# METHODS FOR ANALYZING WALL SLIP IN THE DIE OF A CAPILLARY RHEOMETER AT VARYING OIL CONTENTS

by

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*To God who makes all things possible.*

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# ABSTRACT

Wall slip in the die of a capillary rheometer was investigated. Corn meal and water were mixed to a moisture content of 35% wet basis. Oil was then added at 0%, 2.5%, and 5% of the total mass. A capillary rheometer was used to extrude the mixture at 100C. Three die diameters were studied: 2mm, 4mm, and 8mm. Two length to diameter ratios were studied: L/D=4 and L/D=8. Pressure and flow rate in grams per 30 seconds were collected from the capillary rheometer to perform the Bagley correction, determine the flow behavior index, and correct for slip using the Mooney slip analysis method. Several slip analysis methods were considered prior to the selection of the Mooney method. Overall, the Bagley correction was successful for all die diameters except 2mm. The Mooney method was successful for the 5% added oil content samples. An increase slip velocity was observed as shear stress increased. Mooney plots for 0% and 2.5% resulted in negative shear rate values. An empirical model was developed to predict apparent viscosity of the mixture as a function of total oil content

# 1. INTRODUCTION

## 1.1 Extrusion

Extrusion is when a product is pushed or forced through a die opening. The first extruder patent was reported in 1797 for the fabrication of a lead pipe using a ram to force the lead through a die (Kazemzadeh, 2012). In the food industry extrusion is used for mixing, cooking, forming, and many other processing steps (Berk, 2012). Two major types of extruders used in food production are single-screw and twin-screw.

## 1.2 Single Screw Extruder Scale Down

A small-scale single screw extruder was developed by Purdue University (West Lafayette, Indiana) in partnership with Insta-Pro International (Des Moies, Iowa). The throughput of the smaller scale extruder was designed to be 30kg/hr and was scaled down from a 300 kg/hr extruder. Originally, the project was developed for NASA to process seeds during Advance Life Support systems missions, which occur beyond Low Orbit Earth (Ponrajan, 2015). More recently, Ponrajan (2015) investigated the application of the 30kg/hr single screw extruder for food processing in developing nations. Figure 1.1 shows the extruder screw design used to obtain a throughput of 30kg/hr.

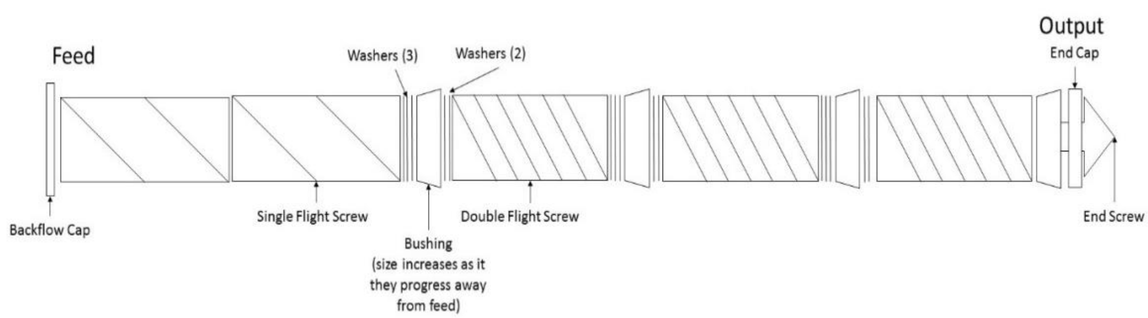


Figure 1.1: Small scale singe-screw extruder schematic (Hauersperger, Tonner, & Okos, 2017).

This work is a continuation of the work done by Penner (2008), Ponrajan (2015), and Tonner (2018) to further develop the understanding of the smaller scale single screw extruder. Penner (2008) reported on geometries and ratios used for the scale down procedure. Ponrajan (2015) focused on finding workable operating conditions and using a capillary rheometer to predict operating conditions for the scaled down extruder. Interest in using an off-line capillary rheometer to predict rheological outcomes of a full-scale extruder continues. A capillary rheometer uses a relatively small amount of product in comparison to a single screw extruder. Tonner (2018) began the development of a finite element model of the extruder and investigated the effect of extrusion conditions on melt viscosity. Both Tonner (2018) and Ponrajan (2015) reported on the rheology of a mixture of corn meal and water. Tonner (2018) and Ponrajan (2015) also both reported a difference between shear rate reported by the rheometer and the actual shear rate as a potential cause of error in their results. Further investigation into the slip occurring in this product is needed to further the development of the finite element extruder model.

## 1.3 Wall Slip

Wall slip is a decrease in the wall shear rate. A migration of larger diameter particles with higher effective viscosity toward the center of the capillary increases the concentration of lower viscosity particles near the wall (Jana, Kapoor, & Acrivos, 1995). The lower viscosity at the wall results in a greater change in velocity over the region, the phenomena is known as wall slip. Figure 1.2 depicts the difference in velocity profile when slip is and is not present.

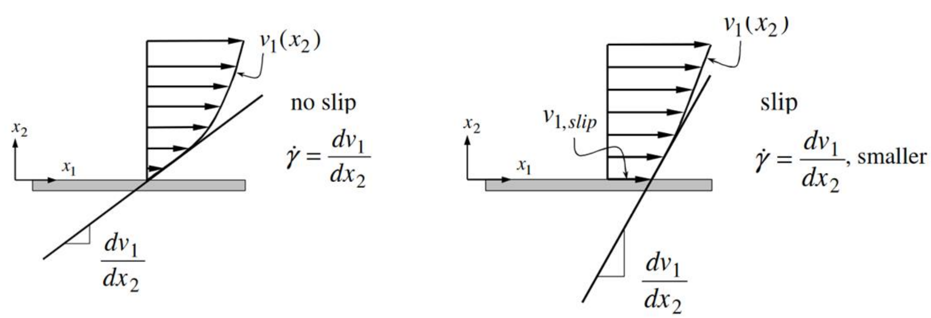


Figure 1.2: Wall slip causes a decrease in wall shear rate (Morrison, 2001).

## 1.4 Problem Statement and Objectives

Wall slip in the die of an extruder can cause large discrepancies between actual shear rate values and those calculated from force. An understanding of the correct method for slip correction is desired. Shear rate correction values for a range of die lengths and diameters at varying operating conditions are needed for the continued development of a finite element model of the 30kg/hr single screw extruder.

Objectives:

1. Determine the appropriate method for wall slip correction.
2. Find shear rate correction for the previously studied mixture of corn meal and water using varying oil contents.

# 2. LITERATURE REVIEW

## 2.1 Extrusion

Extrusion is extensively used in many industries including the production of food and feed. Early examples of extrusion in the food industry consist of forming extrusion to produce pasta or sausage (Levine & Miller, 2007). More recently, extrusion is used for many unit operations such as mixing, puffing, separation, moisture reduction, and cooking (Guy, 2001). Many of the modern uses for extrusion occur under high shear rate and pressure conditions. High shear rate and pressure can cause large changes in structure such as gelatinization and crystallization of starches. An in line rheometer should be used to capture how these structural changes influence the rheological properties of a product (Campanella, Li, Ross, & Okos, 2002). Capillary rheometers are useful for determining viscosities of material at high shear rates similar to those experienced in an extruder. When using a capillary rheometer, the product experiences pressure induced laminar flow through the die (Karnis, Goldsmith, & Mason, 1966). Pressure induced laminar flow is known as Poiseuille flow. For Poiseuille flow in a capillary there are seven main assumptions which can be found in Table 2.1 (Morrison, 2001).

Table 2.1**:** Assumptions for Poiseuille flow in a capillary (Morrison, 2001)

| **Poiseuille Flow in a Capillary** |
|---|
| 1. Flow only occurs in one direction |
| 2. Incompressible fluid |
| 3. Symmetry across the middle of the capillary |
| 4. Variation in the z-direction is negligible |
| 5. Stress tensor is symmetric |
| 6. Pressure change in the z-direction is constant |
| 7. Finite stress at the center of the capillary |

In order to obtain an accurate viscosity from capillary rheometer experiments corrections must be made to data based on the assumptions stated in Table 2.1. This includes corrections for non-Newtonian fluids, entrance effects, and wall effects.

## 2.2 Entrance Effects

Entrance effects are caused by sudden changes to the velocity profile of the fluid as it is extruded from the large diameter reservoir through the much smaller diameter capillary die (Jastrzebski, 1967). The change in velocity profile results in a pressure loss. Change in pressure is used to calculate shear stress. Shear stress is calculated by Equation 2.1 where $\tau_w$ is the wall shear stress in MPa, $\Delta P$ is the change in pressure in MPa, D is the die diameter in mm, and L is the die length in mm. The total applied pressure to the piston can be much greater than the actual pressure drop along the length of the die (Jastrzebski, 1967). If left uncorrected, entrance effects cause elevated shear stress calculations. The Bagley correction is widely used to correct for entrance effects and can be used to calculate both the pressure change due to entrance effects and the theoretical length of the die after entrance effects are considered. Equation 2.1 is applicable to both Newtonian and non-Newtonian fluids.

$$\tau_w = \frac{\Delta P * D}{4 * L} \qquad \text{Equation 2.1}$$

### 2.2.1 Bagley Correction

The Bagley correction adjusts for inaccuracies in calculated wall shear stress by accounting for the pressure losses due to entrance effects (Bagley, 1957). Table 2.2 gives the method for performing the Bagley correction as outlined by Morrison (2001) where $\dot{\gamma}_a$ is the apparent wall shear rate in $s^{-1}$, Q is the volumetric flow rate in $mm^3/s$, R is the radius of the capillary in mm, $\tau_{rz}$ is shear stress in MPa, $\Delta P$ is the change in pressure in MPa, and L is the die length in mm. Pojaran (2015) and Tonner (2018) both found that the Bagley correction can produce erroneous results if product leaks past the rheometer piston back to the capillary reservoir. Tonner (2018) reported that 1 mm of product slip past the piston back to the capillary reservoir can cause a 30% decrease in the wall shear rate. The Bagley correction is performed by plotting the change in pressure versus the length to radius ratio at various shear rates. Each line requires a constant shear rate and a constant capillary die radius. An example of a Bagley plot is shown in Figure 2.1.

