data an apparent viscosity mean and standard deviation is determined at each average shear rate. This initial step sets an allowable window for acceptable measurements.

Now, an unknown sample is evaluated. Torque readings are taken at two different speeds. Then, the apparent viscosities values are compared with the acceptable apparent viscosity range at the same shear rates. On the basis of these findings, the product is either accepted or rejected. The initial speed (or average shear rate) must result in a sufficiently large apparent viscosity value compared to the second speed. Otherwise, the shear-thinning behavior may not be taken into account. For example, if the two chosen speeds were at taken at 50 and 100 the apparent viscosity measurements may be very similar. As a results, the extent of shear- thinning may be ignored. The magnitude of the flow behavior index, calculated from two apparent viscosity values, may also be used as a quality control criterion.

The above quality control tests is based on apparent viscosity values. An advantage of this protocol is that the degree of shear-thinning is implicitly incorporated into the test by taking two measurements at different speeds. For example, if n was less than acceptable for a sample the apparent viscosity may pass through the first range and pass above the second range; thus, the sample would be rejected. A single point test cannot reveal the true behavior of a non-Newtonian sample.

4.4. Fluid Food - Mitschka Method

The Mitschka method is an approximation to characterize the rheological behavior of power law fluids using the Brookfield viscometer and disc spindles. Table 4.3 lists the n values determined from Brookfield data for fluid foods evaluated in this study. Comparing the various n values shows good agreement was found within each sample

Table 4.3. Flow behavior indexes from the slope of $\log_{10}$(N) versus $\log_{10}$(shear stress) for various spindles.

spindle	banana puree	Catalina dressing	chocolate syrup	pancake syrup	enchilada sauce
2	N/A	N/A	0.44	0.48	0.20
3	0.23	0.18	0.53	0.50	0.20
4	0.22	0.19	0.58	0.50	0.18
5	0.22	0.20	0.64	0.51	0.19
6	0.22	0.20	N/A	N/A	N/A

with the exception of chocolate syrup where n ranged from 0.44 to 0.64. During data collection of the chocolate syrup using the Brookfield, the percent torque fluctuated and continued to decrease. A stable value was not obtained for nearly 3 minutes after the impeller speed was set. Generally, this is a characteristic associated with slip. Violation of the no-slip condition leads to low n values. Based on the amount of surface area of spindle 2 and the low n value, slip may have occurred.

To determine rheological parameters, average shear rate was plotted against apparent viscosity (Fig. 4.10 - 4.14). Data from all spindles were combined. K and n, found by linear regression, are listed in Table 4.4. For all foods, K from the Mitschka and Mitschka approximation method were similar to the Haake RS-100 results. The n values for Catalina Dressing and enchilada sauce were in good agreement with those calculated from the Haake data. The banana puree flow behavior index for the Mitschka method and Mitschka approximation were 0.21 and 0.20, respectively, while the Haake results yielded n = 0.26. Chocolate syrup flow behavior index found using the Mitschka methods were significantly lower than the Haake RS-100 value.

Rheological parameters of the power law model found by regression analysis are linked. Hence, to avoid examining K and n separately, the apparent viscosities at several shear rates may be compared (Table 4.5). The apparent viscosities found by the Mitschka method were very close to the apparent viscosities found by the Mitschka approximation. Both methods also compared well to the Haake RS-100 results. Clearly, the Mitschka method generates

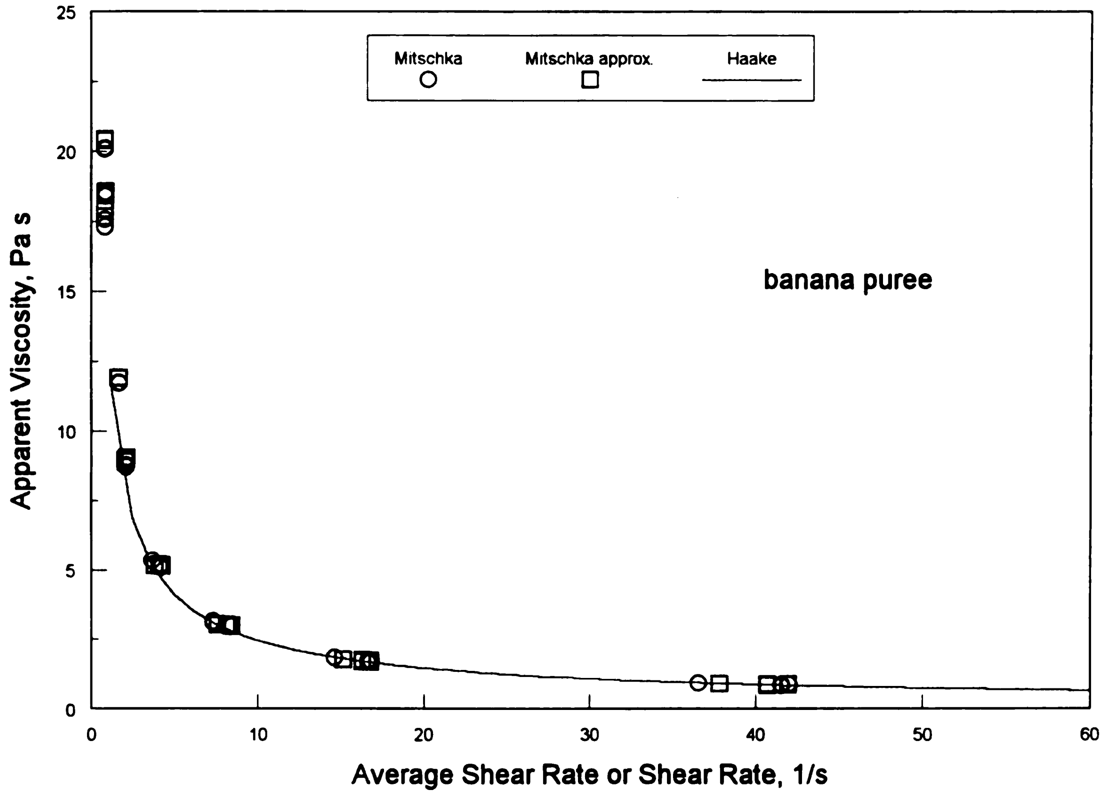

Figure 4.10. Apparent viscosity versus average shear rate (Mitschka and Mitschka approximation) or shear rate (Haake) for banana puree at 24.0 °C.

