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Yield stress of concentrated wheat straw suspensions

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YIELD STRESS OF CONCENTRATED WHEAT STRAW SUSPENSIONS

by

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Punjab University, Pakistan 1994

A Project Report
presented to Ryerson University
in partial fulfillment of the
requirements for the degree of
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In the Program of
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Abstract

The yield stress of concentrated wheat straw suspensions was measured by a vane with a Bohlin rheometer. Wheat straw fibers were made by grinding (wet and dry) wheat straw and separated into four sizes by sieving (8, 12, 20 and 40 mesh). The yield stress was found to increase with concentration as well as with the size of the wheat straw fibers. The yield stress data fitted to power law equation proposed by Kerekes *et al.* (1985) for pulp fibers. It was found that only 40 mesh size wheat straw fibers agreed with the relation. Effect of stress ramp rate was studied on 5% (20 mesh) w/v, wheat straw suspension. It was observed that stress ramp rate had an effect on measured yield stress value. No yield stress was observed in the suspension of 5% (40 mesh) w/v, due to sedimentation of wheat straw fibers. For effective utilization and mixing of enzymes with wheat straw fibers during hydrolysis, knowledge of yield stress of suspension was important.

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Chapter 1 - Introduction

Energy consumption has increased steadily over the last century as the world population has grown and more countries have become industrialized. Crude oil has been the major resource to meet the increased energy demand. Campbell and Laherrere (1998) concluded that a decline in worldwide oil production will begin before 2010. According to them, the annual global oil production would decline from the current 25 billion barrels to approximately 5 billion barrels in 2050. Because the economy in the US and many other nations depend on oil, the consequences of inadequate oil availability could be severe. Therefore, there is a great deal of interest in exploring alternative energy sources.

Bioethanol derived from lignocellulosic residues has been receiving a lot of attention as of late. The existence of an abundant low-valued feedstock for the production of fuel grade ethanol is indeed raising the importance of its utilization. Governments in countries without or with limited deposits of crude oil are especially interested in the potential of such an endeavour for reasons of national security (Wyman, 1996). Environmentalists view bioethanol as an environmentally friendlier alternative to the combustion of fossil fuels, although some consider it as a non-renewable commodity whose negative impact on the environment was calculated as several times larger than those of traditional fossil fuels (Patzek, 2004).

Some of the strongest arguments for ethanol come in its potential to provide cleaner burning fuels. Not only are carbon dioxide emissions decreased but ethanol on the whole burns cleaner than conventional gasoline. Its oxygen content helps gasoline burn more efficiently, cutting tailpipe pollutants including carbon monoxide and benzene. Even addition of 10% ethanol to conventional gasoline can cut carbon monoxide emissions by 20% and overall gasoline toxicity by 30% (Putsche and Sandor, 1996). Bioethanol is not only a possible alternative to a looming energy crisis, it holds the key to a likely solution as well (Szengyel, 2000).

The conversion of lignocellulosic biomass to fermentable sugars for the production of ethanol is nothing new. Various processes have been around for well over hundred years now. Acidic hydrolysis is usually a faster reaction than enzymatic hydrolysis (Sheehan and Himmel, 1999), but the later option has other advantages. For instance, the hydrolysis can be performed in milder conditions, which consumes less energy. Also, the amount of waste to be disposed of is significantly reduced, which lessens not only an economical burden but also environmental concerns (Howard *et al.*, 2003).

Bioethanol can be produced from lignocellulosic materials (e.g., agricultural residues, trees and used paper) and various plant materials (e.g., corn and wheat). Wheat straw is an abundant source of biomass readily available as agricultural residue. Because its cellulose content is high (roughly 34 wt. % dry weight), it is a good source of sugars in the bioprocess for ethanol for use as a fuel extender. Bioconversion of lignocellulosic biomass to ethanol occurs in two steps. The cellulosic chain is first depolymerised to yield the monomeric unit, glucose, which is subsequently fermented to produce ethanol.

Since enzymes are very costly, their effective utilization of is very important. This can be achieved by using adequate mixing to ensure sufficient contact between substrate (wheat straw) and enzymes, and by providing the environmental conditions in which the enzyme remain stable and hydrolyze maximum amount of substrate (Hanna *et al.*, 2001).

One way of reducing the cost of the enzymatic process is to use concentrated wheat straw suspensions. Since wheat straw suspensions are concentrated, they will exhibit yield stress. Under the conditions of mutual attraction the fibers interact with each other to form flocs, which in turn interact to create a continuous, three dimensional networks (flocculated structure) which can preclude flow at low stresses. The suspension flows only when the stress is large enough to break down the structure. On this basis the yield stress has been related to the strength of the coherent network structure as the force per unit area required to break down the structure (Nguyen and Boger, 1983). So the knowledge of yield stress of concentrated suspension is important for better mixing.

The aim of this study is to measure the yield stress of concentrated wheat straw suspensions and to study the effect of concentration and size of wheat straw fibers on the yield stress of the suspensions.

Chapter 2 – Literature Review

Ethanol is a renewable oxygenated fuel. Developing ethanol as fuel, beyond its current role as fuel oxygenate, will require developing lignocellulosic biomass as a feedstock because of its abundance and low cost (Badal and Michael, 2006). Wheat straw can be converted to ethanol fuel by enzymatic hydrolysis. Although enzymatic hydrolysis of lignocellulose has been greatly improved by new technologies, there are still challenges that need further investigation. These challenges include maintaining a stable performance of enzymes and development of more efficient pretreatment technologies for biomass. Since the stirring is a prerequisite for the hydrolysis in pure aqueous medium in order to assure a good contact between the enzymes and biomass (Maria *et al.*, 2001) and the enzyme activity is influenced by the condition of production. So the investigation of favorable mixing pattern and environmental conditions for enzymes stability is important to make the enzymatic hydrolysis process economical. The highly concentrated suspensions show non Newtonian behavior and possess yield stress (Nguyen and Boger, 1983). Therefore, mixing is difficult in such suspensions (Poi and Allen, 1992). Yield stress plays a significant role determining the mixing of pulp suspensions (Ein-Mozaffari *et al.*, 2005). The mixer capacity must be selected so that the threshold value to get the suspension to move is overcome in all of the volume, not just in the space around the mixer. Inadequate mixer capacity in yield stress suspensions will result in dead zones where no mixing action takes place. Thus, knowledge of yield stress is important for better mixing, good mass and heat transfer in bioreactors.