Table 2.1: Procedure for Bagley correction (Morrison, 2001)

| |
|---|
| 1. Calculate $\dot{\gamma}_a = \frac{4Q}{\pi R^3}$ |
| 2. Plot ΔP vs. L/R at constant $\dot{\gamma}_a$ |
| 3. Find the y-intercept = $\Delta P_{\text{end effects}}$ |
| 4. $\Delta P_{\text{corrected}} = \Delta P - \Delta P_{\text{end effects}}$ |
| 5. $\tau_{rz} = \frac{\Delta P_{\text{corrected}} R}{2L}$ |

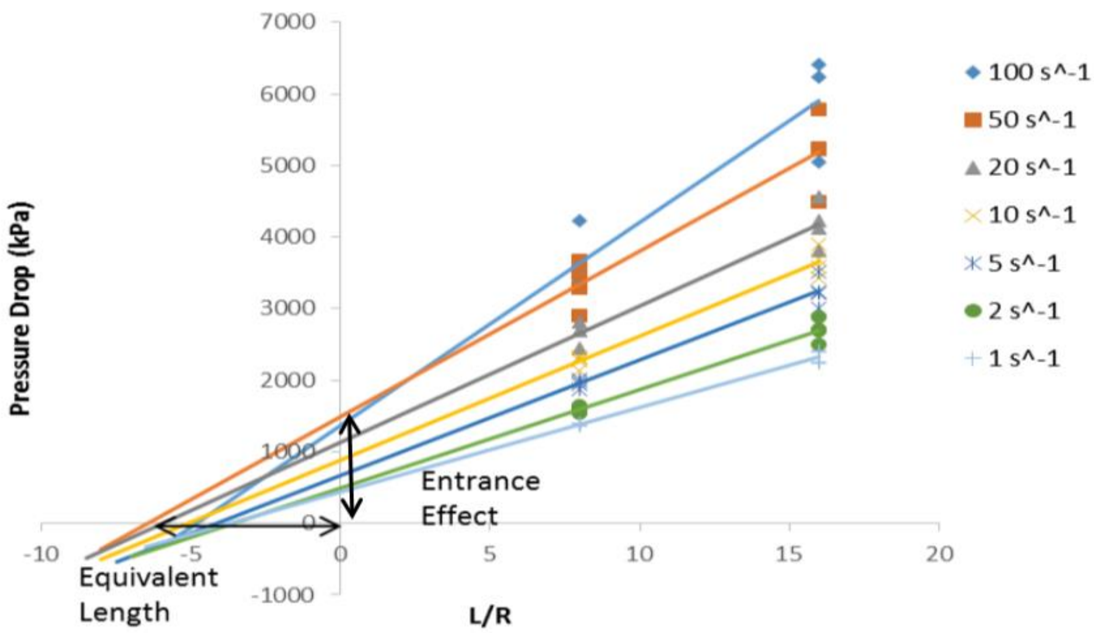


Figure 2.1: Example of a Bagley plot used to correct for entrance effects (Tonner, 2018).

## 2.3 Wall Effect and the Importance of Slip

The assumption of a no-slip boundary condition is prominent in fluid mechanics. Kokini and Derisoglu (1990) showed that rheological data uncorrected for slip can contain errors in the shear rate values that are as large as 70-80%. When dealing with food products, especially those with high moisture and oil contents, slip must be accounted for. When glass capillaries were used to view Poiseuille flow a radial migration of particles towards the center of the capillary was observed (Karnis, Goldsmith, & Mason, 1966). The radial migration of particles caused a concentration of the liquid phase near the wall, which in turn caused a change in velocity profile near the wall

(Karnis, Goldsmith, & Mason, 1966). The change of velocity profile near the wall is the phenomena known as slip.

A significant number of studies have been done on slip during the extrudate of polymers. While polymers are different from food products, these studies can be used to draw conclusions about the causes of slip in a capillary. Benbow and Lame (1963) concluded that instability at the wall originates very near to the entrance of the die and this instability causes slip. They also concluded that the die material plays an important role in development of slip (Benbow & Lamb, 1963). Another contributing factor to slip velocity is the geometry of the die. Several studies have concluded that slip velocity is dependent on the length to diameter ratio of the rheometer die (Hatzikiriakos & Dealy, 1992) (Kalika & Denn, 1987).

### 2.3.1 Product Quality

Consistent product quality is important in all manufacturing processes. Wall slip during extrusion changes the physical appearance of the extrudate. Without the presence of slip, extrudate appears smooth. Stick-slip is caused by the extrusion of a non-homogeneous mixture ( Rodríguez-González, Pérez-González, Marín-Santibáñez, & de Vargas, 2009). Stick-slip is visibly characterized by intermixed areas of shark-skin and smooth extrudate. Slip causes the surface of extrudate to become visibly rough, often known as the shark skin effect (Denn, 2001). Both the stick -slip and the shark skin effects must be studied immediately after product exits the die (Piau, El Kissi, & Tremblay, 1990). Product relaxation or shrinking long after it exits the die can also cause extrudate to take on the appearance of stick-slip or shark skin (Piau, El Kissi, & Tremblay, 1990). Figure 2.2 shows product with no slip, figure 2.3 shows product experiencing stick-slip, and figure 2.4 shows the full shark skin effect that is caused by wall slip. Figures 2.2-2.4 were taken from Pudjijanto and Denn (1994).

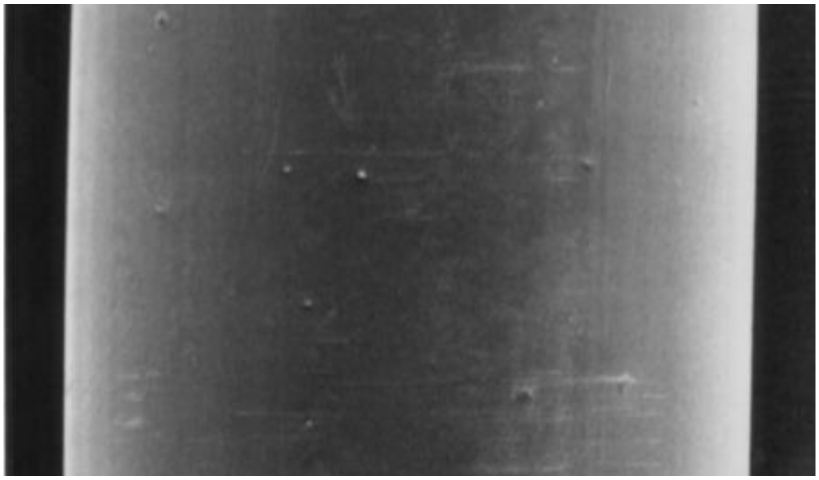

Figure 2.2: No slip during extrusion of polyethylene (Pudjijanto & Denn, 1994).

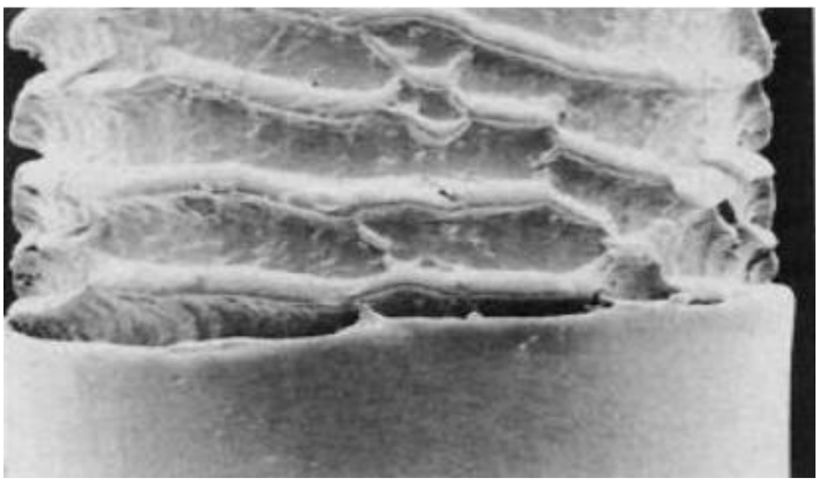

Figure 2.3: Stick-slip during extrusion of polyethylene (Pudjijanto & Denn, 1994).

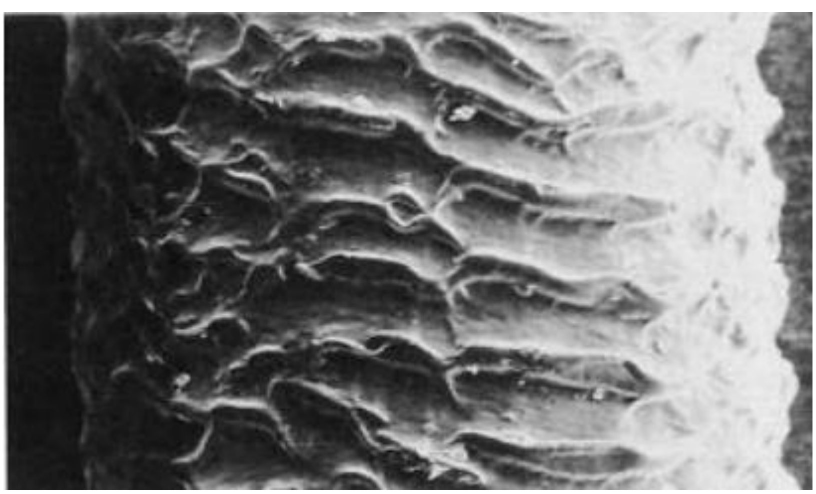

Figure 2.4: Shark skin during extrusion of polyethylene (Pudjijanto & Denn, 1994).

## 2.4 Mooney Slip Analysis Method

Apparent shear rate is calculated by Equation 2.2 where $\dot{\gamma}_a$ is the apparent wall shear rate in $s^{-1}$, Q is the volumetric flow rate in $mm^3/s$, and R is the radius of the capillary in mm. Slip at the wall causes a disparity between the Q value calculated from piston velocity and the actual flow rate coming out of the capillary rheometer. Mooney (1931) was the first to propose a method to correct shear rate values for slip occurring at the wall. He proposed that slip is caused by a larger velocity gradient in a layer at the wall that is small in comparison to the diameter of the capillary (Mooney, 1931). The Mooney slip analysis method plots shear rate versus the inverse of the squared radius. Each line on the plot must be at a constant shear stress and consist of data from dies with the same length to diameter ratios (Morrison, 2001).

$$\dot{\gamma}_a = \frac{4*Q}{\pi*R^3} \qquad \text{Equation 2.2}$$

### 2.4.1 Assumptions

The method published by Mooney (1931) makes the broad assumption that only fluidity and slip affect the measurements. Mooney (1931) also did not account for entrance or exit effects and assumed a Newtonian fluid. The Mooney-Rabinowitsch relation was developed in 1929 as a correction for the Newtonian assumption made in many rheological models (Rabinowitsch, 1929). The Mooney-Rabinowitsch relation uses the slope from a log-log plot of shear stress versus shear rate to determine the behavior index of the material (Rabinowitsch, 1929). However, this relation assumes a time independent laminar flow and that there is no slippage at the wall (Jastrzebski, 1967). It is common practice to perform a correction to the shear rate, that is a slip correction, prior to correction for the Newtonian fluid assumption.

Jastrzebski's (1967) execution of the Mooney slip analysis method uses the Bagley correction for entrance effect, compares dies of the same length to diameter ratio, and also performs the correction for non-newtonian velocity profiles. The well executed application of all the mentioned corrections means the study can be used as a model for experimental and data analysis procedures.

## 2.5 Other Slip Analysis Methods

The Mooney slip analysis method requires a high quantity of experiments meaning it is very time intensive. Because of the time intensive nature of performing the Mooney slip analysis procedure, many studies have been done to try and find alternative ways to correct for slip with varying levels of success. Many studies also obtain physically impossible negative shear rates from the Mooney slip correction and search for alternatives for that reason (Chuan & Zara, 2014).