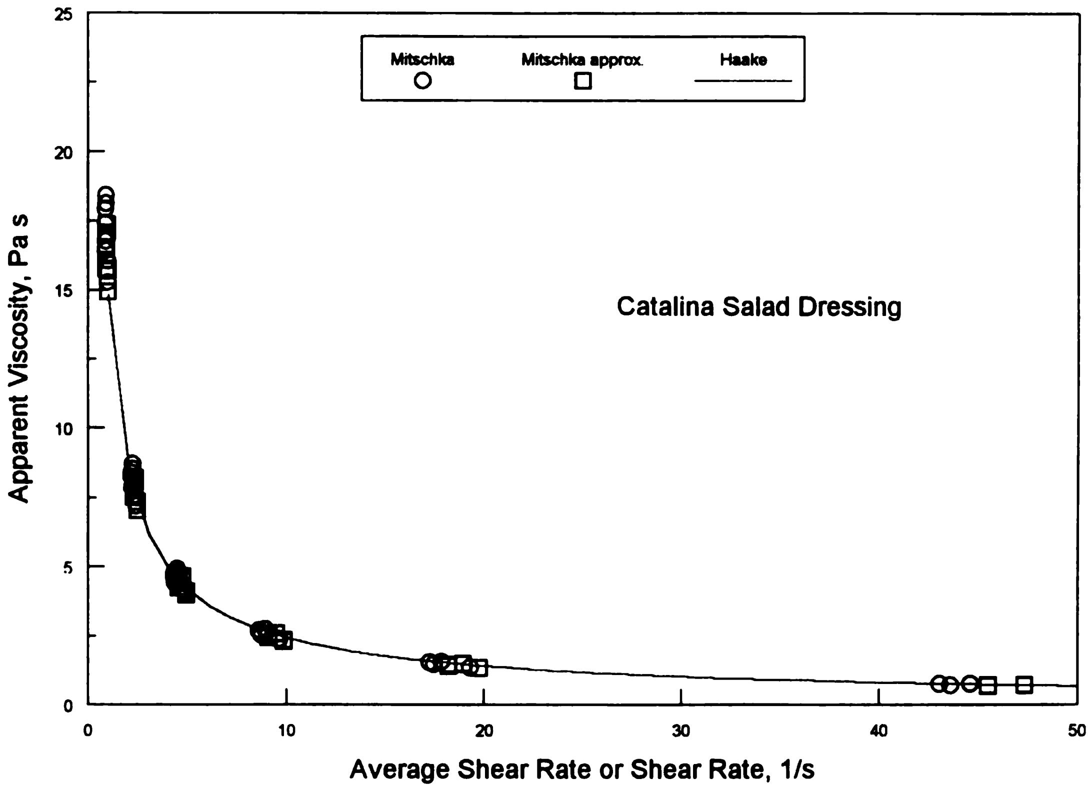

Figure 4.11. Apparent viscosity versus average shear rate (Mitschka and Mitschka approximation) or shear rate (Haake) for Catalina salad dressing at 23.8 °C.

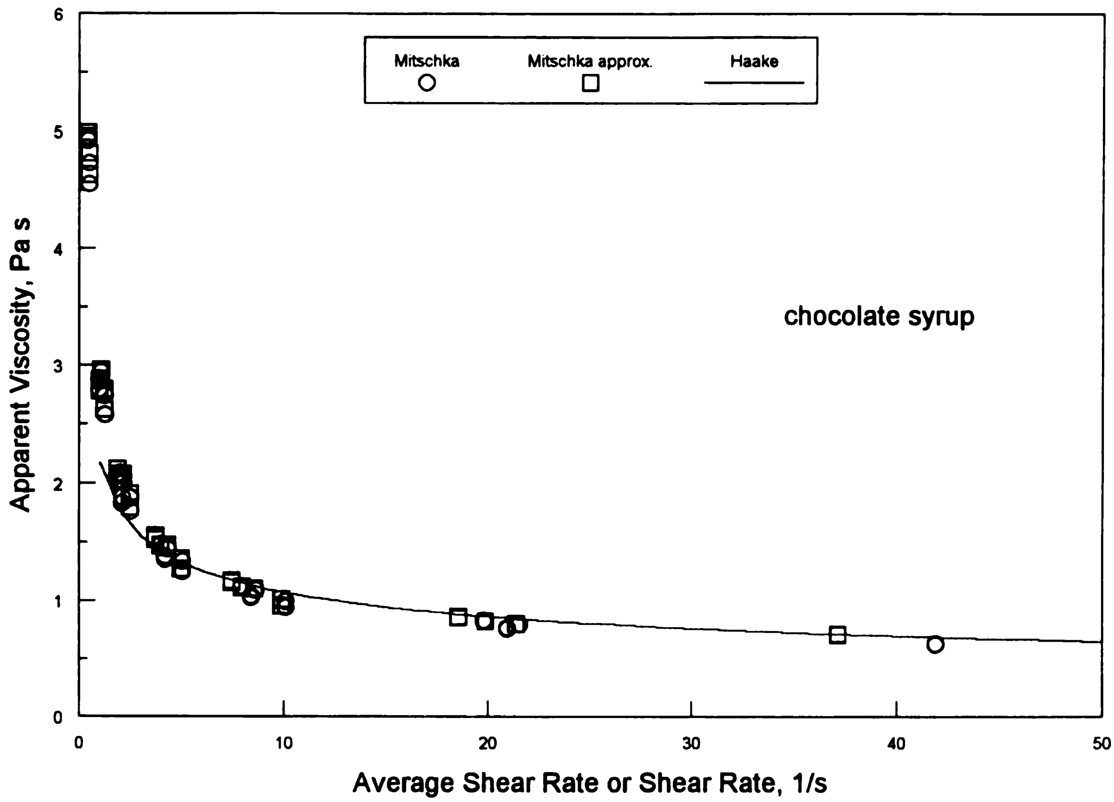

Figure 4.12. Apparent viscosity versus average shear rate (Mitschka and Mitschka approximation) or shear rate (Haake) for chocolate syrup at 24.0 °C.