As far as the author could determine, there is no work reported on measurement of yield stress of concentrated wheat straw suspensions. But a lot of work has been done on determining the yield stress of other types of suspensions. This section reviews the wheat straw fibers, rheology (yield stress), and measurement of yield stress (vane).

2.1 Wheat Straw Fiber suspensions

Wheat is an annual agricultural crop grown for the grain portion of the plant that is a valuable food product. The rest of the wheat plant is wheat straw consisting of stems and leaves. Wheat straw is an abundant by-product from wheat production. The average yield of wheat straw is 1.3-1.4 lb per lb of wheat grain (Montane *et al.*, 1998). A sketch of wheat straw is shown in Figure 1.

Figure 2.1 Sketch of Wheat Straw

The stem is the main axe or branch of a plant. It holds up the plant into the air and provides a pathway for fluid transport through plant. A node is the region of stem from which leaves or branches arise, while an internode is the region of stem between nodes. A leaf sheath is the tubular portion of a leaf that encloses the stem while the leaf blade is the flat, expanded portion of a leaf. Wheat straw needs water four times its dry weight for soaking up (Maria *et al.*, 2001). The plant parts contribute different masses as shown in Table 2.1.

Table 2.1 Physical Content of Wheat Plant(Source: Ernst *et al.*, 1960)

	Mass percent
Internodes	68.5
Leaves- sheaths	20.3
Leaves blades	5.5
Nodes	4.2
Grain	1.5

The chemical composition of wheat straw from Pakistan, Denmark, America and Canada is shown in Table 2.2. The variation in composition is due to soil and growth conditions.

Table 2.2 The chemical composition of wheat straw from Pakistan, Denmark, America and Canada

	Pakistan (Ali <i>et al.</i> , 1991)	Denmark (Misra, 1987)	America (Misra, 1987)	Canada (Panthapulakkal <i>et al.</i> , 2006)
Holocellulose	58.5	72.9	68.1	79.44
α - cellulose	33.7	41.6	39.9	47.55
Hemicellulose	25.0	31.3	28.2	31.89
Lignin	16-17	20.5	16.7	18.59
Ash	7.5-8.5	3.7	6.6	
Silica & silicates	4.5-5.5	2.0		
EtOH-Benzene Extract	5.8	2.9	3.7	

Cellulosic (lignocellulosic) materials are comprised of cellulose, hemicellulose and lignin. Lignin provides structural support to the plant. Thus, in general, trees have higher lignin contents than grasses. Unfortunately, lignin which contains no sugars encloses the cellulose and hemicellulose molecules, making it difficult for enzymes to reach them.

Hemicellulose is comprised of long chains of xylose molecules, pentoses containing 5 carbon, some glucose (6-carbon or hexose sugar), mannose, galactose. Hemicellulose is easily broken down into its individual sugars. The exact sugar composition of hemicellulose varies depending on the type of plant (Badger, 2002). Cellulose is a linear mono-polymer chain solely comprised of D-glucose units linked to one another via β -1, 4 glycosidic bonds. One chain end is termed the reducing end because the hemiacetal of the glucose molecule is able to open and expose the reducing aldehyde. The other chain end is called the non-reducing end because the one carbon in the hemiacetal is involved in the glycosidic bond, which prevents the ring from opening (Mosier *et al.*, 1999). Cellulose is a polymer of glucose and is difficult to break down into glucose because of its crystalline structure.

Pretreatment step is used to make the cellulose and hemicellulose components more accessible. So by mechanical treatments like milling and grinding wheat straw can be converted into smaller fibers. These fibers can be suspended in water and, after addition of enzymes, hydrolysis can be accomplished. The following section provides the information about fiber suspensions.

All fibers are considered straight circular cylinders with uniform length l and diameter d . They are also considered slender, i.e., the fiber aspect (axial) ratio $l/d \gg 1$. Fiber suspensions generally have much higher viscosity and elasticity than those of sphere particles at the same concentration. Therefore, fiber concentration of suspensions should be low enough to flow uniformly; otherwise the fibers easily flocculate. This is the main reason why the cellulose fiber concentration of suspensions is less than 1% wt. in papermaking process (Tatsumi *et al.*, 2002).

Many researchers tried to clarify the characteristics of fiber suspensions. Mason (1950) proposed the following equation for critical concentration, at which fibers are no longer able to undergo free rotation since sufficient room for them to move is lacking.

$$c_0 = \frac{V_a}{V_e} = \frac{3}{2p^2} \quad (2.1)$$

where c_0 is the critical concentration (the concentration at which behavior of a suspension shifts to a non-Newtonian from Newtonian flow), V_a is the actual volume of a fiber, V_e is the effective volume of the rotating fiber (the volume swept out by a rotating fiber and equal to that of a sphere whose diameter equals the length of a fiber), and p is the aspect ratio (length / diameter) of the fiber.

Kerekes *et al.* (1985) proposed the following power equation

$$\tau_0 = k(c_m)^\alpha \quad (2.2)$$

where τ_0 is the yield stress of the pulp suspension, c_m is the mass concentration of the suspension, and k and α are constants. They have reported that the constants are unique for each suspension and their values are in the interval of $1.18 < k < 24.5$ and $1.26 < \alpha < 3.02$, respectively.

Bennington *et al.* (1990) have extended the equation theoretically:

$$\tau_0 = kEp^2 c_v^3 \quad (2.3)$$

where c_v is the volumetric concentration of the suspension, p is the aspect ratio and E is Young's modulus of the fiber. However, they found that this equation does not agree with their experimental results as to the powers of E and p .

Fibers have larger excluded volume than spherical particles. Therefore, the interaction between fibers is strong even at low fiber suspension concentration. The interaction is more remarkable as the fiber aspect ratio becomes larger. In this case, the fibers tend to form flocs rather than remaining individual fibers because the former is more stable thermodynamically. It is expected that wheat straw fibers will behave in this manner.

Tatsumi *et al.* (2002) studied the effect of fiber concentration and axial ratio on the rheological properties of various types of suspensions of cellulose fibers. All suspensions showed non-Newtonian flow even at very low concentrations. Over their critical concentration, each suspension also had a yield stress.

Leonard and Klingenberg (2003) studied the effect of fiber shape, interfiber friction, and fiber stiffness on the viscosity of fiber suspensions. The viscosity of the suspensions was found to increase as the fiber curvature, the coefficient of friction, or the fiber stiffness increased. They observed that fiber shape also affected the yield stress of fiber suspensions.

So it can be assumed that concentrated wheat straw fibers suspensions will show non-Newtonian behavior and will possess yield stress. Since the yield stress is important rheological property, so the understanding of rheology is important to know.