A simple variation on the Mooney procedure was proposed by Ren, Huang and Liu (2016). They proposed that by having a fixed die diameter and correcting for length the large experimental load from the traditional Mooney technique can be overcome (Ren, Huang, & Liu, 2016). The proposition that performing experiments at different length to diameter ratios goes against the geometric dependency of the Mooney correction as reported by Morrison (2001). Adding an additional correction for die geometry adds a layer of complexity to the already significant amount of corrections needed to obtain rheological values from capillary rheometer experiments on non-Newtonian fluids. Hatzikiriakos and Dealy (1991) attempted to study slip without the influences of entrance effects or a pressure gradient. They accomplished this using a sliding plate rheometer at steady shear and a critical shear stress for the slippage of polyethylene was found (Hatzikiriakos & Dealy, 1991). Rapid and unpredictable deformations led them to conclude a dynamic model was needed for the evolution of slip throughout the process (Hatzikiriakos & Dealy, 1991). A finite element model of a capillary rheometer was developed by Chuan and Zara (2014) to analyze wall slip. As a result of the finite element wall it became apparent that at high levels of slip the Cox-Mertz rule provides shear viscosity that is higher than complex viscosity (Chuan & Zara, 2014). This highlights another important reason to correct for slip prior to calculating viscosity. However, their study did not account for back mixing that happens at the rapid diameter change from the reservoir to the capillary die or for potential slippage on the sides of the piston. Further development of the finite element model would need to be done in order to have a true predictor for slip analysis.

Potentially the most accurate way to obtain the velocity profile inside a capillary rheometer is with Nuclear Magnetic Resonance (NMR). Gibbs *et al.* (1996) used a phase-based NMR technique to track the velocity of different slices within the capillary. Slip velocities obtained from the NMR technique matched those from performing the Mooney slip analysis (Gibbs, et al., 1996). NMR would be a good alternative to the Mooney slip analysis method if an NMR is available.

Although some studies obtained physically impossible negative shear rates from the Mooney correction, it appears that, as long as assumptions are met, the Mooney method is an appropriate method for calculating slip velocity and correcting for wall shear rate.

## 2.6 Conclusion

While others have tried to develop their own methods for analyzing slip, the Mooney slip method outlined by Jastrzebski (1967) is the most mathmatically sound. Many have struggled to perform the Mooney slip analysis method and have left the correction out of their calculations. While performing a correction for slip is experimentally tedious, the large impact of the correction on rheolocigal calulations makes it necessary. Slip correction values for a mixture of corn meal, oil, and water have not been found in literature and are necessary for the development of a reliable model of an extruder.

# 3. MATERIALS AND METHODS

## 3.1 Materials

Degermed, dehulled fine yellow cornmeal (M77 fine yellow cornmeal) donated by Agricor, Inc. was used in this study. The proximate composition and particle size provided in the certificate of analysis can be found in Table 2.1.1 and Table 2.1.2. Mazola corn oil was purchased and used to vary the oil content of the cornmeal.

Table 3.1 Proximate composition of cornmeal used in this study.

| **Component** | **Percentage** |
|---|---|
| Moisture | 12.60% |
| Fat | 0.94% |
| Protein | 5.83% |

Table 3.2 Particle size of cornmeal used in this study based on ability to pass through sieve size.

| **Granulation** | **Percentage** |
|---|---|
| Held on US #30 | 0.00% |
| Held on US #40 | 30.12% |
| Held on US #80 | 66.10% |
| Through US #80 | 3.76% |

## 3.2 Sample Preparation

Cornmeal was removed from the storage cooler and allowed to come to room temperature. A rapid moisture analyzer (Mettler Toledo) was used to determine the moisture content of the cornmeal. Water was then added using a benchtop laboratory mixer (KitchenAid Mixer) to increase the moisture content to 35% wet basis. The moisture content of 35% wet basis was chosen based on previous work by Ponrajan, 2016 and Tonner, 2018. Corn oil was added after sample was increased to 35% w.b. moisture content. Oil was added at 0%, 2.5%, and 5% of the moisture adjusted mass. Prepared samples were placed in large plastic storage bags with the air pressed out and allowed to equilibrate in the storage cooler overnight. Samples were removed from the cooler and allowed to return to room temperature before running them through the capillary rheometer.

Moisture content was verified using a rapid moisture analyzer after sample had equilibrated to room temperature.

### 3.3 Capillary Rheometer

A Rosand RH2000 twin bore capillary rheometer was used in this study. The rheometer was equipped with a 3000 psi pressure transducer on the left bore and a 1500 psi transducer on the right bore. Figure 3.1 shows the capillary rheometer. Nine different die diameter and length combinations were used and can be found in Table 2.3.1. Piston velocities of 5, 10, 20, 50, 75, and 100 mm/min were tested to achieve the requirements of constant sheer rate and sheer stress for the Bagley end effects correction and the Mooney slip analysis method. The Bagley correction requires different L/D at constant sheer rate (Morrison, 2001). Die with the same L/D at constant sheer stress are required for the Mooney slip analysis method (Morrison, 2001).


Figure 3.1: Capillary rheometer (Malvern Panalytical, 2019)

Table 3.3 Length and diameter of dies used in this study. The Bagley correction requires different L/D at constant sheer rate (Morrison, 2001). Die with the same L/D at constant sheer stress are required for the Mooney slip analysis method (Morrison, 2001).

| Length | Diameter | L/D |
|---|---|---|
| 0.25 | 2 | 0.13 |
| 0.25 | 4 | 0.06 |
| 0.25 | 8 | 0.03 |
| 4 | 2 | 2 |
| 8 | 4 | 2 |
| 16 | 8 | 2 |
| 8 | 2 | 4 |
| 16 | 4 | 4 |
| 32 | 8 | 4 |

### 3.3.1 Pressure Calibration

Prior to running any experiments, the rheometer needed calibration. Calibration was accomplished using a dead weight tester over the range of 0 psi to 1500 psi. Due to load restrictions on the dead weight tester higher pressures could not be calibrated. The rheometer was set to stop the experiment if the pressure reached a value higher than the calibration limit.

### 3.3.2 Procedure

Sample was removed from the cooler three hours before running any experiments to allow adequate time for it to reach room temperature. At the same time point, the capillary rheometer was turned on and set to 100°C. Once equilibrium had been reached, dies were attached, and each bore was filled with sample. A plug and hammer were used to lightly compact the sample to assure there were no major air gaps.

To prevent the buildup of product from interfering with results, the rheometer was thoroughly cleaned with a solution of soap and water between each run. The water was allowed to boil inside each bore and a brush was used to dislodge any particles. After the soapy water was drained, the bores were dried, and new dies were attached.

### 3.3.3 Data Collection

Each run was performed at a constant piston velocity, which gives a constant shear rate. Pressure was recorded by the rheometer every two seconds for the duration of the run. The flow rate was calculated based on the piston velocity and diameter of the capillary. Flow rate was also measured by massing extrudate over a time period of 30 seconds.

## 3.4 Analysis Methods

Sheer stress was calculated with the recorded pressure data using Equation 3.1 where $\tau_w$ is the wall shear stress, $\Delta P$ is the change in pressure, D is the die diameter, and L is the die length. Apparent wall shear rate was calculated using Equation 3.2 where $\dot{\gamma}_a$ is the apparent wall shear rate, Q is the volumetric flow rate, and R is the radius of the capillary. Pressure readings for each run were averaged over the duration extrudate was flowing from the capillary rheometer.

$$\tau_w = \frac{\Delta P * D}{4 * L} \qquad \text{Equation 3.1}$$

$$\dot{\gamma}_a = \frac{4 * Q}{\pi * R^3} \qquad \text{Equation 3.2}$$

To account for the material being non-Newtonian, the Weissenberg-Rabinowitsch correction was applied using Equation 3.3 where $\dot{\gamma}_w$ is the wall shear rate and the power law index n is as defined in Equation 3.4.

$$\dot{\gamma}_w = \left(\frac{3n+1}{4n}\right) * \dot{\gamma}_a \qquad \text{Equation 3.3}$$

$$n = \frac{d(\ln(\tau_w))}{d(\ln(\dot{\gamma}_a))} \qquad \text{Equation 3.4}$$

Apparent viscosity was then calculated as wall shear stress divided by wall shear rate, as shown in Equation 3.5 where $\eta$ is apparent viscosity.

$$\eta = \frac{\tau_w}{\dot{\gamma}_w} \qquad \text{Equation 3.5}$$

### 3.4.1 Bagley Correction

The capillaries used in this study were too short to ignore velocity variations that come with developing flow. Therefore, the Bagley correction was used to correct for the entrance and exit effects caused by the development of flow. Pressure transducers in the capillary rheometer were located at the bottom of the reservoir right before the entrance to the die. The location of the pressure transducers inside the capillary eliminates the need to account for a change in pressure over the barrel (Morrison, 2001). Bagley plots were made by plotting the change in pressure versus the length to radius ratio of the capillary. Each line in the plot is required to be at a constant shear rate. The y-intercept of the line is the pressure correction for the end effects and was subtracted from the measured change in pressure. The corrected change in pressure was used to calculate shear stress. An example of a Bagley plot for 0% oil added can be seen in Figure 3.2. A pressure correction of 1.2408MPa is needed for a shear rate of 0.0096 $s^{-1}$ and a correction of 2.7782 MPa is needed for a shear rate of 0.048 $s^{-1}$

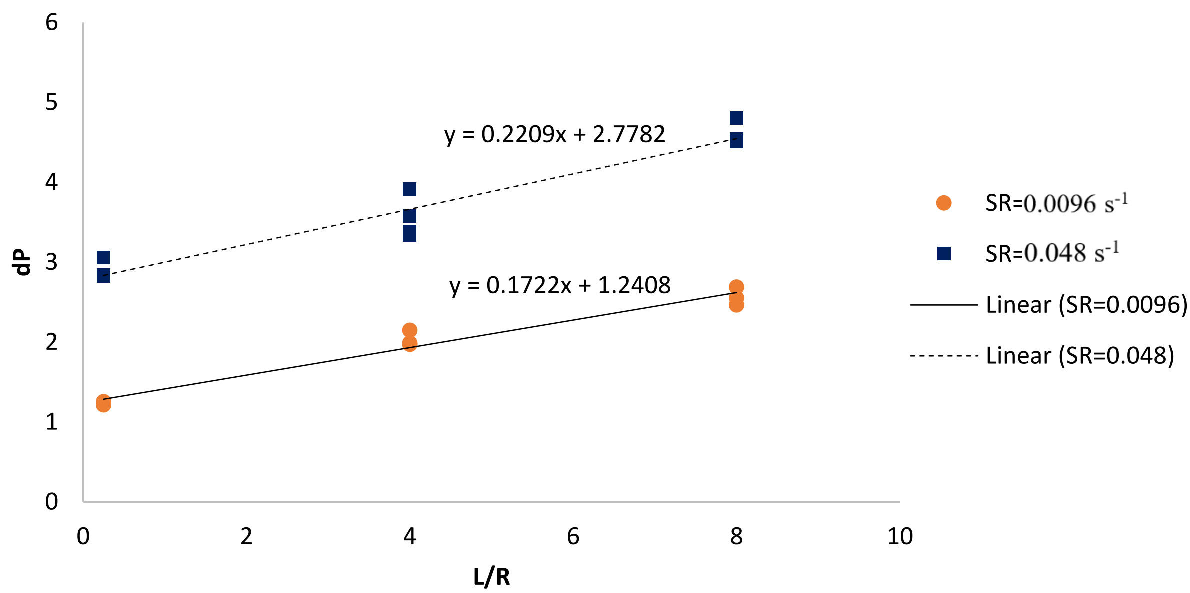


Figure 3.2: Example of a Bagley correction plot.