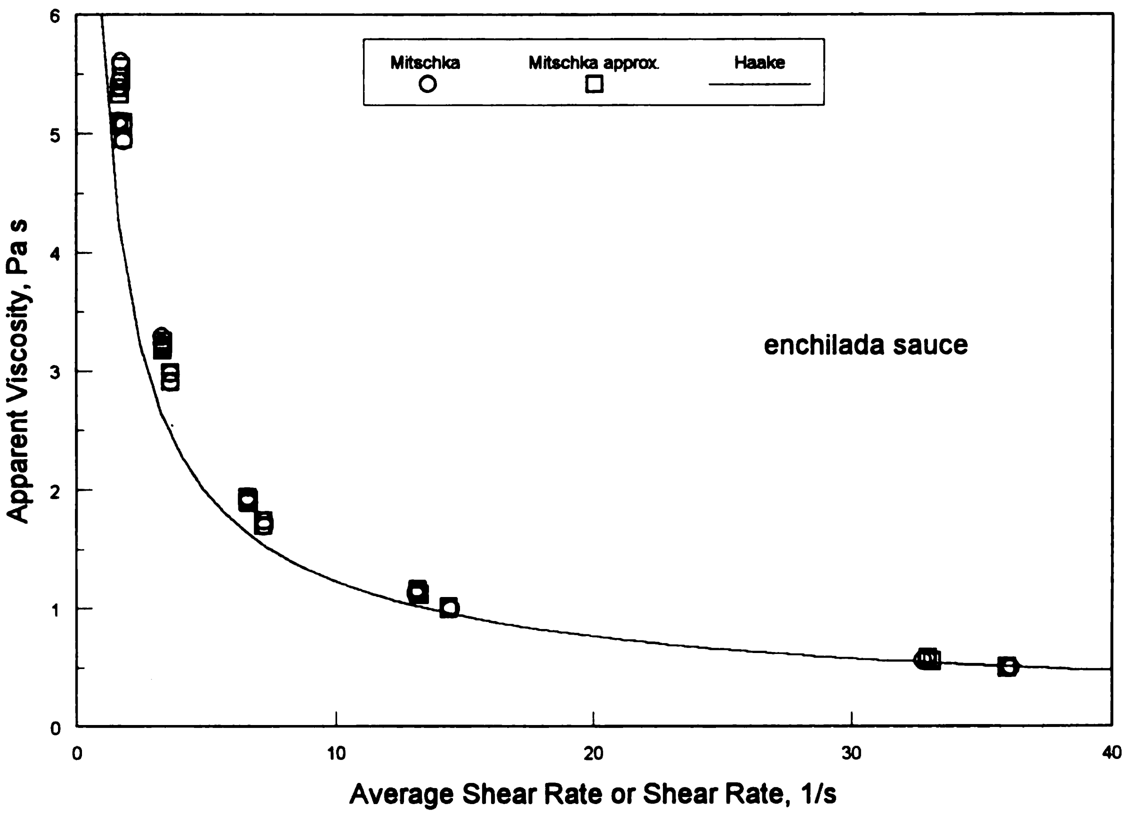

Figure 4.13. Apparent viscosity versus average shear rate (Mitschka and Mitschka approximation) or shear rate (Haake) for enchilada sauce at 26.0 °C.

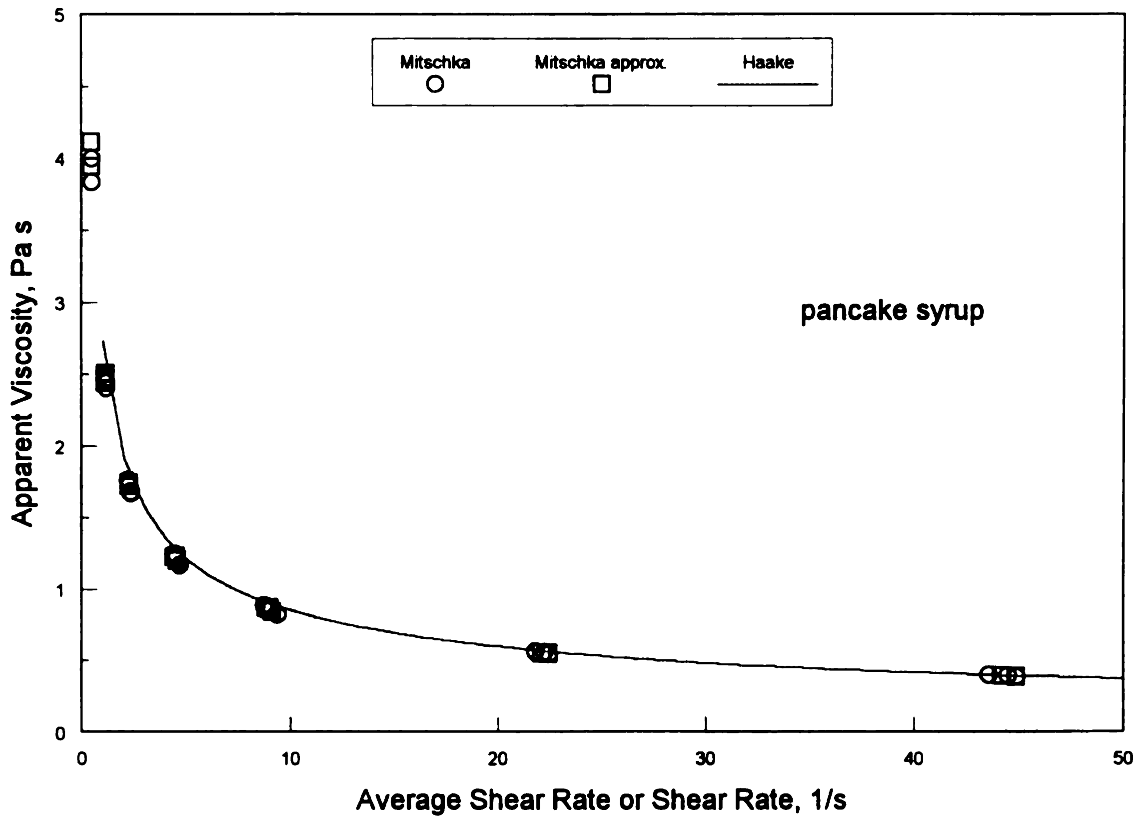

Figure 4.14. Apparent viscosity versus average shear rate (Mitschka and Mitschka approximation) or shear rate (Haake) for pancake syrup at 23.1 °C.

Table 4.4. Rheological parameters for food obtained using the Brookfield spindles.

Fluid Food	Method	K (Pa s^n)	n (-)	r^2
banana puree	Mitschka	16.09	0.21	1.00
	Mitschka approximation	16.13	0.20	1.00
	Haake RS-100	13.41	0.26	1.00
Catalina dressing	Mitschka	15.41	0.19	1.00
	Mitschka approximation	15.27	0.19	1.00
	Haake RS-100	15.04	0.21	1.00
chocolate syrup	Mitschka	2.91	0.55	0.98
	Mitschka approximation	2.94	0.56	0.98
	Haake RS-100	2.19	0.69	0.92
enchilada sauce	Mitschka	7.23	0.28	0.99
	Mitschka approximation	5.96	0.31	1.00
	Haake RS-100	7.22	0.28	0.99
pancake syrup	Mitschka	2.6	0.5	1.00
	Mitschka approximation	2.62	0.5	1.00
	Haake RS-100	2.76	0.49	0.99

Table 4.5. Apparent viscosities at various shear rates for Brookfield spindles.