2.2 Rheology

Rheology is the science of deformation and flow of matter, investigating the response of materials to applied stress or strain (Barnes *et al.*, 1989). Mathematically, rheological behavior can be expressed by constitutive equations. The most essential parameters defining the constitutive equations are stress and strain.

2.2.1 Shear stress and shear strain

When a material is subjected to a force (F), the sample responds by deforming. Stress (τ) is defined as the amount of force applied on the material per unit area (A) of the application surface with units of Pascal (Pa)

$$\tau = \frac{F}{A} \quad (2.4)$$

The resulting deformation is measured as strain (ε or γ), computed as the relative displacement in the material. Accordingly, strain, a dimensionless quantity, is the ratio of the change in length to original length.

Normal and shear are two stress defined by the direction of force application. When a force is applied perpendicularly to the material surface, normal stress is observed which induces a normal strain (ε). Similarly, a force applied parallel to the material surface causes a shear stress and consequently a shear strain (γ).

Shear strain rate ($\dot{\gamma}$) is the time rate of change of strain occurring in the material and can be defined as the rate at which adjacent layers of a material move with respect to each other (Hadley and Weber, 1975).

Shear rate is a velocity related parameter and is usually expressed in reciprocal seconds (1/s):

$$\dot{\gamma} = \frac{d\gamma}{dt} \quad (2.5)$$

Apparent viscosity (η) of fluids is described as the resistance to flow offered by fluid layers during the application of a shearing force and is calculated according to the following equation.

$$\eta = \frac{\tau}{\dot{\gamma}} \quad (2.6)$$

Viscosity is one of many such parameters, describing stress-strain and stress-strain rate relationships, used to characterize materials.

2.2.2 Rheological characterization of materials

Rheological characterization is based on a response to an applied load, force or deformation, and materials can thus be rheologically classified as elastic (ideal solids), viscous (ideal fluids), or viscoelastic.

Ideal solids (elastic) and ideal fluids (viscous) represent extremes for rheological analyses, and the substances between the two extreme scenarios are called viscoelastic materials. Ideal solids store energy gained during deformation and, following the removal of the load, return to an original shape using the stored energy. Hooke's law is observed in such solids which state that the load applied to a body is directly proportional to the imposed deformation. In rheological terms, stress and strain are related linearly to each other:

$$\tau = G\gamma \quad (2.7)$$

where G is the Hookean shear modulus of the material, a constant unique to the material. On the other hand, ideal liquids (considered Newtonian fluids) do not store, but dissipate energy completely in the form of heat. For ideal fluids, the stress applied to the fluid is proportional to strain rate. Hence, the rheological model representing ideal liquid is

$$\tau = \mu\dot{\gamma} \quad (2.8)$$

and μ is the Newtonian viscosity. Fluids having a constant viscosity for any shear rate are called Newtonian and their viscosity is called Newtonian viscosity. If the ratio of shear stress to shear rate is not constant, the fluid is called non-Newtonian, and their viscosity is called apparent or non-Newtonian viscosity. Viscoelastic materials exhibit a combination of elastic and viscous effects simultaneously (Barnes *et al.*, 1989).

2.2.3 Yield Stress

Most fluids do not display Newtonian behavior and are called non-Newtonian materials. Certain non-Newtonian materials exhibit a property known as yield stress, see Fig 2.2.

Figure 2.2 Behavior of Newtonian and yield stress fluid

(Source: McCabe *et al.*, 1993)

Yield stress is defined as the minimum stress required to initiate flow. Accordingly, these non-Newtonian materials are expected to be viscoelastic solids prior to yielding and viscoelastic plastic liquids post yielding (Guo *et al.*, 2003). Yield stress is often a desirable property and is accountable for the functional performance of many products. For example, paints are required to be suitably thin for application purposes. At the same time, paints are also required to exhibit a yield stress to supply sufficient resistance to avoid sagging or flow after application. Many food products, including ketchup, mayonnaise, and salad dressings, are specifically designed to impart a yield stress. For example, a salad dressing is expected to be thick and not flow freely from a container, but at the same time should not require high stresses to initiate flow. Thus, the application and intensity of yield stress are significant components for product quality. Barnes

(1999a) discussed the concept of yield stress in a review paper. Some noteworthy features are that yield stress has been traditionally measured as the stress where a significant decrease in viscosity is observed. However, a logarithmic plot of viscosity against the logarithm of applied stress shows that the material flow occurs in a range of stress values and not at a unique value, see Fig 2.3. Hence when the time of observation is extended to geological scale, even mountains flow.

Figure 2.3 Yield stress determination, (Source: Barnes, 1999a)

2.2.4 Existence of Yield Stress

Barnes and Walters (1985) stated that yield stress is an ideal concept, one that does not exist in reality. If a material flows at high stresses it will also flow, however slowly, at low stress. Also, materials known to have a unique yield stress started to flow below yield stress when tested with modern equipment, like controlled stress rheometers at low rates (Barnes, 1999b). Finally, Barnes and Walters (1985) stated that experimental verification of yield stress would involve an infinite time, and therefore be impossible to perform. The proposed concept by Barnes and Walters received criticism from Hartnett and Hu (1989), who identified yield stress as an 'engineering reality'. Using a falling ball technique, these researchers observed no change in the position of a nylon ball after several months in a solution of high viscosity. Thus, the solution showed a significant

yield stress. Barnes (1999b), defending their earlier position on the non-existence of yield stress, suggested the use of a microscope to observe the change in position. Astarita (1990) and Evans (1992) bridged the two extreme theories by stating that the entire concept revolves around the order of the distance measured and experimental time scale. Accordingly, the existence of the yield stress depends on the nature of the experiment being conducted. Schurz (1992) and Cheng (1986) affirmed that an apparent yield stress, which would be distinguished according to a significant measurable time and lowest possible shear rate, could be more meaningful. Therefore, a yield stress is believed to be a time-dependent property, varying inversely with the time period employed for the test. A longer measurement time (lower rate) will generate a smaller yield stress value. Accordingly, a higher strain rate would produce a higher yield stress and vice versa, see Figure 2.4 (Nguyen and Boger, 1992).

Figure 2.4 Yield stress determination at different shear rates

Note: Shear rate 1 < Shear rate 2 < Shear rate 3

Uhlherr *et al.* (2005) believed that the material yielding is determined by a critical strain, the yield strain (γ_o), rather than by a critical stress or critical shear rate. The measurement device employed for the test is also believed to affect the yield stress value. Although the yield stress concept might be in debate, the concept remains useful, and the

yield stress remains an important parameter in rheological characterization of complex fluids and in understanding their processing characteristics in industrial applications.