### 3.4.2 Mooney Slip Analysis

A no-slip condition was assumed at the capillary wall in the Weissenberg-Rabinowitsch correction. However, slip does occur throughout this study and the Mooney slip analysis method was performed to correct the changes in apparent shear rate due to slip velocity. Correcting the wall shear rate is necessary to obtain the apparent viscosity.

Mooney slip analysis was done using the shear stress calculated from the Bagley corrected change in pressure. Apparent shear rate was plotted versus the inverse of the capillary radius. Each line is required to be at a constant shear stress. Since it is difficult to obtain a constant shear stress under the operating conditions, a small range was used for each line. De Vargas, 1993 found slip velocity also changed with capillary length to diameter ratio. Many other studies also found a similar result (Morrison, 2001), so analysis was performed using dies of the same length to diameter ratio. The y-intercept of the plot is the apparent shear rate corrected for slip. An example of the plot used to obtain slip corrected shear rate can be seen in Figure 3.3.

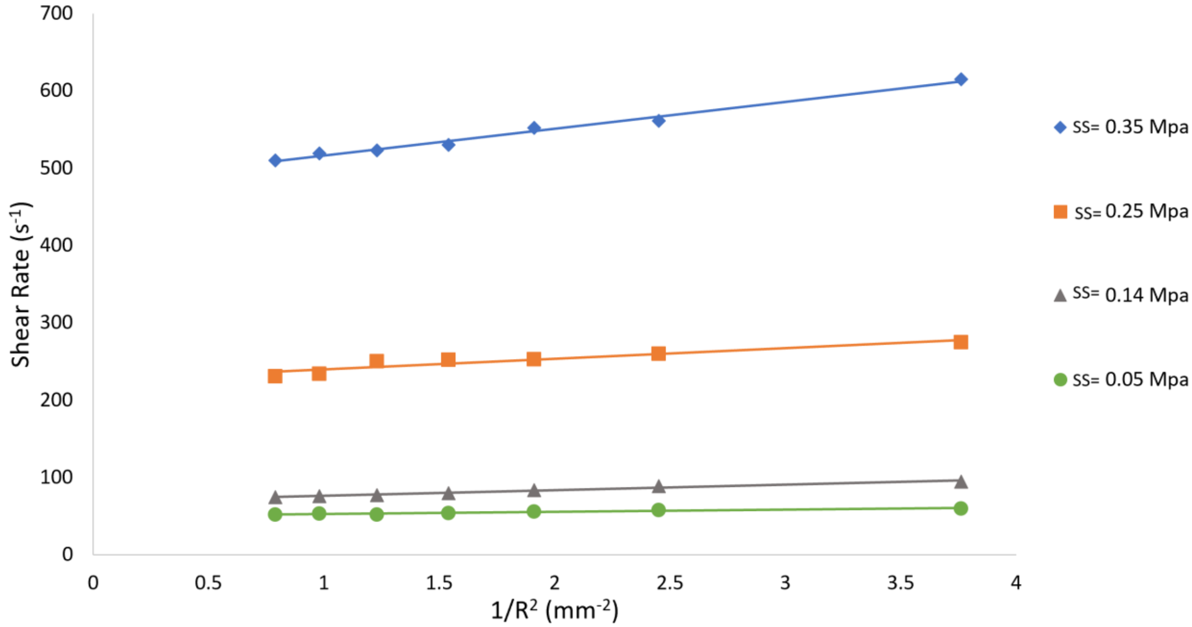


Figure 3.3: Example of a plot for Mooney slip analysis.

# 4. RESULTS AND DISCUSSION

## 4.1 Bagley Correction

The Bagley correction was applied to correct for entrance effects by adjusting the change in pressure over the capillary die. End effects occur because of the sudden and rapid change in diameter from the reservoir to the die. At long die lengths the Bagley correction becomes negligible. Because of the location of the pressure transducers, and the short die length, the Bagley correction had to be performed at each oil content. Figures 4.1-4.3 show the Bagley plots used. Figure 4 shows a comparison of the Bagley plots for all three different oil contents at the same shear rate. The Bagley correction for each shear rate consistently increased as oil content increased. Meaning, as oil content increases it takes longer for flow to become fully developed.

Ponrajan (2015) and Tonner (2018) both reported issues with obtaining negative viscosities after performing the Bagley correction. In this study, only did the smallest die diameter, 2mm, had issues with the Bagley correction. Sometimes the correction to the pressure was greater than the actual pressure drop over the die. It should be noted that the smaller diameter dies only had a failed Bagley correction at the highest piston speeds tested, 50, 75, 100 mm/min. Highest piston speeds in combination with the lowest diameter created the highest pressures obtained in this study (3-5 Mpa). When the pressure in the capillary rheometer reach that level the standard issue O-rings shred throughout the process. When this happens product can migrate back up the piston causing a difference between calculated shear rate and actual. The compromised seal also lowers the pressure change reported by the rheometer. According to Tonner (2018), the pseudo shear rate can be lowered by as much as 30% by as little as 1mm of product slippage back up the capillary. In the future, a softer O-ring should be used. The silicone O-ring used in this study had a hardness of 70. Harder O-rings tend to shred and break at lower pressures than the softer ones.

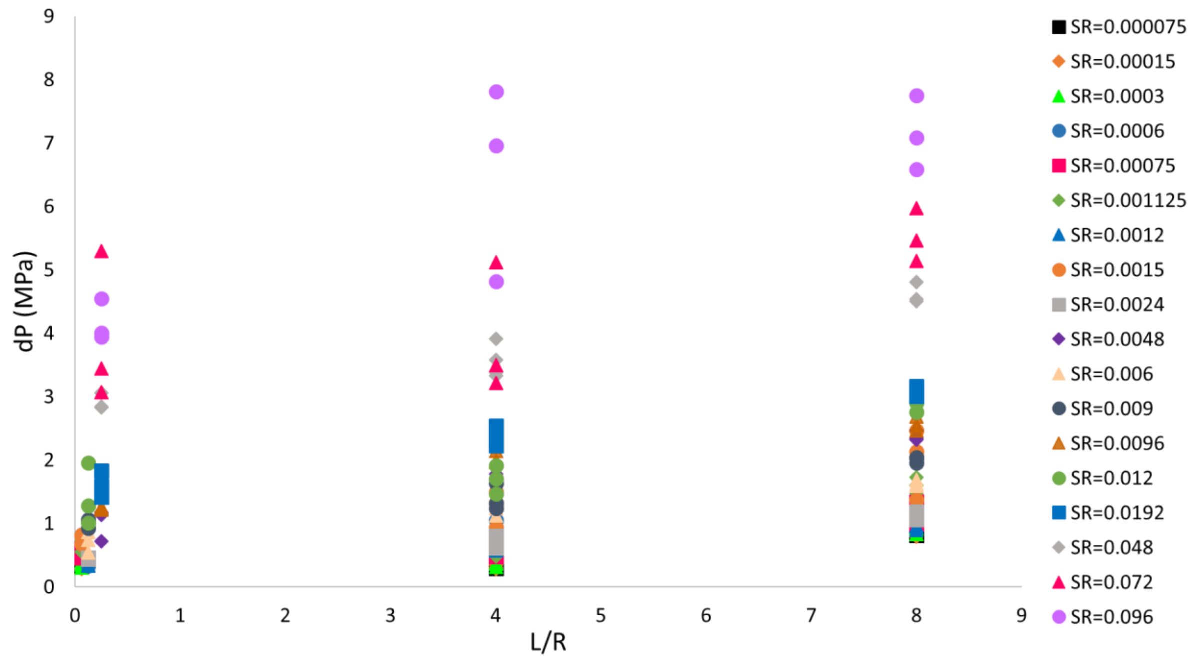


Figure 4.1: Bagley plot for 0% added oil.