Food	Method	Apparent Viscosity, Pa s			
		5*	10*	20*	40*
banana puree	Mitschka	4.51	2.61	1.51	0.87
	Mitschka approximation	4.45	2.56	1.47	0.84
	Haake RS-100	4.08	2.44	1.46	0.87
Catalina dressing	Mitschka	4.18	2.39	1.36	0.78
	Mitschka approximation	4.14	2.37	1.35	0.77
	Haake RS-100	4.22	2.44	1.41	0.77
chocolate syrup	Mitschka	1.41	1.03	0.76	0.55
	Mitschka approximation	1.45	1.07	0.79	0.58
	Haake RS-100	1.33	1.07	0.86	0.70
enchilada sauce	Mitschka	2.27	1.38	0.84	0.51
	Mitschka approximation	2.27	1.38	0.84	0.51
	Haake RS-100	1.96	1.22	0.75	0.48
pancake syrup	Mitschka	1.16	0.83	0.58	0.41
	Mitschka approximation	1.17	0.83	0.58	0.41
	Haake RS-100	1.21	0.85	0.60	0.42

* Shear Rate, 1/s

acceptable apparent viscosites for the purpose of conducting quality control tests on non-time-dependent fluid foods.

Spindle number 1 and 7 are used to measure high and low viscosity fluids, respectively. The foods tested in this study did not create a large enough torque response when using spindle number 7 and caused torque overload when using spindle number 1. However, silicon oils were tested using all spindles (number 1 - 7). Usually, Newtonian viscosities are calculated using Brookfield factors but analyzing Newtonian viscosities by the Mitschka methods is useful when evaluating the accuracy of this method for n values near 1.0. Fig. 4.15 - 4.17 compares the viscosities determined by the Mitschka methods with the Haake viscosities for Newtonian fluids (n=1.0).

From the data presented in Fig. 4.15 and 4.16, Table 4.6 was constructed to compare the percent deviation of the average viscosity from the Mitschka methods to the average viscosity from the Haake RS-100 for Newtonian fluids. The maximum error caused by a single spindle for the Mitschka method was 5.39% occurring with spindle 7. The Mitschka approximation caused errors as large as 28.2 % and 24.8 % when using spindles 1 and 7, respectively.

The discrepancies reported above occurred when measuring Newtonian fluids. $k\alpha\gamma$ from the Mitschka method are only variables used to estimate the average shear rate and the $k\alpha\gamma$ used in the Mitschka approximation are a result of a power law curve fit. When evaluating the usefulness of the Mitschka approximation it may be insightful to examine the deviation from the actual $k\alpha\gamma$ caused by the linear regression. Based on the error shown in Table 4.7 and the percent deviation of the predicted $k\alpha\gamma$ and the actual $k_{\alpha\gamma}$ values given

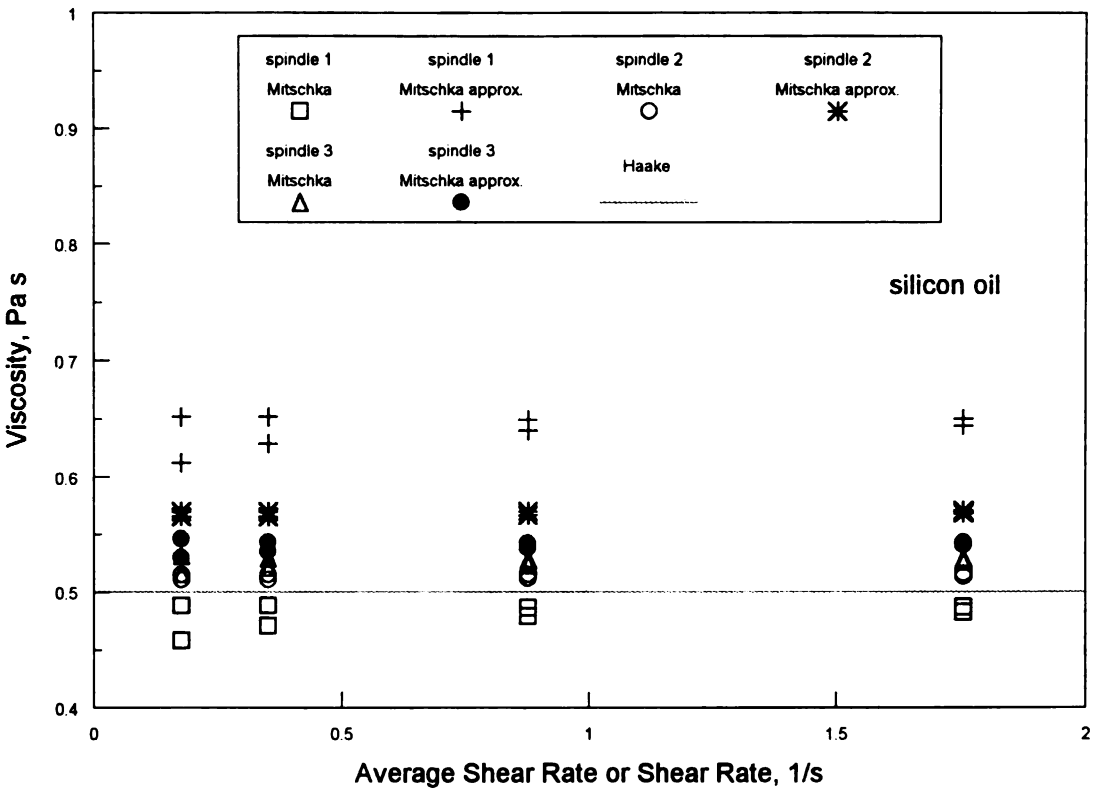

Figure 4.15. Apparent viscosity versus average shear rate (Mitschka and Mitschka approximation) or shear rate (Haake) for silicon oil at 24.0 °C.