2.3 Measurement of Yield Stress

Broadly speaking, there are two methods for yield stress determination, indirect and direct methods.

Indirect methods involve extrapolation of shear stress-shear rate data obtained from conventional rheometers to zero shear rate. This can be performed either without or with the use of a rheological model such as Bingham, the Herschel-Bulkley or the Casson model, see Figure 2.5.

Figure 2.5 Determination of yield stress by extrapolation (Nguyen and Boger, 1983)

The extrapolated yield stress relies on accurate experimental data in the low shear rate range (Nguyen and Boger, 1983). However this is often difficult to obtain owing to the inherent nature of the yield stress material. Slip flow of sample is common problem which occurs in conventional rheometers in the low shear rate range (Magnin and Piau, 1990).

Direct methods for yield stress measurement rely on an independent assessment of the stress at which the material starts to flow. The most common examples of direct measurements using conventional rheometers are creep/recovery, stress relaxation and stress growth techniques. The creep/recovery technique involves applying a constant stress in steps to a material, and observing over time the creep response and/or the recovery response after removal of the stress. The yield stress is determined as the stress at which the strain starts to increase indefinitely with time, and incomplete strain recovery is observed upon removal of the stress. In the stress relaxation technique the material is first sheared at a constant shear rate or shear stress, and is then brought to rest. The yield stress is determined as the limiting or residual stress remaining in the material upon cessation of flow. In the stress growth technique the material is sheared at a low and constant shear rate and the resulting shear stress measured as a function of time. The yield stress is typically determined as the maximum stress in the stress-time profile (Liddell and Boger, 1995). In stress ramp technique, the shear stress is increased linearly at a constant rate from zero to a level well above the yield stress and the resulting deformation is recorded. There are two ways of determining the yield stress from stress ramp measurements and these are shown in Figures 2.6 and 2.7.

Figure 2.6 Determination of yield stress with viscosity versus stress curve

In figure 2.6, the yield stress is determined as the stress at which the apparent viscosity is maximum. There is no real flow before this point.

Figure 2.7 Determination of yield stress with strain versus stress curve

From figure 2.7, it is clear that as long as the sample is elastic the strain is proportional to the applied stress, but when the sample structure is broken, the strain varies far more rapidly with stress. The yield stress is taken at the curve inflexion point. The Bohlin rheometer uses this stress ramp technique to measure yield stress. A vane was used as measuring system to measure the wheat straw suspensions.

2.3.1 Vane for Yield Stress Measurement

A vane consists of a number of thin blades (usually 2 to 8) fitted at equal angles around a cylindrical shaft (Poi and Allen, 1992).

Figure 2.8 The vane in side and top view

It was Russel (1936) who used the vane first time to study the coagulation of sodium clay gels. He used vane instead of cylinder to eliminate the possibility of slipping. Usually multiphase systems show yield stress. These multiphase systems exhibit the problem of slip when tested at low shear rates with smooth surfaces geometries. Displacement of the dispersed phase from solid boundaries cause the formation of a thin, particle-depleted, low viscosity boundary layer initiating the slip of the material during experimentation (Zhu *et al.*, 2001).

Then vane geometry has been available for Haake viscometers (Germany) since 1955. According to the company, the vane was introduced to overcome slip problem and to measure yield stress. In 1983 Nguyen and Boger further developed the vane for practical measurement of the yield stress of concentrated suspensions. Keentok(1982) studied the theoretical aspects of vane and confirmed the idea that vane can be used for yield stress measurement. Since then vane method has been applied for yield stress measurements on greases (Keentok, 1982), oil-in-water emulsions (Yoshimura *et al.*, 1987), and illitic suspensions (James *et al.*, 1987). Moreover, Barnes and Carnali (1990) showed that vane was a suitable rheometer geometry based on numerical simulation and tests on a sodium carboxymethylcellulose (CMC) solution and clay suspension.

In addition to the elimination of wall slip, vane geometry has the advantage that any disturbance caused by the insertion of the vane into the sample can be kept to minimum.

2.3.2 Vane Rheometry Calculations

Following are the assumptions for vane method

- the vane forms a rigid cylinder with the material trapped within the blades
- the material trapped within the blades rotates along with the vane during rotation, and hence no secondary flow occurs between the blades
- the yield surface has a cylindrical shape
- the stress distribution is uniform over the surface of the vane cylinder
- the vane geometry is considered to be two dimensional since the vane length is assumed to be infinitely long

The computation of yield stress using the vane rheometer is based on the geometry of vane. Certain specifications have been established for the positioning of the vane during experimentation (Nguyen and Boger, 1983; Liddell and Boger, 1996), see Fig. 2.9.

Fig. 2.9 Vane in a cup

- (i) Ratio of the vane length (H) to vane diameter (D) should lie within 1.5 and 4.

(ii) Depth of the vane surface from the surface of material (Z_1) can be zero or the ratio of Z_1 to the vane diameter should be greater than 1.0.

(iii) Ratio of the height of vane from bottom of cup (Z_2) to vane diameter should be greater than 0.5.

The ratio ensures that the end effects at the top and bottom of the vane are negligible

The vane is inserted in a yield stress sample and is rotated at constant and slow rate. The total torque required to overcome the yield stress of the fluid can be broken down into two parts

(I) torque (T_c) required for the vane's cylindrical surface,

$$T_c = [(\pi DH)(\tau_o)] \left[\frac{D}{2} \right] = \frac{\pi HD}{2} \tau_o \quad (2.9)$$

In the expression, πDH is the area of stress application (cylindrical surface area), and $D/2$ is the distance from the point of force application to the vane's cylindrical surface,

(II) the torque (T_h) required for the upper and lower horizontal surfaces of the vane,

$$T_h = 2 \int_0^{\frac{D}{2}} 2\pi r^2 dr \tau_e \quad (2.10)$$

Where, τ_e is the shear stress on the vane ends. The area of stress application (upper and lower horizontal surfaces of the vane) is $2\pi r^2$.

Assuming τ_e varies with the radius (r) according to a power law relationship,

$$\tau_e = \left(\frac{2r}{D} \right)^m \tau_o \quad (2.11)$$

where m is a constant. This assumption is somewhat arbitrary, but reasonable.