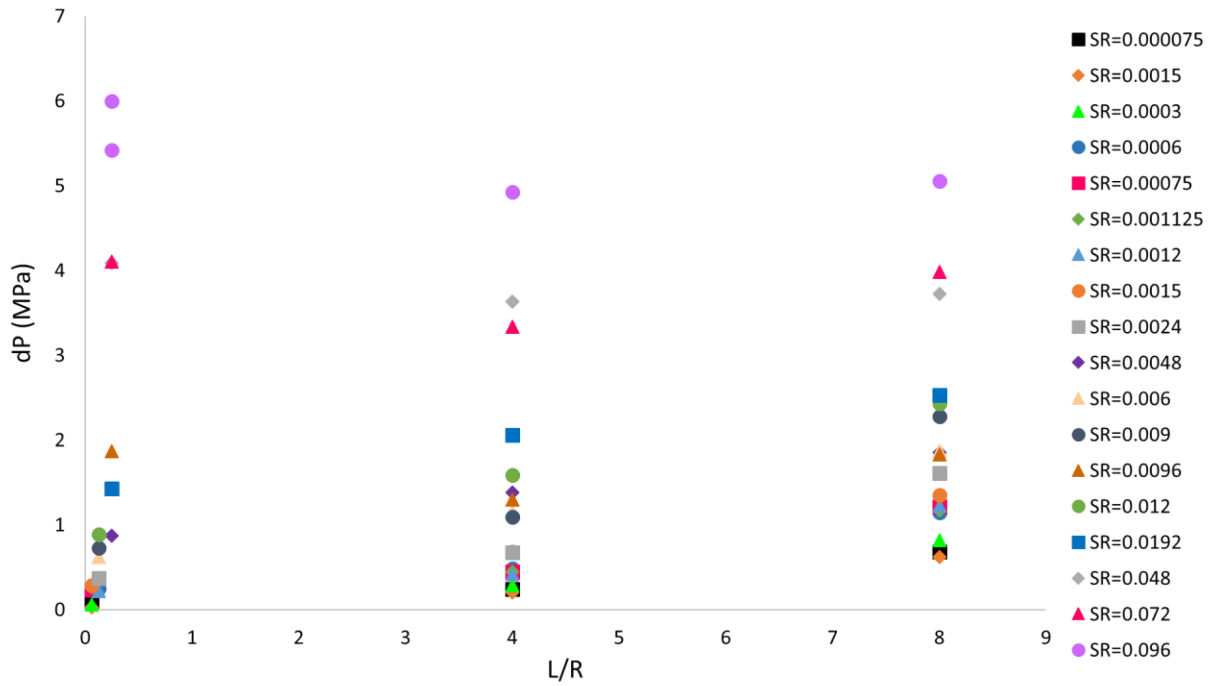


Figure 4.2: Bagley plot for 2.5% added oil

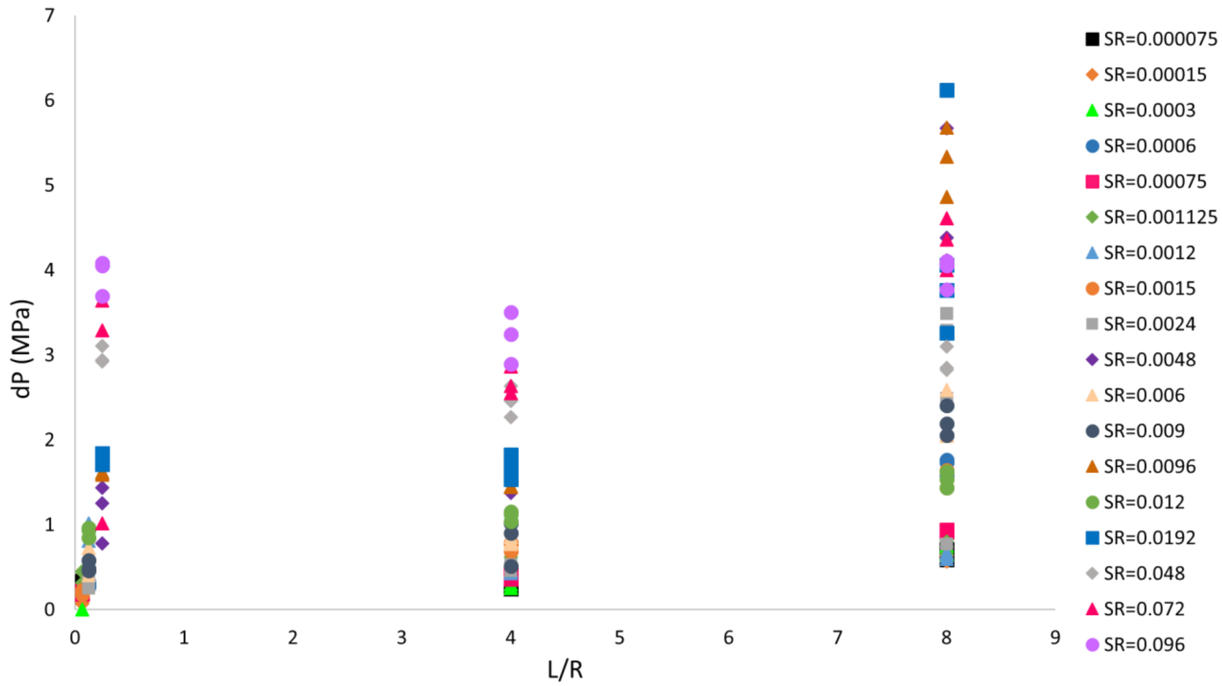


Figure 4.3: Bagley plot for 5% added oil

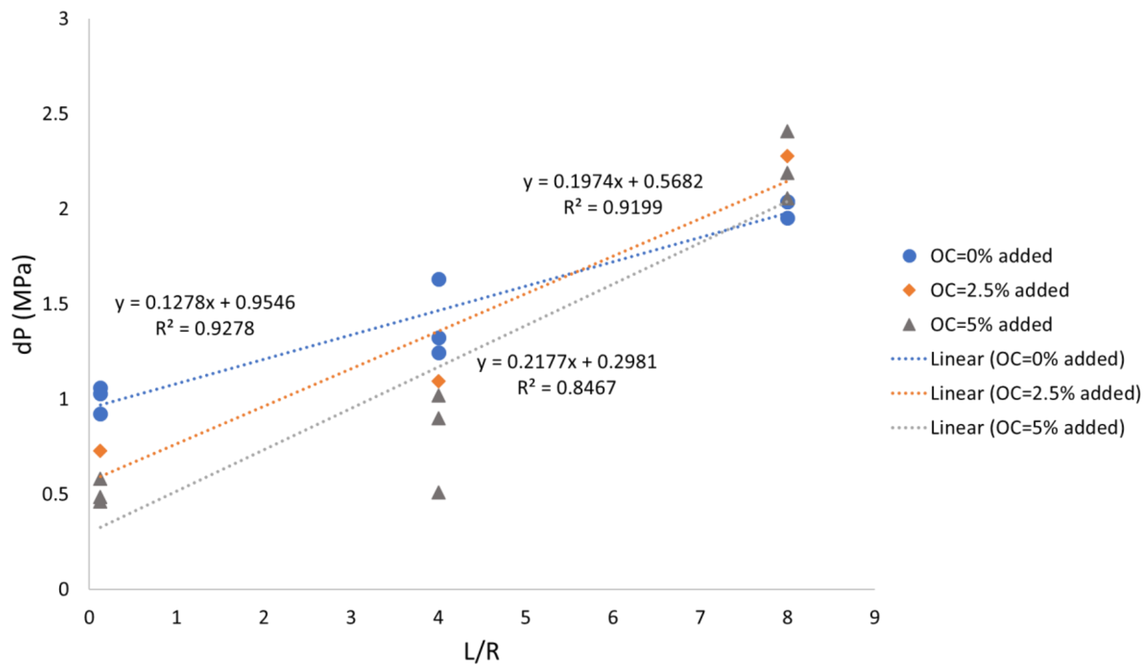


Figure 4.4: Bagley plot of all oil contents at a shear rate of 0.009 s^-1

Table 4.1: Comparison of Bagley correction at a shear rate of 0.009 $s^{-1}$

| Oil Content [%] | Bagley Correction |
|---|---|
| 0 | 0.1278 |
| 2.5 | 0.1974 |
| 5 | 0.2177 |

## 4.2 Flow Behavior Index

After the Bagley correction was performed, plots were made to find the flow behavior index of the product. These plots can be found in Figures 4.5-4.7. Because of the low range of shear rates studied, taking the natural log created negative values. It should also be noted that if you follow the trajectory of this plot, high shear rates would create products with very small apparent viscosities. I can be seen that in some circumstances the Bagley correction had the opposite of the intended effect on the viscosity versus shear rate plots. Correction should bring the lines closer together and improve the correlation. However, because of the elevated correction values from O-ring breakage R^2 values decreased from around 0.97 to as low at 0.79.

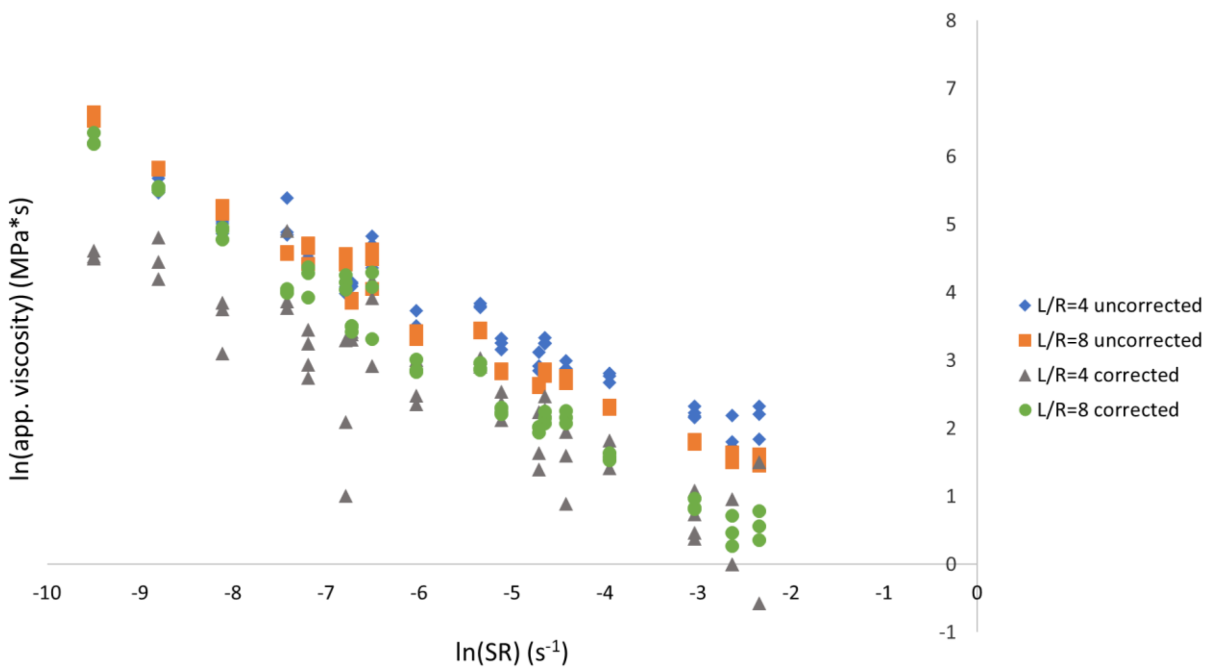


Figure 4.5: Log-log plot of apparent viscosity versus shear rate at 0% oil added.

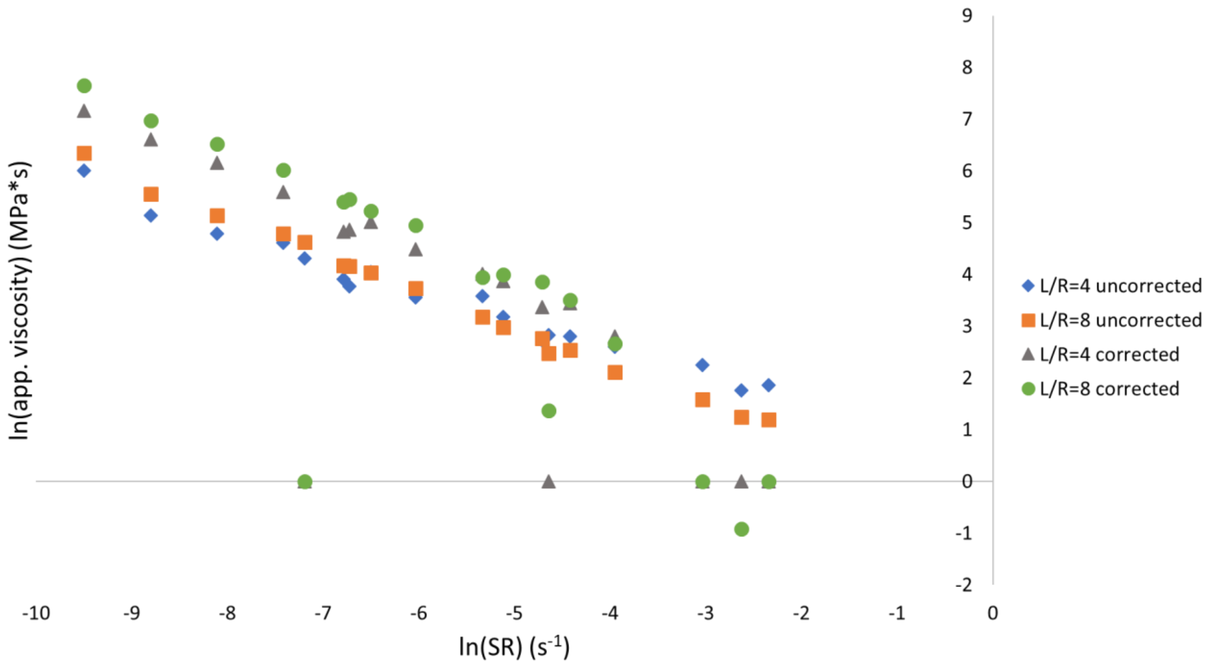


Figure 4.6: Log-log plot of apparent viscosity versus shear rate at 2.5% oil added.