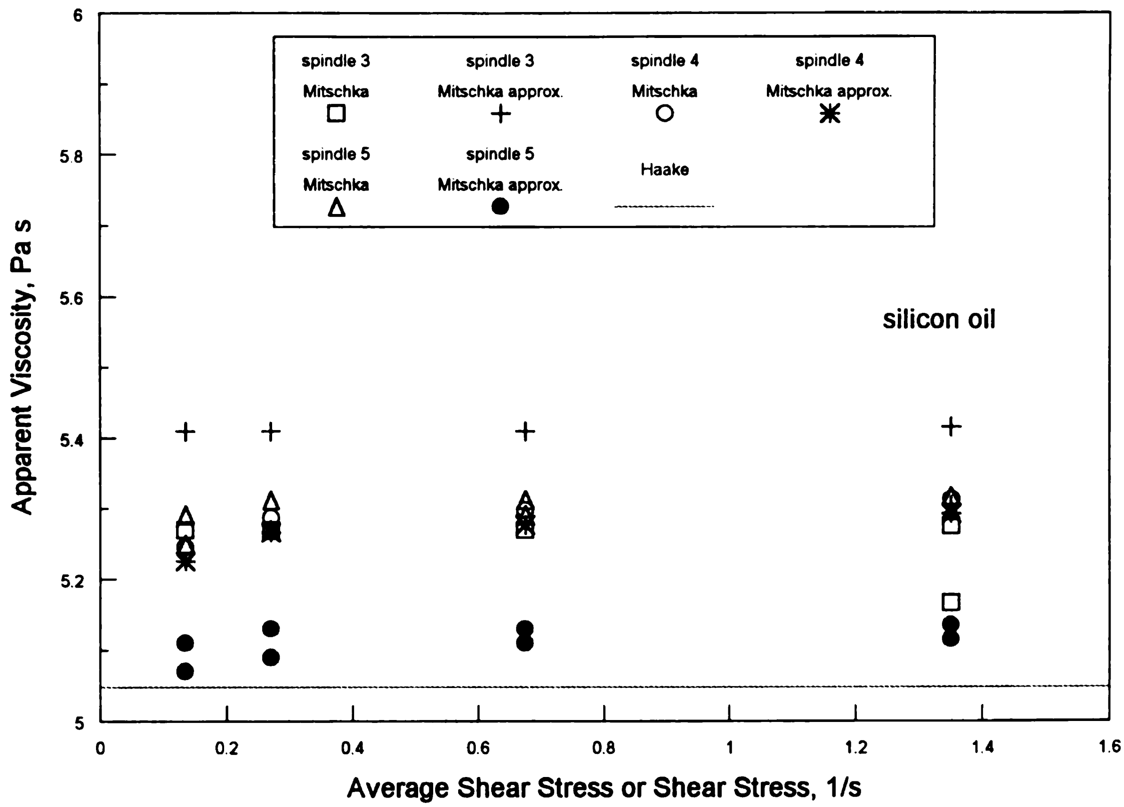

Figure 4.16. Apparent viscosity versus average shear rate (Mitschka and Mitschka approximation) or shear rate (Haake) for silicon oil at 24.0 °C.

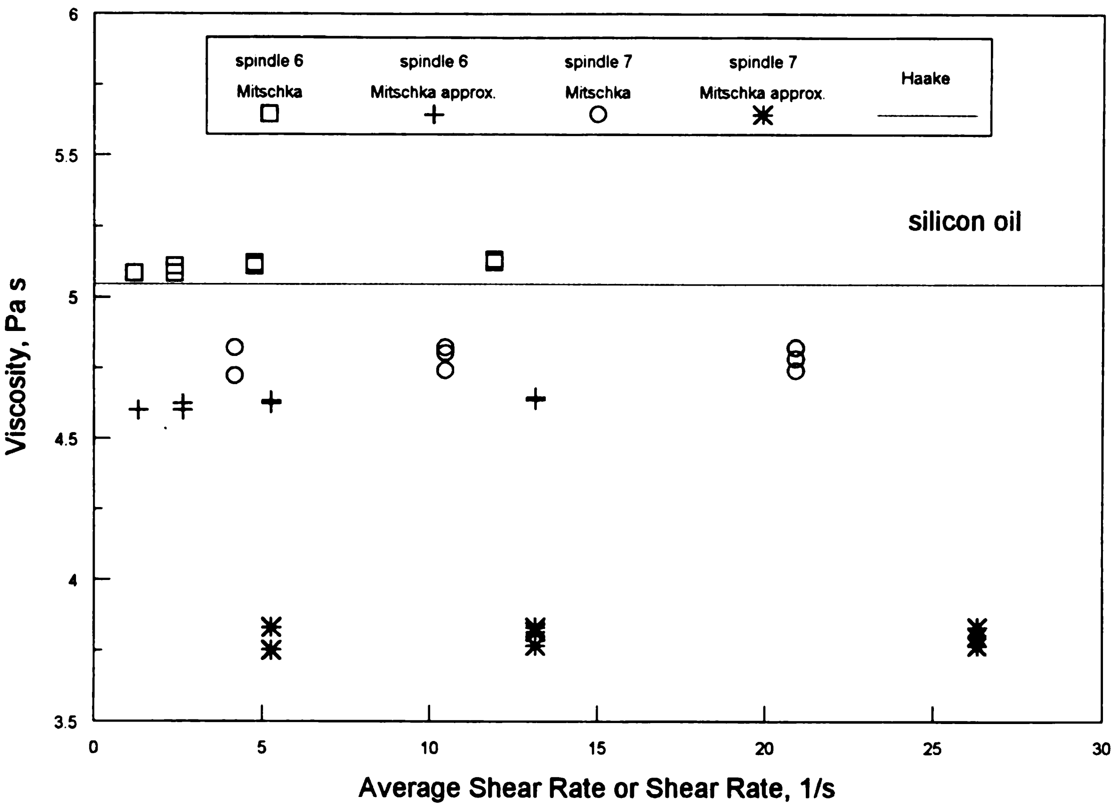

Figure 4.17. Apparent viscosity versus average shear rate (Mitschka and Mitschka approximation) or shear rate (Haake) for silicon oil at 24.0 °C.

Table 4.6. Percent deviation of the Mitschka method and Mitschka approximation from the Haake RS-100 data for silicon oil.

Brookfield spindle	Mitschka method	Mitschka approximation
1	-3.92	28.22
2	2.8	13.6
3*	5.2	8.0
3**	4.16	6.93
4	4.77	4.36
5	4.83	1.25
6	1.17	-8.46
7	-5.39	-24.82

*0.5 Pa s silicon oil
**5.05 Pa s silicon oil

by Mitschka (1982), the approximation is not recommended for spindles 1 and 7. Even though the error caused by the approximation for spindle 6 ranges from -12 % to 5.5 %, the Mitschka approximation could be applied when estimating the shear rate assuming this data is combined with shear rates obtained from other spindles. Spindle 6 was used to measure the properties of banana puree and Catalina Dressing. From the apparent viscosity versus shear rate plots for these samples (Fig. 4.10 and 4.11), any error that may have been caused by the curve fit of $k_{\alpha\sigma}$ versus n was not evident.

Considering the results presented in this study, the application of the Mitschka method to determine rheological properties of power law foods is acceptable. The Mitschka approximation may be used to simplify the determination of shear rate for spindles 2 - 6.