Substituting this value of τ_e in equation 2.10,

$$T_h = 4\pi \int_0^{\frac{D}{2}} r^2 \left(\frac{2r}{D} \right)^m \tau_o dr \quad (2.12)$$

$$T_h = \frac{2^m (4\pi \tau_o)}{D^m} \int_0^{\frac{D}{2}} r^{2+m} dr \quad (2.13)$$

Substituting $r = D/2$

$$T_h = \frac{2^{m+2} \pi \tau_o \left(\frac{D}{2}\right)^{3+m}}{D^m (3+m)} \quad (2.14)$$

$$T_h = \frac{\pi \tau_o D^3}{2(3+m)} \quad (2.15)$$

Hence, the total torque can be determined as

$$T_o = T_c + T_h \quad (2.16)$$

$$T_o = \frac{\pi H D^2}{2} \tau_o + \frac{\pi \tau_o D^3}{2(3+m)} \quad (2.17)$$

$$T_o = \frac{\pi \tau_o D^3}{2} \left(\frac{H}{D} + \frac{1}{m+3} \right) \quad (2.18)$$

Re- writing the above expression, the yield stress of the material (σ_o) can be determined as,

$$\tau_o = \frac{2T_o}{\pi D^3} \left(\frac{H}{D} + \frac{1}{m+3} \right)^{-1} \quad (2.19)$$

Substituting $m = 0$ is usually satisfactory and errors in using Eq 2.19 for $m > 1$ decreases with larger values of H/D . If $m=1$, errors less than or equal to 3.7% may be obtained when $H/D > 2$ (Barnes, 1999). Conventionally 'm' is assumed to be zero.

There are many general areas where the vane geometry has been used to measure yield stress. These areas are inorganic colloidal dispersions, liquid-like foods, bioengineering systems (fermentation broths), foams, etc. Some examples of these are given in Tables 2.3-2.4.

Table 2.3 Uses of vane for yield stress determination in inorganic colloidal dispersions

Type of dispersion	References
Bentonite	Alderman <i>et al.</i> , 1991
Illite suspensions	James <i>et al.</i> , 1987
TiO ₂ pigment	Liddell and Boger, 1996
Discotic clay	Pignon <i>et al.</i> , 1996
Dense fly-ash slurries	Iyer and Stanmore, 1999
Brown coal slurries	Leong <i>et al.</i> , 1987
Coal-water mixture	Turain <i>et al.</i> , 1993
Aqueous foams in dry limit	Gardiner <i>et al.</i> , 1998

Table 2.4 Uses of vane for yield stress determination in food and bioengineering systems

Type of food system	References
Frozen ice cream	Briggs <i>et al.</i> , 1996
Fruit pectin jam	Houska <i>et al.</i> , 1997
Processes cheese analogue	Mleko and Foegeding, 2000
Apple pulps	Missaire <i>et al.</i> , 1990
Proteins foams	Pernell <i>et al.</i> , 2000
Various food	Rao and Steffe, 1997
Gellan gum gels, soybean protein	Truong and Daubert, 2000
Molten chocolate	Wilson <i>et al.</i> , 1993
Fermentation broth, ketchup	Leongpoi and Allen, 1992
Various fermentation broths	Mohseni <i>et al.</i> , 1997

Based on the above discussion, it is clear that highly concentrated fiber suspensions posses a yield stress. The vane can be used to measure the yield stress of fiber suspensions.

Research objective

The main objectives of the present study were to determine the yield stress of concentrated wheat straw suspensions and to study the effect of fiber size and concentration of wheat straw suspensions on the yield stress. Bohlin rheometer was used to accomplish this study.

Chapter 3 – Material and Methods

This section provides the details of raw material their source and equipment used to accomplish this investigation.

3.1 Material

Wheat straw, standard viscosity oil and Colgate toothpaste was used in this study. Wheat straw was three years old, so it was almost dry.

Wheat straw mechanical pretreatment

Wheat straw (Soft White Superior, generously provided by Mr. John Morrisson, Cookston, Ontario) was used in this study. Wheat straw was chopped into pieces of approximately 6 cm at the Department of Agriculture, University of Guelph, using a mechanical chopper.

Thirty-five grams of chopped wheat straw were placed in a home blender with 1 L water. The mixture was ground 15 minutes by pressing grind key on blender. The thick slurry was then spread on a plastic sheet for air drying for 48 hours. The dried lumps of wheat straw were then ground again in the same blender, without water, for 10 minutes. This finer mixture was kept in plastic bags until sieving.

Approximately 15.0 g of the finer mixture were placed on the top sieve mesh No. 8. This sieve was followed by sieve mesh Nos. 12, 20, and 40 (lower sieve), and finally a solid pan at the bottom. The mesh size and corresponding diameters are provided in the following Table 3.1.

Table 3.1 Mesh number and diameter of sieve

Mesh (#)	Diameter (mm)
8	2.36
12	1.68
20	0.833
40	0.417

According to Jud and Kononoff (1996), sieves were shaken manually to and fro 7 times and then the stack was tilted one quarter and shaken again 5 times. These 12 shakings constituted 1 set. This procedure was repeated 7 times, for a total of 8 sets. Care was taken to avoid vertical motion of wheat straw during the shaking. Wheat straw was recovered from the individual sieves (Nos. 8, 12, 20, 40) and weighed.

Using four sizes (40, 20, 12 and 8 mesh) of wheat straw fibers, four concentrations (5, 10, 15 and 20% w/v) of wheat straw suspensions were made in distilled water respectively. Only freshly prepared samples were used to measure yield stress to avoid any contamination caused by bacteria.

Standard viscosity oil No.12

This oil provided with Bohlin rheometer was used to calibrate the cone and plate and the vane attachments of the rheometer. It had a viscosity of 0.972 Pa.s at 25 °C.

Toothpaste

Colgate regular toothpaste purchased from local store was used to measure yield stress by cone and plate and the vane attachments of rheometer.

3.2 Rheological measurements

The rheological measurements were made using standard viscosity oil # 12, toothpaste, and wheat straw suspensions on a Bohlin C-VOR 150 (Malvern Instruments Ltd., Worcestershire, UK) rheometer.

Cone and plate was used to calibrate the rheometer using standard oil # 12 and toothpaste. The cone with a 4° angle and having a diameter of 40mm, supplied with rheometer, was used.

The vane was fabricated in the workshop of the department of Chemical Engineering at Ryerson University. The material used for vane fabrication was aluminum. The vane dimensions are shown in Table 3.2.

Table 3.2 Vane dimensions

Number of blades	Height (mm)	Diameter (mm)	Height/Diameter (-)
4	38.1	19.8	1.92

The container used to hold the samples for vane test was a stainless steel cup (height = 60mm; diameter = 27.5mm).