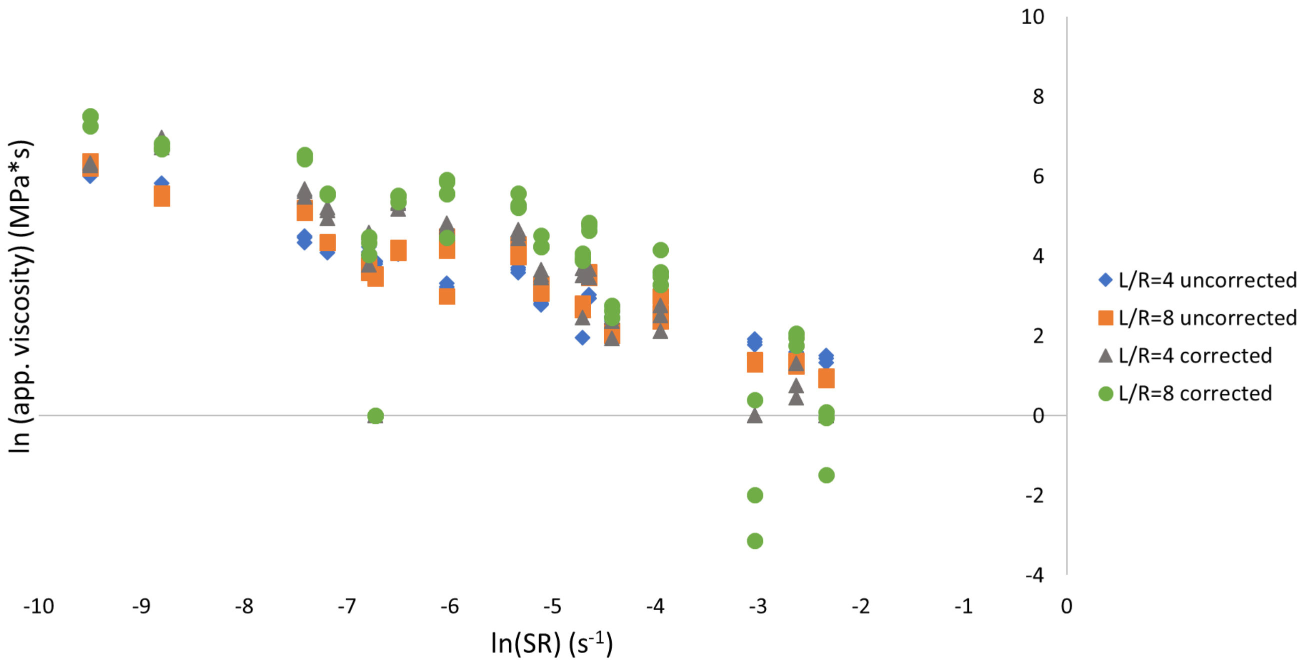


Figure 4.7: Log-log plot of apparent viscosity versus shear rate at 5% oil added.

## 4.3 Mooney Slip Analysis

The change in pressure versus the diameter was plotted to make sure slip was occurring before proceeding with the Mooney correction. If no slip occurred, there would be no difference in pressure between the three tested diameters. The downward trend in pressure seen as diameter increases indicates slip is occurring. While the trend is subtle at 0% oil content added, it becomes more apparent as added oil content increased. This downward trend can be seen in Figures 4.8-4.10 for both length to radius ratios. Figures 4.11 and 4.12 show plugs that were removed from the rheometer after a sample was run. They show a concentration of oil at the wall of the capillary, especially at the bottom of the plug that was closest to the rapid and sudden change in diameter at the entrance of the capillary die. This agrees with Mooney (1931) and Karnis, Goldsmith, & Mason (1966) who both concluded that slip occurs because of an elevated concentration of the liquid phase at the wall of the capillary. Figure 4.13 further supports the evidence of slip because a shark-skin effect can be seen on the extrudate. Piau, El Kissi, and Tremblay (1990) reported on wall slip causing a shark-skin effect on extrudate.

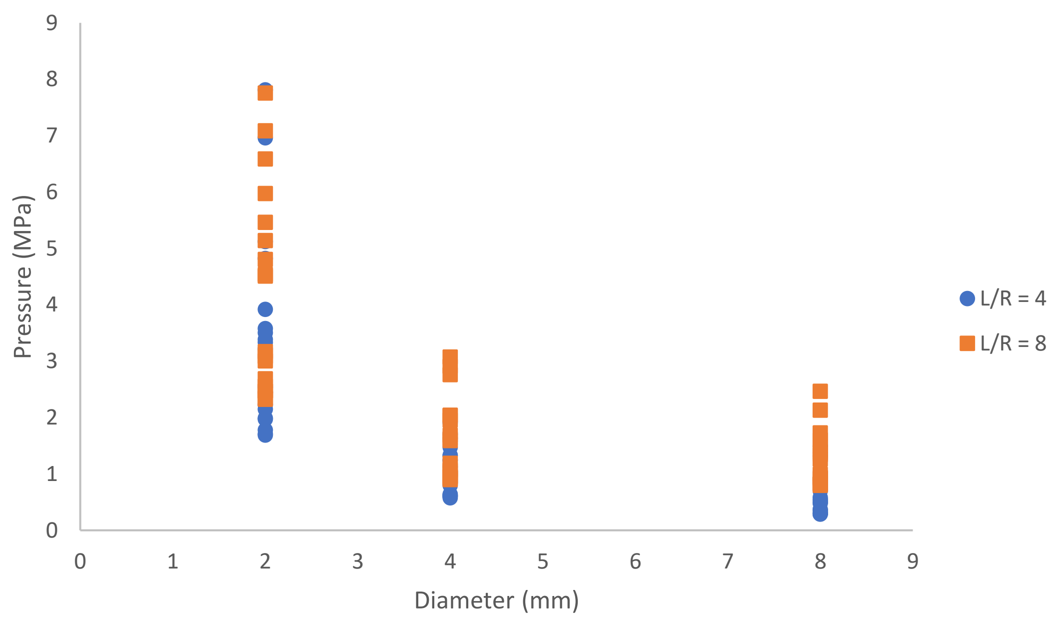


Figure 4.8: Plotting change in pressure versus diameter shows slip is happening even at 0% added oil content.

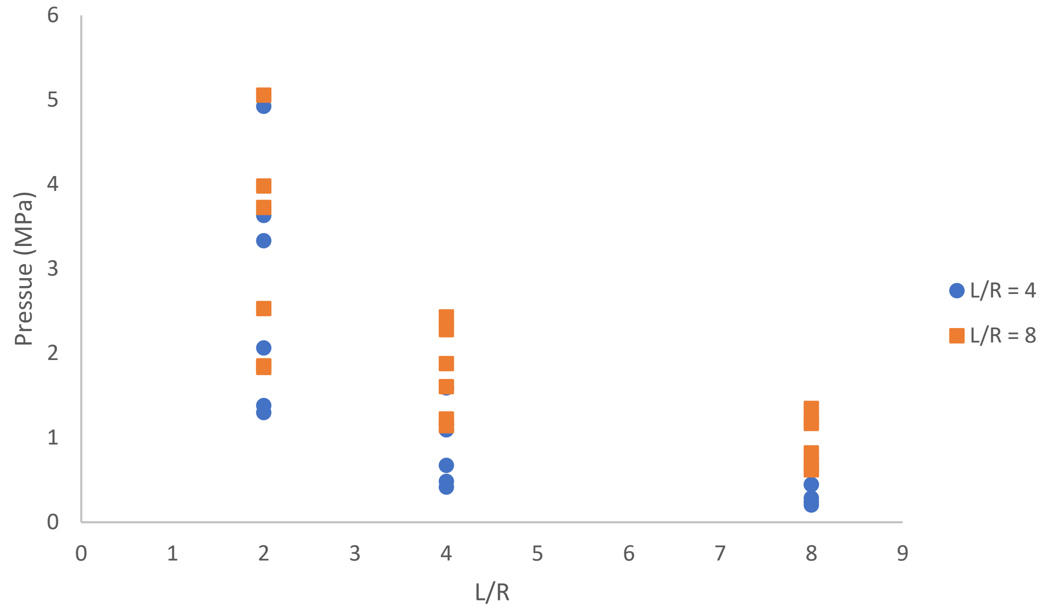


Figure 4.9: Plotting change in pressure versus diameter shows slip is happening at 2.5% added oil content.

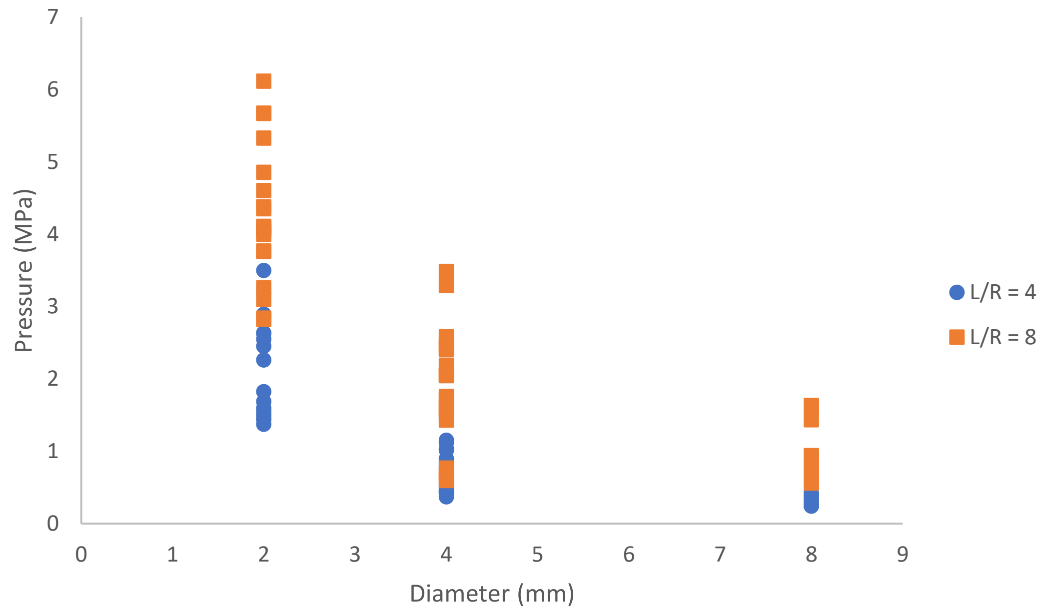


Figure 4.10: Plotting change in pressure versus diameter shows slip is happening at 5% added oil content.

Figure 4.11: Plug removed from capillary rheometer after 2.5% oil content added run. Oil collection near the bottom of the plug is apparent.

Figure 4.12: Plug removed from capillary rheometer after 2.5% oil content added run. There is a visible ring of higher oil content near the outside of the plug.