Resulting curves from the Mitschka and Mitschka approximation were compared with the Haake curve. The statistical test employed, described by Neter and Wasserman (1974), determined if the regression lines (Mitschka and Haake or Mitschka approximation and Haake) were equivalent. Results showed that the Mitschka and Mitschka approximation curve were not the same curve as the Haake. Due to the numerous data point collected and the smoothness of fit for the Haake curve and the few point available for the Mitschka method curves, a suitable statistical test was not available to determine if the curves were indeed identical. Clearly, from Fig. 4.10 - 4.14, the curves are sufficiently accurate from a practical sense.

Figure 4.7. Percent error caused by the Mitschka approximation.

	Brookfield Spindle Number						
n (-)	1	2	3	4	5	6	7
0.1	11.3	-7.8	-6.1	-3.9	-0.5	-12.0	24.7
0.2	6.3	-3.8	-3.0	-1.9	-0.3	-6.4	10.7
0.3	5.9	-1.4	-1.4	-1.1	-0.4	-5.5	2.3
0.4	8.0	0.4	-0.6	-0.8	-0.9	-5.6	-3.4
0.5	11.2	2.0	0.0	-0.8	-1.5	-6.2	-8.0
0.6	15.1	3.6	0.5	-0.8	-2.0	-6.9	-11.3
0.7	19.6	5.4	1.1	-0.9	-2.4	-7.8	-14.2
0.8	23.9	6.9	1.5	-0.8	-2.7	-8.4	-16.4
0.9	28.7	8.7	2.0	-0.8	-3.2	-8.8	-18.7
1.0	33.5	10.6	2.3	-0.4	-3.4	-9.5	-20.5

Chapter 5

Summary and Conclusions

A Brookfield viscometer is a relatively inexpensive rheological instrument. However, absolute viscosity measurements can only be made for Newtonian fluids. This research investigated the capability of using the Brookfield viscometer for measuring power law food properties.

Mixer viscometry techniques (slope and matching viscosity method) were applied to the Brookfield small sample adapter and flag impeller. As a result, a method to calculate apparent viscosity and average shear rate was developed. Apparent viscosity for both the slope and the matching viscosity method is calculated as

$$\eta = \frac{M}{Ad^3\Omega}$$

Average shear rate for the slope and matching viscosity method is a function of angular velocity and k':

$$\dot{\gamma}_a = k'(\Omega)$$

where k', from the slope method, is

$$k' = 2.92$$

and, from the matching viscosity method, is

$$k' = 2.54(\Omega)\text{-}0.176$$

Based on the research presented, apparent viscosity measurements can be successfully obtained using the small sample adapter and flag impeller. Also, accurate values of the flow behavior index may be determined from regression analysis of the torque versus angular velocity data curve plotted on log-log scales. The small sample adapter and flag impeller are valuable Brookfield accessories that may be very useful in quality control tests.

Mitschka (1982) proposed a method to calculate apparent viscosity of power law fluids using the Brookfield viscometer and traditional spindles. The application of this method, and a simplified approximation of the Mitschka method, was evaluated for fluid foods. The approximation developed a power law curve that expressed $k\alpha\gamma$ variables as a function of n:

$$k_{\alpha\gamma} = 0.263(n)^{-0.771}$$

This expression eliminated the need to linearly interpolate between n values. Mitschka approximation may be applied with confidence to spindles numbered 2 - 6. Outside this range it generates unacceptable levels of error. Both methods predicted K, n, and apparent viscosities well.

Chapter 6

Future Study

The following would be good topics for future research:

1. Investigating the effect immersion depth of the flag impeller in the small sample adapter has on k' for both the slope and matching viscosity methods.

2. The average shear rate relationship for the matching viscosity method is determined from reference fluids as

$$\dot{\gamma}_a = \left[\frac{\eta}{K}\right]^{\frac{1}{(n-1)}}$$

   When n = 1.0, the power term goes to infinity. Further study is needed to determine the maximum allowable n value for the reference fluid before the power term begins to skew the results.

3. This research evaluated the Mitschka and Mitschka approximation method for fluids with n values less than 0.6. A suggestion for future study is to examine the validity of the Mitschka methods for fluids with n values from 0.6 to 1.0.

4. Mixer viscometry principles could be applied to disc spindles. Evaluate how resulting k' values for each spindle compare with Mitschka values.

Appendix A. Experimental data for the small sample adapter reseach

Table A.1. Brookfield data for the determination of A using Newtonian fluids and small sample adapter.

Fluid	Impeller Speed (rpm)	Torque x 10^{-5} (N m)	Temperature (°C)
5.0 Pa s silicone oil	2.5	2.23	28.7
	5	4.42	
	10	8.80	
	20	17.6	
	2.5	2.25	
	5	4.40	
	10	8.77	
	20	17.6	
5.0 Pa s silicone oil	2.5	1.76	38.0
	5	3.54	
	10	7.12	
	20	14.2	
	2.5	1.76	
	5	3.52	
	10	7.06	
	20	14.2	
5.0 Pa s silicone oil	5	3.00	46.2
	10	5.98	
	20	12.0	
	50	30.1	
	5	3.00	
	10	6.02	
	20	12.0	
	50	30.1	
5.0 Pa s silicone oil	5	2.30	60.2
	10	4.62	
	20	9.25	
	50	23.3	
	5	2.30	
	10	4.62	
	20	9.27	
	50	23.3	

Table A.1. (Cont'd.)

Fluid	impeller speed (rpm)	Torque x 10-5 (N m)	Temperature (°C)
5.0 Pa s silicone oil	5	1.90	71.0
	10	3.83	
	20	7.71	
	50	19.3	
	5	1.92	
	10	3.83	
	20	7.71	
	50	19.4	
glycerin	10	2.05	23.1
	20	4.10	
	50	10.3	
	100	20.5	
	10	2.05	
	20	4.10	
	50	10.2	
	100	20.5	
honey	1	2.57	21.5
	2.5	6.31	
	5	12.8	
	10	25.2	
honey	1	2.77	22.2
	2.5	6.94	
	5	13.8	
	10	27.5	

Table A.2. Haake RS-100 data for the determination of A using Newtonian fluids.

Fluid	Viscosity (Pa s)	Temperature (°C)
5.0 Pa s silicone oil	4.509	28.7
5.0 Pa s silicone oil	3.798	38.0
5.0 Pa s silicone oil	3.276	46.3
5.0 Pa s silicone oil	2.664	60.3
5.0 Pa s silicone oil	2.264	71.0
glycerin	1.039	22.8
glycerin	1.004	23.1
honey	14.436	22.2
honey	16.257	21.5

Table A.3. Brookfield data for power law fluid food using the small sample adapter and flag impeller.