To run this vane on rheometer parameters (form factors) were required which were obtained from Bohlin rheometer support centre. These parameters were inserted in rheometer software. The viscosity of standard oil was measured with the vane. The value of viscosity was in the range of 0.7068 to 0.7339 Pa.s. Then the parameter C1 was reset as per Malvern support centre technologist. Initially the value of parameter C1 was 29874, which was changed to 39000 later. Then results obtained were satisfactory.

3.2.1 Principle of Bohlin rheometer (controlled stress mode)

In controlled stress mode, the rheometer uses a constant torque motor. The software automatically converts the applied value of torque to shear stress through a parameter, called "form factor", specific to each type of measuring device (cone-and-plate, vane, etc.). An angular position sensor detects the movement of the measuring system attached to the shaft of the vane. The reading from the position sensor is converted to strain using another "form factor". By monitoring the change of strain as a function of time, at controlled stress, the shear rate value can be obtained.

3.2.2 Procedure to measure yield stress on Bohlin rheometer

The vane was fixed to the upper chuck of the rheometer, and the empty cup was set in place. The key marked ZERO was pressed to set the correct gap between the bottom of the vane and the cup. The instrument automatically finds the correct zero position for this vane and cup. After the OK light came on, the software was set to "vane". In viscometric mode of operation, "shear profile" was selected when performing yield stress measurements (controlled stress mode). The lowest (1 Pa) and maximum (300 Pa) values of shear stress expected to include the yield stress value of the studied suspension were set, and a ramp time of 100 seconds was selected. The vane was then reset into its uppermost position. Distilled water was added to wheat straw fibers of 40 mesh size, and mixed thoroughly to make a 5% w/v suspension. The suspension was immediately poured in the cup, and the down arrow on the rheometer was pressed. The vane took almost 55 seconds to automatically be inserted in the sample. The START button was pressed to begin the run. At the end of the run, the vane was moved up, and the cup was removed. Both were washed with tap water and dried with tissue paper.

After completion of the run, the experimental data collected by the rheometer software were transferred to an Excel document. The apparent viscosity calculated by the software

versus the shear stress values were plotted. For higher suspension concentrations and/or higher fiber mesh sizes, freshly-prepared samples were run at increased values of maximum shear stress, at the same 100-s ramp time, until the rheometer software detected a yield stress.

3.3 Statistical Analysis

The precision of the data reported in Results section is represented by error bars defined as:

Confidence interval (95%) = mean value $\pm$ 1.96 $\times$ standard deviation (SD)

Chapter 4 - Results and Discussion

4.1 Calibration and preliminary yield stress determination

The viscosity of standard oil # 12 was verified using the plate and cone geometry of a Bohlin rheometer. Three oil samples, replicated three times resulted in the average values of viscosity of 0.8929, 0.9370 and 0.9591 Pa.s, respectively. See Table A 1-3, in Appendix A for the viscosity data. Figure 4.1 shows the average viscosity of each of three experiments. The error bars represent the confidence interval at 95% probability. Since all three error bars almost overlap, there is 95% probability that mean are the same.

Figure 4.1: Viscosity of standard oil # 12 measured by cone and plate. Horizontal line is the standard oil viscosity, and vertical error bars are 95% confidence interval of mean

Then the vane fabricated in our workshop was used to determine the viscosity of standard oil #12 with the parameters (form factors) reported earlier.

In four different experiments, the average viscosity of each experiment was 0.9699, 0.937, 0.9393 and 0.9699 Pa.s respectively. Figure 4.2 shows the viscosity of each four

experiments. The vertical bars are the error bars at 95% probability. Since all error bars overlap, so there is 95% probability that their mean is the same.

Figure 4.2: Viscosity of standard oil # 12 measured by vane. Horizontal line is the standard oil viscosity, and vertical error bars are 95% confidence interval of mean

So it can be seen that viscosity measured by cone and plate is 96.5% accurate and vane is 98.2% accurate.

The yield stress of toothpaste was then measured by a cone and plate (Figure 4.3a) and by the vane (Figure 4.3b). Figures showed that viscosity increased up to maximum value, where yield stress was detected, shown by vertical arrow. After that viscosity was decreased due to flow. The value of yield stress obtained using cone and plate and vane were 63 and 73 Pa, respectively. The 95% confidence interval for these values was 68 ± 9.799 Pa, where 68 Pa was the mean. So statistically these values were same. Hence, vane could generate reliable results. Thus, vane could be used to measure the yield stress of wheat straw suspensions.

(a)

(b)

Figure 4.3 Yield stress determination of Colgate toothpaste using (a) a cone and plate and (b) a vane geometry. Shear stress ramp: 1-100 Pa, Ramp time: 120s

4.2 Yield stress of concentrated wheat straw suspensions

Figure 4.4 shows the apparent viscosity versus shear stress for 5% w/v wheat straw suspension (8 mesh). The apparent viscosity increased rapidly up to maximum value of 7.78×10^5 Pa.s. At maximum of viscosity curve, the software records the shear stress as yield stress and its value is 411 Pa. By observing the data obtained during this experiment, it can be seen that value of maximum viscosity (7.78×10^5 Pa.s) is same for shear stress of 390 Pa and 411 Pa. It means software records the shear stress as yield stress at maximum apparent viscosity after which viscosity decreases. After maximum viscosity has been achieved the curve goes down indicating that flow has been initiated. This part of curve shows that suspension behaves like a non-Newtonian fluid, because its viscosity is not remaining constant as a Newtonian fluid would.

Figure 4.4 Apparent viscosity and shear stress for 5% w/v (8 mesh) wheat straw suspension. Shear stress sweep: 1-1000 Pa, Ramp time: 100 seconds

4.2.1 Measurement pitfalls

Figure 4.5 shows the apparent viscosity versus shear stress for 5% w/v wheat straw suspension (40 mesh). Software detected a yield stress of 23 Pa, although no yield stress could be seen on the graph. The reason is that 40 mesh wheat straw is very fine fibers and when suspension was made in water, it settled down quickly. Since there is a gap under vane, and fibers settled in this area, so upper portion of suspension did not show any yield stress. From figure 4.5, it is clear that suspension showed Newtonian behavior because there is no change in viscosity after approximately 10 Pa. The value of viscosity is 0.07 Pa.s, which is higher than water viscosity (0.001 Pa.s). So this contains some dissolved lignin and dirt etc., which are responsible for higher viscosity than water. This solution did not offer resistance to moving vane and splashed outside cup.