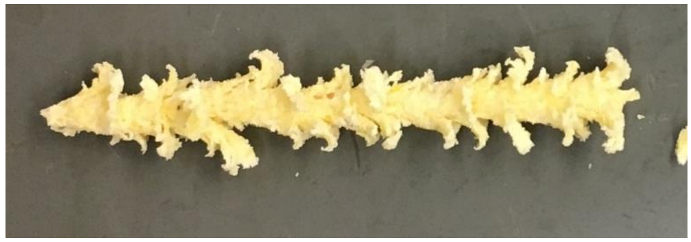

Figure 4.13: Shark-skin effect on extrudate. Piau, El Kissi, & Tremblay (1990) attribute this phenomena to slip.

Since it was apparent from the change in pressure versus diameter plots that slip was occurring, the Mooney procedure was performed as outlined by Jastrzebski (1967). Sear rate versus shear stress was plotted for each of the dies. Figure 4.14 shows the shear rate versus shear stress plot for the die with length 8 mm and diameter 4 mm and the die with length of 16 mm and diameter of 4 mm. R^2 values of 0.85 and 0.89 were recorded respectively. Jastrzebski (1967) used this best fit line to predict shear rate values at constant shear stresses. This method reduces the number of experiments needed to obtain the wall slip correction. However, without a R^2 value of 1, using a line of best fit to predict shear rate values that were then corrected may result in erroneous results.

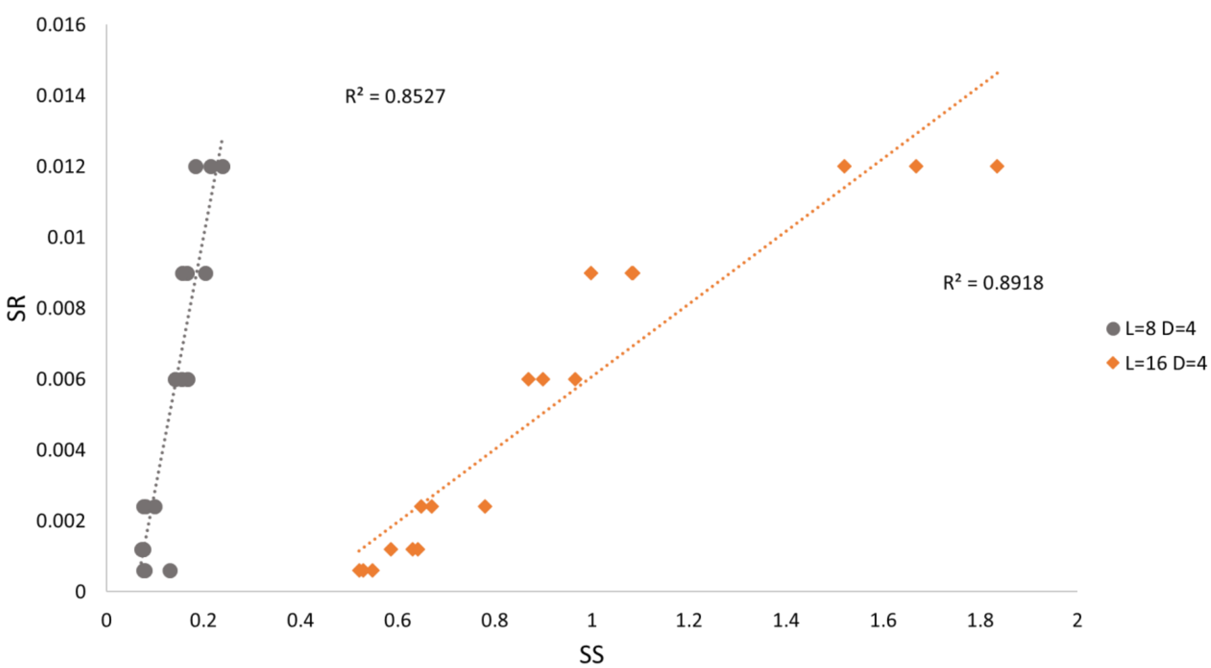


Figure 4.14: Plot example to find shear stress values for the Mooney analysis.

Once lines of best fit were obtained for each die values of shear stress were chosen, and the lines of best fit were used to calculate shear rate. The intercept of each line is the shear rate correction for the specified die at the chosen shear stress. The slope of each line is 4 times the slip velocity at the given conditions. Data for dies with a 2mm diameter was left out of the Mooney calculation for 5% oil added samples. The line of best fit for shear rate versus shear stress gave a negative slope. Meaning shear rate decreased as shear stress increased. When pressure increases the amount of flow should also increase, they should not have an inverse relationship for this material. This issue may have been caused by problems with the shortest 2mm die. Product would get caught in narrow exit region causing the die to behave like a 6mm die that had a 2mm opening. Most likely this occurred due to die swell.

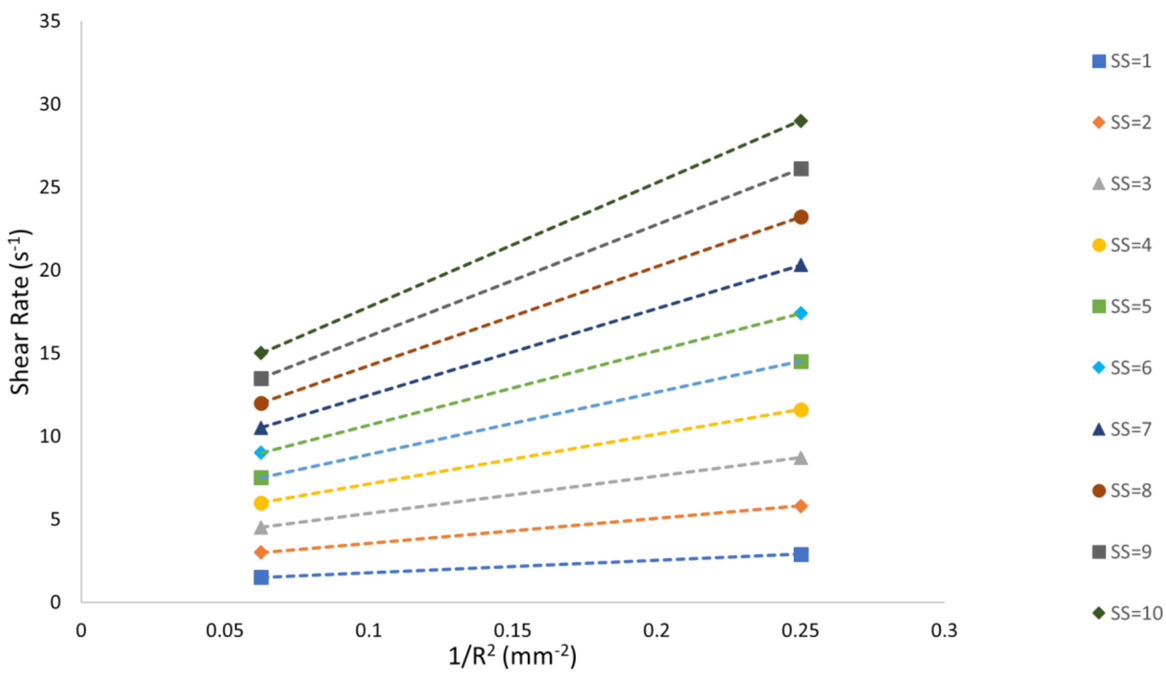


Figure 4.15: Mooney plot for an added oil content of 5% and L/R=4. Shear stress is in Pa-s.

The Mooney correction for 0% and 2.5% oil added returned negative values for shear rate correction. Similar issues were reported by Rides *et al.* (2007), Chaun and Zara (2014), and Lanteri *et al.* (1996). Cornfield *et al.* (1999) reported that small inaccuracies in die measurements can have a monumental impact on the Mooney correction. The roughness of the material can also impact the slip velocity (Halliday & Smith, 1995). Both die dimensions and roughness of material could have had an impact on the Mooney slip analysis. Not all of the dies used were cut with precision machinery and some were made out of different types of metal.

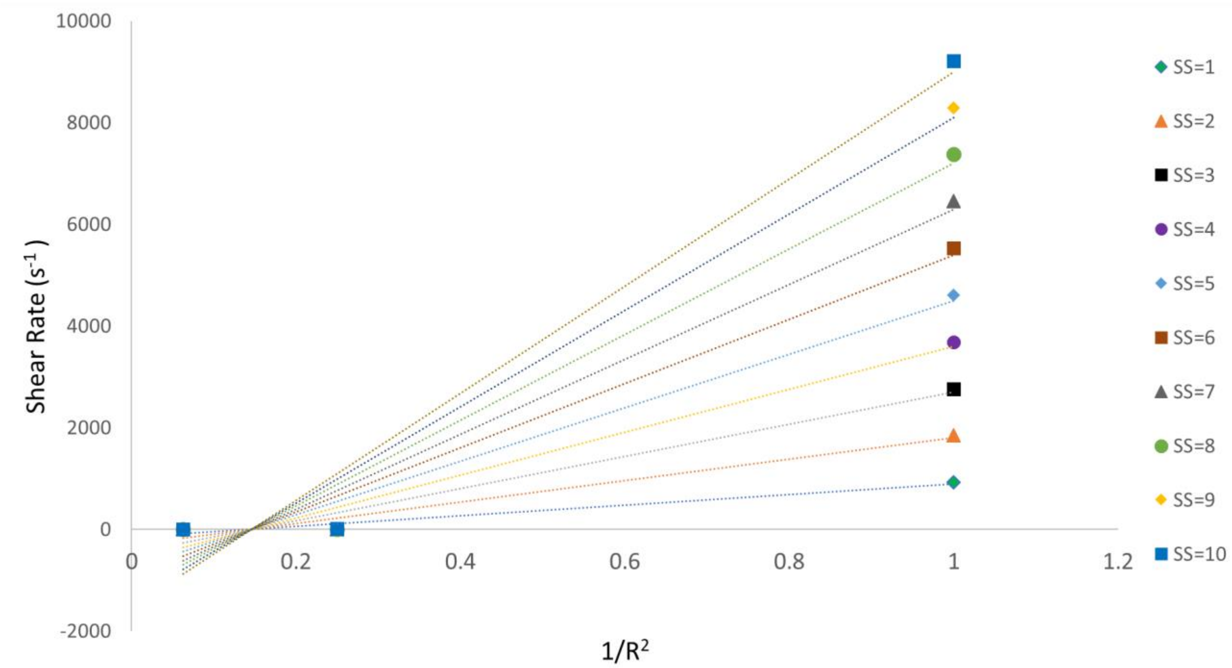


Figure 4.16: Mooney plot for an added oil content of 0%. Shear stress is in Pa-s.

## 4.4 Empirical Model

Since the ratio of water to corn meal was held constant, differences in the viscosity can be attributed to oil content. Yanniotis, Skaltsi, & Karaburnioti (2006) used an Arrhenius type equation to predict the viscosity of honey at different temperatures. A similar approach was taken to predict viscosity from oil content using an empirical model. After both the Bagley and Mooney corrections were performed, apparent viscosity was calculated by Equation 4.1 where $\eta_{app}$ is apparent viscosity in MPa·s, $\dot{\gamma}$ is shear rate in s$^{-1}$, and $\tau$ in shear stress in MPa.

$$\eta_{app} = \frac{\dot{\gamma}}{\tau} \qquad \text{(Equation 4.1)}$$

The apparent viscosities at each oil content were averaged. Data points from failed Mooney corrections were excluded because of the resulting negative viscosities. Potential reasons for failed Mooney corrections were discussed in previous sections.