Fluid Food	Impeller Speed (rpm)	Torque x10-5 (N m)	Temperature (°C)
banana baby food	0.5	11.1	29.2
	1	12.9	
	2.5	16.4	
	5	19.5	
	10	24.0	
	20	29.9	
	50	40.9	
	100	53.1	
	0.5	10.7	
	1.0	12.5	
	2.5	15.8	
	5	19.2	
	10	23.4	
	20	29.2	
	50	40.1	
	100	52.5	
chocolate syrup	5	3.02	21.7
	10	4.6	
	20	7.12	
	50	13.9	
	100	24.0	
	5	2.77	
	10	4.17	
	20	6.54	
	50	12.6	
	100	21.7	
enchilada sauce	0.5	4.74	24.7
	1	5.26	
	2.5	6.09	
	5	6.77	
	10	7.67	
	20	8.75	
	50	10.6	
	100	12.4	
	0.5	4.85	

Table A.3 (Cont'd.)

Fluid Food	Impeller Speed (rpm)	Torque x10-5 (N m)	Temperature (°C)
enchilada sauce	1	5.37	24.7
	2.5	6.22	
	5	7.03	
	10	7.89	
	20	9.00	
	50	10.9	
	100	12.8	
mustard	0.5	14.7	23.4
	1	14.9	
	2.5	16.2	
	5	17.6	
	10	19.7	
	20	22.8	
	50	28.4	
	100	35.1	
	0.5	14.9	
	1	15.6	
	2.5	17.1	
	5	18.7	
	10	20.8	
	20	23.8	
	50	28.7	
	100	35.6	
strawberry syrup	5	2.19	24.2
	10	3.11	
	20	4.56	
	50	7.96	
	100	12.7	
	5	2.10	
	10	3.04	
	20	4.47	
	50	7.82	
	100	12.6	
turkey gravy	0.5	5.93	24.5
	1	6.79	
	2.5	8.23	

Table A.3 (Cont'd.)

Fluid Food	Impeller Speed (rpm)	Torque x10-5 (N m)	Temperature (°C)
turkey gravy	5	9.68	24.5
	10	11.4	
	20	13.5	
	50	17.8	
	100	22.4	
	0.5	5.71	
	1	6.58	
	2.5	8.07	
	5	9.47	
	10	11.2	
	20	13.4	
	50	17.6	
	100	22.4	

Table A.4. Haake RS-100 results for power law fluid foods used in small sample adapter and flag impeller research.

Fluid Food	K (Pa s^n)	n (-)	Temperature (°C)
banana baby food	21.35	0.31	29.2
chocolate syrup	3.06	0.68	21.7
enchilada sauce	5.05	0.27	24.7
mustard	19.91	0.26	23.4
strawberry syrup	1.65	0.59	24.2
turkey gravy	9.73	0.32	24.5

Appendix B. Experimental data for the Mitschka method evaluation

Table B.1. Brookfield data using spindles for banana puree at 24.0 °C.

Spindle No.	Impeller Speed (rpm)	% Torque**
3	0.5	50.50
	1.0	59.85
	2.0	69.60
	0.5	50.85
	1.0	59.70
	2.0	69.75
4	0.5	23.75
	1.0	28.90
	2.5	35.20
	5.0	40.40
	10.0	46.60
	20.0	52.30
	50.0	69.25
	0.5	23.75
	1.0	28.70
	2.5	35.00
	5.0	40.10
	10.0	46.40
	20.0	53.80
	50.0	68.15
5	1.0	13.90
	2.5	17.35
	5.0	20.15
	10.0	23.45
	20.0	27.25
	50.0	34.25
	1.0	13.70
	2.5	17.25
	5.0	20.15
	10.0	23.45
	20.0	26.55
	50.0	33.05

Table B.1 (Cont'd.)

Spindle No.	Impeller Speed (rpm)	% Torque**
6	5.0	8.30
	10.0	9.80
	20.0	11.40
	50.0	14.55
	5.0	8.30
	10.0	9.70
	20.0	11.45
	50.0	14.55

**% Torque based on Brookfield RV Viscometer where the maximum capacity is 0.0007187 N m (7187 dyne cm).

Table B.2. Brookfield data using spindles for Catalina Salad Dressing at 23.8 °C.

Spindle No.	Impeller Speed (rpm)	% Torque**
3	0.5	49.60
	1.0	55.45
	2.5	64.90
	5.0	73.00
	10.0	82.80
	20.0	93.65
	0.5	47.20
	1.0	52.80
	2.5	62.30
	5.0	70.90
	10.0	81.05
4	0.5	26.70
	1.0	30.00
	2.5	35.25
	5.0	39.60
	10.0	45.10
	20.0	51.75
	50.0	62.75
	100.0	75.05
	1.0	30.50
	2.5	36.00
	5.0	40.80
	10.0	45.60
	20.0	52.10
	50.0	62.95
5	0.5	12.70
	1.0	14.30
	2.5	16.90
	5.0	19.10
	10.0	21.80
	20.0	25.20
	50.0	30.80
	1.0	14.70

Table B.2 (Cont'd.)

Spindle No.	Impeller Speed (rpm)	% Torque**
5	2.5	17.20
	5.0	19.60
	10.0	22.30
	20.0	25.20
	50.0	30.65
6	0.5	5.50
	1.0	6.20
	2.5	7.30
	5.0	8.25
	10.0	9.40
	20.0	10.85
	50.0	13.10
	1.0	6.10
	2.5	7.30
	5.0	8.30
	10.0	9.45
	20.0	10.90
	50.0	13.15

**% Torque based on Brookfield RV Viscometer where the maximum capacity is 0.0007187 N m (7187 dyne cm).

Table B.3. Brookfield data using spindles for chocolate syrup at 24.0 °C.

Spindle No.	Impeller Speed (rpm)	% Torque**
2	0.5	15.90
	1.0	20.00
	2.5	29.00
	5.0	39.60
	10.0	56.60
	20.0	84.00
	0.5	16.20
	1.0	19.25
	2.5	27.25
	5.0	37.25
	10.0	52.85
	20.0	79.55
3	1.0	7.60
	2.5	11.35
	5.0	15.90
	10.0	22.60
	20.0	33.80
	50.0	61.45
	1.0	7.65
	2.5	11.05
	5.0	15.50
	10.0	22.30
	20.0	33.60
	50.0	61.30
4	2.5	5.10
	5.0	7.40
	10.0	10.85
	20.0	16.35
	50.0	30.20
	2.5	5.30
	5.0	7.65
	10.0	10.90

Table B.3 (Cont'd.)