Figure 4.5 Apparent viscosity and shear stress for 5% w/v (40 mesh) wheat straw suspension. Shear stress sweep: 1-70 Pa, Ramp time: 100 seconds

Figure 4.6 shows the viscosity curve for 10% (40mesh) wheat straw suspension. In this experiment software detected the yield stress at 147 Pa, but the maximum apparent viscosity occurred at 153 Pa. So 153 Pa is taken as yield stress. Hence, in all subsequent experiments the yield stress is taken at maximum viscosity. Moreover in this curve there are lot of bumps which may be attributed to fibers interactions and their structure in the suspensions.

Figure 4.6 Apparent viscosity and shear stress for 10% w/v (40 mesh) wheat straw suspension. Shear stress sweep: 1-300 Pa, Ramp time: 100 seconds

4.2.2 Replicates

To check the reproducibility of results a series of experiments were conducted on 5% (20 mesh) wheat straw suspensions. The results were 276, 290, 318, 332, 311, 332, 339 Pa. The average value of yield stress of all seven experiments was 307 Pa. The standard deviation was 23.5159 Pa and 95% confidence interval found to be 307 ± 17.4205 Pa. So with 95% probability it could be concluded that yield stress value will fall between 289.57 and 324.42 Pa.

4.2.3 Potential influence of ramp rate

The effect of shear stress ramp rate on the yield stress is shown in Figure 4.7 for the 5% w/v (20 mesh) wheat straw suspension.

Figure 4.7 Effect of ramp rate on yield stress of 5% (20 mesh) wheat straw suspension

By applying 95% confidence interval, the yield stress was found to lie between $312 \text{ Pa} \leq \tau_0 \leq 346 \text{ Pa}$. No linear correlation could be found from the above data. So the experimental data seem to contradict Nguyen *et al.* (2006), who showed that stress ramp rate had a definite effect on the yielding process of the suspension: the lower the applied ramp rate, the smaller the critical stress observed. Since Nguyen *et al.* (2006) used aqueous suspensions of colloidal TiO_2 for yield stress determination, it can be concluded that wheat straw fibers did not behave like TiO_2 particles. So if ramp rate below 3.5 Pa/s is used, then there is possibility of effect of ramp rate on yield stress values of wheat straw suspension.

4.2.4 Effect of wheat straw concentration on yield stress

Yield stress values for concentrations 5, 10, 15 and 20%, w/v for mesh sizes (40, 20, 12 and 8) are provided in Table A 8 in Appendix A. It is obvious from results that as concentration of wheat straw increased, the yield stress also increased. As can be seen from Figure 4.8, wheat straw seems to follow a relationship of the type $\tau_0 = kc^\alpha$,

proposed by Kerekes *et al.* (1985). As expected, concentration of the fibers in solution had a larger effect when fibers were very small (40 mesh).

Figure 4.8 Logarithmic plots of yield stress versus wheat straw concentrations

The values of α and k for the various sizes of wheat straw are provided in Table 4.1.

Table 4.1 Values of constants α and k for various wheat straw mesh sizes

Mesh size	α	k
40	1.626	3.81
20	0.825	39.6
12	0.360	208
8	0.622	157

Only the values of α and k for 40 mesh size agreed with the values given by Kerekes *et al.* (1985). They used fibers having aspect ratio between 20.3 to 93.7. The reason is that 40 mesh sizes of wheat straw fibers are very small and can be compared to the pulp fiber sizes for which these researchers proposed the relation.

Since k reflects individual fiber characteristics such as size of fiber and α reflects structural property of the whole suspension (Tatsumi *et al.*, 2002), the value of k varies from 3.81 to 156.56, due to big size difference of fibers. There is small variation in α values, which shows almost same structural behavior of different size wheat straw suspensions.

4.2.5 Time dependency of viscosity of wheat straw suspension

5% (20 mesh) wheat straw suspension was used to study the time dependency of viscosity. The apparent viscosity of wheat suspension was measured at a constant shear stress of 150, 400 and 500 Pa (Figure 4.9). One can see that the viscosity increased with time. This increase in viscosity may be due to structure build-up of wheat straw fibers in suspension. In all three experiments viscosity increased in similar fashion and viscosity variation is not prominent. This observed pattern shows that this suspension is not thixotropic since there is no decrease in viscosity over time.

Figure 4.9 Time profile of the apparent viscosity of 5% (20 mesh) suspension at constant shear stress of 150, 400 and 500 Pa

Chapter 5 – Conclusion and Recommendations

The present study investigated the yield stress of concentrated wheat straw suspension by vane using Bohlin rheometer. From the analysis of results obtained, the following conclusions could be drawn:

- The concentration and size of wheat straw fibers had a definite effect on the measured value of yield stress. The yield stress increased with increase in fiber concentration and size.
- Stress ramp rate had a little effect on yield stress of wheat straw suspension but no linear correlation was found.
- Sedimentation effect was prominent only in 5% (40 mesh) w/v, wheat straw suspension, where no yield stress was found.
- No thixotropic behavior of wheat straw suspension (5%, 20 mesh) was observed.

It is recommended that minimum stress ramp rate must be used during yield stress measurement to have reproducible results. Also to have more precise results a rough surface cup must be used to eliminate wall slip if present. It is recommended that flow behavior of wheat straw suspension after yielding must be studied in more detail, to determine whether it is shear thickening or shear thinning. To observe the further behavior of viscosity with respect to time, higher shear stress should be used.

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Appendix A – Viscosity Determination

Table A 1. Viscosity determination of standard oil # 12 using a cone and plate
at 25°C at a constant shear stress of 1 Pa

Observation (#)	Time (s)	Shear Rate (s ⁻¹)	Viscosity (Pa.s)
1	20.02	1.111	0.9002
2	40.08	1.111	0.9003
3	60.18	1.115	0.8966
4	80.27	1.118	0.8940
5	100.4	1.125	0.8887
6	120.5	1.122	0.8914
7	140.6	1.122	0.8912
8	160.7	1.123	0.8904
9	180.8	1.128	0.8869
10	200.9	1.124	0.8893
Mean viscosity, Pa.s			0.8929

Table A 2. Viscosity determination of standard oil # 12 using a cone and plate
at 25°C at a constant shear rate of 0.519 s⁻¹

Observation (#)	Time (s)	Shear stress (Pa)	Viscosity (Pa.s)
1	20.02	0.4838	0.9326
2	50.09	0.4901	0.9446
3	80.16	0.4824	0.9299
4	110.3	0.4924	0.9491
5	140.4	0.4807	0.9266
6	170.5	0.4907	0.9458
7	200.6	0.4832	0.9313
8	230.7	0.4872	0.9390
9	260.7	0.4865	0.9378
10	290.9	0.4846	0.9340
Mean viscosity, Pa.s			0.9370

Table A 3. Viscosity determination of standard oil # 12 using a cone and plate
at 25°C at a constant shear stress of 10 Pa

Observation (#)	Time (s)	Shear rate (s^{-1})	Viscosity (Pa.s)
1	20.02	10.38	0.9637
2	40.08	10.39	0.9628
3	60.14	10.41	0.9607
4	80.21	10.42	0.9597
5	100.3	10.42	0.9594
6	120.4	10.44	0.9582
7	140.5	10.44	0.9575
8	160.6	10.44	0.9574
9	180.7	10.46	0.9564
10	200.8	10.46	0.9561
Mean viscosity, Pa.s			0.9591

Table A 4. Viscosity determination of standard oil No. 12 using a vane at 25°C at a
constant shear stress of 10 Pa

Observation (#)	Time (s)	Shear Rate (s^{-1})	Viscosity (Pa.s)
1	20.02	10.3	0.9713
2	40.08	10.3	0.9705
3	60.14	10.31	0.9701
4	80.21	10.31	0.9701
5	100.3	10.31	0.9698
6	120.4	10.31	0.9696
7	140.5	10.31	0.9695
8	160.6	10.31	0.9694
9	180.7	10.31	0.9695
10	200.8	10.31	0.9695
Mean viscosity, Pa.s			0.9699

Table A 5. Viscosity determination of standard oil No. 12 using a vane at 25°C at a constant shear stress of 0.519 Pa

Observation (#)	Time (s)	Shear Rate (s ⁻¹)	Viscosity (Pa s)
1	20.03	0.5542	0.9365
2	40.13	0.5503	0.9432
3	60.22	0.5516	0.9409
4	80.32	0.5544	0.9361
5	100.4	0.5504	0.9430
6	120.5	0.5515	0.9412
7	140.6	0.5551	0.9349
8	160.7	0.5519	0.9404
9	180.8	0.5524	0.9395
10	200.9	0.5532	0.9382
Mean viscosity, Pa.s			0.9393

Table A 6. Viscosity determination of standard oil No. 12 using a vane at 25°C at a constant shear rate of 0.5188 s⁻¹

Observation (#)	Time (s)	Shear stress (Pa)	Viscosity (Pa s)
1	20.02	0.4838	0.9326
2	50.09	0.4901	0.9446
3	80.16	0.4824	0.9299
4	110.3	0.4924	0.9491
5	140.4	0.4807	0.9266
6	170.5	0.4907	0.9458
7	200.6	0.4832	0.9313
8	230.7	0.4872	0.9390
9	260.7	0.4865	0.9378
10	290.9	0.4846	0.9340
Mean viscosity, Pa.s			0.9370

Table A 7. Viscosity determination of standard oil No. 12 using a vane at 25°C at a constant shear stress of 10 Pa

Observation (#)	Time (s)	Shear Rate (s ⁻¹)	Viscosity (Pa.s)
1	20.02	10.3	0.9709
2	40.08	10.3	0.9703
3	60.14	10.3	0.9701
4	80.21	10.31	0.9701
5	100.3	10.31	0.9697
6	120.4	10.31	0.9695
7	140.5	10.31	0.9695
8	160.6	10.31	0.9694
9	180.7	10.31	0.9695
10	200.8	10.31	0.9695
Mean viscosity, Pa.s			0.9699

Table A 8. Yield stress values at various concentrations of wheat straw for mesh sizes of 40, 20, 12 and 8

Concentration (%, w/v)	Yield stress Pa (40 mesh)	Yield stress Pa (20 mesh)	Yield stress Pa (12 mesh)	Yield stress Pa (8 mesh)
5	— [*]	171	399	411
10	153	195	426	739
15	354	405	516	765
20	462	510	684	1035

^{*} Sedimentation occurred before yield stress could be detected

Appendix B – Yield Stress Values According to Highest Viscosity Values

Figure B 1. Apparent viscosity and shear stress for 15% w/v (40 mesh) wheat straw suspension. Shear stress sweep: 1-600 Pa, Ramp time: 100 seconds

Figure B 2. Apparent viscosity and shear stress for 20% w/v (40 mesh) wheat straw suspension. Shear stress sweep: 1-600 Pa, Ramp time: 100 seconds

Figure B 3. Apparent viscosity and shear stress for 5% w/v (20 mesh) wheat straw suspension. Shear stress sweep: 1-300 Pa, Ramp time: 100 seconds

Figure B 4. Apparent viscosity and shear stress for 10% w/v (20 mesh) wheat straw suspension. Shear stress sweep: 1-300 Pa, Ramp time: 100 seconds

Figure B 5. Apparent viscosity and shear stress for 15% w/v (20 mesh) wheat straw suspension. Shear stress sweep: 1-500 Pa, Ramp time: 100 seconds

Figure B 6. Apparent viscosity and shear stress for 20% w/v (20 mesh) wheat straw suspension. Shear stress sweep: 1-600 Pa, Ramp time: 100 seconds

Figure B 7. Apparent viscosity and shear stress for 5% w/v (12 mesh) wheat straw suspension. Shear stress sweep: 1-700 Pa, Ramp time: 100 seconds

Figure B 8. Apparent viscosity and shear stress for 10% w/v (12 mesh) wheat straw suspension. Shear stress sweep: 1-600 Pa, Ramp time: 100 seconds

Figure B 9. Apparent viscosity and shear stress for 15% w/v (12 mesh) wheat straw suspension. Shear stress sweep: 1-1200 Pa, Ramp time: 100 seconds

Figure B 10. Apparent viscosity and shear stress for 20% w/v (12 mesh) wheat straw suspension. Shear stress sweep: 1-1200 Pa, Ramp time: 100 seconds

Figure B 11. Apparent viscosity and shear stress for 10% w/v (8 mesh) wheat straw suspension. Shear stress sweep: 1-1800 Pa, Ramp time: 100 seconds

Figure B 12. Apparent viscosity and shear stress for 15% w/v (8 mesh) wheat straw suspension. Shear stress sweep: 1-1600 Pa, Ramp time: 100 seconds

Figure B 13. Apparent viscosity and shear stress for 20% w/v (8 mesh) wheat straw suspension. Shear stress sweep: 1-1500 Pa, Ramp time: 100 seconds