Spindle No.	Impeller Speed (rpm)	% Torque**
4	20.0	16.50
	50.0	30.40
5	5.0	3.75
	10.0	5.50
	20.0	8.30
	50.0	15.25
	100.0	25.20
	5.0	3.65
	10.0	5.40
	20.0	8.20
	50.0	15.10
	100.0	24.90

**% Torque based on Brookfield RV Viscometer where the maximum capacity is 0.0007187 N m (7187 dyne cm).

Table B.4. Brookfield data using spindles for enchilada sauce at 26.0 °C.

Spindle No.	Impeller Speed (rpm)	% Torque**
2	0.5	50.25
	1.0	62.60
	2.0	75.00
	2.5	79.85
	0.5	48.55
	1.0	61.25
	2.0	74.00
	2.5	78.05
3	0.5	17.60
	1.0	25.00
	2.5	32.85
	5.0	38.50
	10.0	44.90
	20.0	52.25
	50.0	65.70
	100.0	82.80
	0.5	17.30
	1.0	24.30
	2.5	31.95
	5.0	37.55
	10.0	43.75
	20.0	51.30
	50.0	64.20
	100.0	81.35
4	0.5	7.75
	1.0	11.20
	2.5	16.50
	5.0	20.00
	10.0	23.50
	20.0	27.55
	50.0	34.55
	100.0	42.85
	0.5	7.75
	1.0	11.20
	2.5	16.35

Table B.4 (Cont'd.)

Spindle No.	Impeller Speed (rpm)	% Torque**
4	5.0	19.60
	10.0	23.20
	20.0	27.30
	50.0	34.00
	100.0	42.00
5	1.0	5.20
	2.5	8.00
	5.0	10.10
	10.0	12.10
	20.0	14.55
	50.0	18.50
	100.0	23.00
	1.0	5.15
	2.5	7.95
	5.0	9.95
	10.0	11.90
	20.0	14.20
	50.0	18.0
	100.0	21.95

**% Torque based on Brookfield RV Viscometer where the maximum capacity is 0.0007187 N m (7187 dyne cm).

Table B.5. Brookfield data using spindles for pancake syrup at 23.1 °C.

Spindle No.	Impeller Speed (rpm)	% Torque**
2	0.5	10.95
	1.0	15.75
	2.5	24.00
	5.0	33.10
	10.0	46.10
	20.0	64.80
	0.5	10.25
	1.0	15.10
	2.5	23.65
	5.0	32.90
	10.0	45.90
	20.0	64.60
3	2.5	9.90
	5.0	13.90
	10.0	19.65
	20.0	27.75
	50.0	44.00
	100.0	62.20
	2.5	9.80
	5.0	13.85
	10.0	19.60
	20.0	27.65
	50.0	43.85
	100.0	61.90
4	2.5	5.15
	5.0	7.25
	10.0	10.30
	20.0	14.50
	50.0	23.00
	100.0	32.65
	2.5	5.15
	5.0	7.30
	10.0	10.20
	20.0	14.40
	50.0	23.00
	100.0	32.60

Table B.5 (Cont'd.)

Spindle No.	Impeller Speed (rpm)	% Torque**
5	10.0	5.15
	20.0	7.35
	50.0	11.70
	100.0	16.65
	10.0	5.15
	20.0	7.30
	50.0	11.60
	100.0	16.60

**% Torque based on Brookfield RV Viscometer where the maximum capacity is 0.0007187 N m (7187 dyne cm).

Table B.6. Haake RS-100 results for fluid foods used in Mitschka method evaluation.

Fluid Food	K (Pa s^n)	n (-)	Temperature (°C)
banana puree	13.41	0.26	24.0
Catalina Dressing	15.04	0.21	23.8
chocolate syrup	2.19	0.69	24.0
enchilada sauce	7.22	0.28	26.0
pancake syrup	2.76	0.49	23.1

List of References

Castell-Perez, M.E. and J.F. Steffe. 1990. Evaluating shear rates for power law fluids in mixer viscometry. J. Texture Stud. 21:439-453.

Castell-Perez, M.E., J.F. Steffe, and R.G. Moreira. 1991. Simple determination of power law flow curves using a paddle type mixer viscometer. J. Texture Stud. 22:303-316.

Castell-Perez, M.E. and J.F. Steffe. 1992. Using Mixing to evaluate rheological properties. In: Rao, M.A. (editor). Viscoelastic Properties. Elsevier Applied Science Publishers Ltd., Barking, England.

Dolan, K.D. and J.F. Steffe. 1990. Modeling rheological behavior of gelatinizing starch solutions using mixer viscometry data. J. Texture Stud. 21:265-294.

Durgueil, E.J. 1987. Determination of the consistency of non-Newtonian fluids using a Brookfield HBT Viscometer. Proceeding South African Sugar Technologists' Assoc., June 1987.

Ford, E.W. and J.F. Steffe. 1986. Quantifying thixotropy in starch-thickened, strained apricots using mixer viscometry techniques. J. Texture Stud. 17:71-85.

Mackey, K.L., R.G. Morgan and J.F. Steffe. 1987. Effects of shear-thinning behavior on mixer viscometry techniques. J. Texture Stud. 18:231-240.

Metzner, A.B. and R.E. Otto. 1957. Agitation of non-Newtonian fluids. AIChE J. 3:3-10.

Mitschka, P. 1982. Simple conversion of Brookfield R.V.T. readings into viscosity functions. Rheol. Acta 21:207-209.

Neter, J. and Wasserman, W. 1974. Applied Linear Statistical Models. Richard D. Irwin, Inc. Homewood, IL

Rao, M.A. 1975. Measurement of flow properties of food suspensions with a mixer. J. Texture Stud. 6:533-539.

Rao, M.A. and H.J. Cooley. 1984. Determination of effective shear rates in rotational viscometers with complex geometries. J. Texture Stud. 15:327-335.

Rieger, F. and V. Novak. 1973. Power consumption of agitators in highly viscous non-Newtonian liquids. Trans. Instn. Chem. Engrs. 51:105-111.

Savvas, T.A., N.C. Markatos and C.D. Papaspyrides. 1994. On the flow of non-Newtonian polymer solutions. Appl. Math. Modeling 18:14-22.

Steffe, J.F. and E.W. Ford. 1985. Rheological techniques to evaluate the shelf-stability of starch-thickened strained apricots. J. Texture Stud. 16:179-192.

Steffe, J.F., M.E. Castell-Perez, K.J. Rose and M.E. Zabik. 1989. Rapid testing method for characterizing the rheological behavior of gelatinizing corn starch slurries. Cereal Chem. 66:65-68.

Steffe, J.F. 1992. Rheological Methods in Food Process Engineering. Freeman Press, East Lansing, MI.

Williams, R.W. 1979. Determination of viscometric data from the Brookfield R.V.T. viscometer. Rheol. Acta 18:345-359.