Figure 4.17 shows a clear relationship between the natural log of apparent viscosity and the inverse of total oil percentage. Total oil percentage was calculated from the sum of the existing fat percentage shown in Table 3.1 and the added oil percentage of either 0%, 2.5%, or 5%. Equation 4.2 shows the empirical model where $\eta_{app}$ is apparent viscosity in MPa·s and OC is percent oil content. This model should only be used at a moisture content of 35% w.b. as changing moisture content would have an additional impact on apparent viscosity.

$$\eta_{app} = 1.05e^{4.41/OC} \qquad \text{(Equation 4.2)}$$

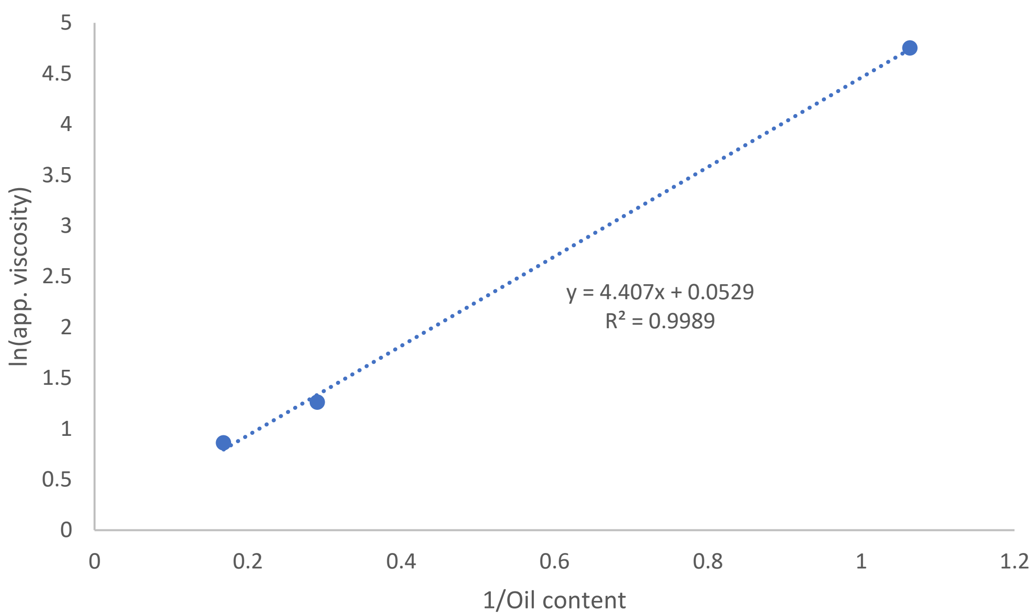


Figure 4.17: Empirical model relating viscosity to oil content.

# 5. CONCLUSION

## 5.1 Conclusion

Overall, the Mooney slip analysis was successful for the sample with 5% oil added. Figure 4.15 provides the shear rate correction for dies with a length to diameter ratio of 4. Slip velocity increased with increasing shear stress. At the lower oil contents of 0% and 2.5% oil added the Mooney slip analysis produced negative shear rates. Obtaining negative shear rates from the Mooney slip correction is a common issue with more complex fluids. These issues stem from the assumptions made over the development of the Mooney method. There is still confidence that the Mooney method is mathematically sound. However, a full understanding of the product being extruded is needed in order to ensure assumptions are met. Small errors in measurements have a large impact on the outcome of the Mooney method and it is therefore necessary to use precision equipment for die measurement. Accurate pressure transducers and flow measurements are also necessary. Despite obtaining negative shear rates for 0% and 2.5% added oil content, an empirical model was developed to predict apparent viscosity of the mixture as a function of total oil content. The developed model should only be used at 35% w.b. moisture because a change in moisture would have an impact on apparent viscosity.

## 5.2 Future Work

- Have the die with a 2mm diameter and 0.25 mm length reworked to prevent sample expansion from causing error in results.
- Determine the time and temperature effect of sample spending a lengthy time in the reservoir during experimentation.
- Determine if lipid-amylose complexes are forming and changing the material the material in the reservoir and during extrusion.

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# 7. APPENDIX A

Data for 0% oil content added

| Run # | Bagley Correction | Speed mm/min | Length mm | Diameter mm | L/R | dP average | Corrected dP |
|---|---|---|---|---|---|---|---|
| 1 | 0.238254 | 5 | 0.25 | 8 | 0.0625 | 0.364114 | 0.12586 |
| 2 | 0.238254 | 5 | 0.25 | 8 | 0.0625 | 0.345957 | 0.107703 |
| 3 | 0.238254 | 5 | 0.25 | 8 | 0.0625 | 0.323102 | 0.084848 |
| 1 | 0.203592 | 10 | 0.25 | 8 | 0.0625 | 0.287873 | 0.084281 |
| 2 | 0.203592 | 10 | 0.25 | 8 | 0.0625 | 0.277152 | 0.07356 |
| 3 | 0.203592 | 10 | 0.25 | 8 | 0.0625 | 0.293747 | 0.090156 |
| 1 | 0.258334 | 20 | 0.25 | 8 | 0.0625 | 0.306948 | 0.048614 |
| 2 | 0.258334 | 20 | 0.25 | 8 | 0.0625 | 0.355696 | 0.097362 |
| 3 | 0.258334 | 20 | 0.25 | 8 | 0.0625 | 0.401485 | 0.143151 |
| 1 | 0.390365 | 50 | 0.25 | 8 | 0.0625 | 0.599689 | 0.209324 |
| 2 | 0.390365 | 50 | 0.25 | 8 | 0.0625 | 0.529316 | 0.138951 |
| 3 | 0.390365 | 50 | 0.25 | 8 | 0.0625 | 0.462087 | 0.071721 |
| 1 | 0.459783 | 75 | 0.25 | 8 | 0.0625 | 0.729263 | 0.26948 |
| 2 | 0.459783 | 75 | 0.25 | 8 | 0.0625 | 0.673875 | 0.214092 |
| 3 | 0.459783 | 75 | 0.25 | 8 | 0.0625 | 0.554601 | 0.094818 |
| 1 | 0.71526 | 100 | 0.25 | 8 | 0.0625 | 0.672961 | -0.0423 |
| 2 | 0.71526 | 100 | 0.25 | 8 | 0.0625 | 0.804231 | 0.08897 |
| 3 | 0.71526 | 100 | 0.25 | 8 | 0.0625 | 0.82305 | 0.10779 |
| 1 | 0.403354 | 5 | 0.25 | 4 | 0.125 | 0.332657 | -0.0707 |
| 2 | 0.403354 | 5 | 0.25 | 4 | 0.125 | 0.424653 | 0.0213 |
| 3 | 0.403354 | 5 | 0.25 | 4 | 0.125 | 0.36466 | -0.03869 |
| 1 | 0.314578 | 10 | 0.25 | 4 | 0.125 | 0.330277 | 0.015699 |
| 2 | 0.314578 | 10 | 0.25 | 4 | 0.125 | 0.344525 | 0.029947 |
| 3 | 0.314578 | 10 | 0.25 | 4 | 0.125 | 0.335766 | 0.021188 |
| 1 | 0.409962 | 20 | 0.25 | 4 | 0.125 | 0.439713 | 0.029751 |
| 2 | 0.409962 | 20 | 0.25 | 4 | 0.125 | 0.456636 | 0.046674 |
| 3 | 0.409962 | 20 | 0.25 | 4 | 0.125 | 0.459218 | 0.049256 |
| 1 | 0.723424 | 50 | 0.25 | 4 | 0.125 | 0.739486 | 0.016061 |
| 2 | 0.723424 | 50 | 0.25 | 4 | 0.125 | 0.869444 | 0.14602 |
| 3 | 0.723424 | 50 | 0.25 | 4 | 0.125 | 0.541056 | -0.18237 |
| 1 | 0.954563 | 75 | 0.25 | 4 | 0.125 | 1.059475 | 0.104912 |
| 2 | 0.954563 | 75 | 0.25 | 4 | 0.125 | 1.028675 | 0.074112 |
| 3 | 0.954563 | 75 | 0.25 | 4 | 0.125 | 0.924675 | -0.02989 |
| 1 | 1.236578 | 100 | 0.25 | 4 | 0.125 | 1.951592 | 0.715013 |
| 2 | 1.236578 | 100 | 0.25 | 4 | 0.125 | 1.00824 | -0.22834 |
| 3 | 1.236578 | 100 | 0.25 | 4 | 0.125 | 1.2818 | 0.045222 |
| 1 | 0.981969 | 5 | 0.25 | 2 | 0.25 | 0.720255 | -0.26171 |
| 2 | 0.981969 | 5 | 0.25 | 2 | 0.25 | 1.171825 | 0.189856 |
| 3 | 0.981969 | 5 | 0.25 | 2 | 0.25 | 1.130486 | 0.148517 |
| 1 | 1.240846 | 10 | 0.25 | 2 | 0.25 | 1.216379 | -0.02447 |
| 2 | 1.240846 | 10 | 0.25 | 2 | 0.25 | 1.215619 | -0.02523 |
| 3 | 1.240846 | 10 | 0.25 | 2 | 0.25 | 1.2559 | 0.015054 |
| 1 | 1.59266 | 20 | 0.25 | 2 | 0.25 | 1.5962 | 0.00354 |
| 2 | 1.59266 | 20 | 0.25 | 2 | 0.25 | 1.832233 | 0.239573 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 3 | 1.59266 | 20 | 0.25 | 2 | 0.25 | 1.411928 | -0.18073 |
| 1 | 2.778175 | 50 | 0.25 | 2 | 0.25 | 3.057649 | 0.279475 |
| 2 | 2.778175 | 50 | 0.25 | 2 | 0.25 | 2.826111 | 0.047936 |
| 3 | 2.778175 | 50 | 0.25 | 2 | 0.25 | 2.841818 | 0.063643 |
| 1 | 3.625396 | 75 | 0.25 | 2 | 0.25 | 3.071075 | -0.55432 |
| 2 | 3.625396 | 75 | 0.25 | 2 | 0.25 | 5.294557 | 1.669161 |
| 3 | 3.625396 | 75 | 0.25 | 2 | 0.25 | 3.448258 | -0.17714 |
| 1 | 4.387154 | 100 | 0.25 | 2 | 0.25 | 4.544925 | 0.157771 |
| 2 | 4.387154 | 100 | 0.25 | 2 | 0.25 | 4.003969 | -0.38318 |
| 3 | 4.387154 | 100 | 0.25 | 2 | 0.25 | 3.942675 | -0.44448 |
| 1 | 0.238254 | 5 | 16 | 8 | 4 | 0.291981 | 0.053727 |
| 2 | 0.238254 | 5 | 16 | 8 | 4 | 0.293533 | 0.055279 |
| 3 | 0.238254 | 5 | 16 | 8 | 4 | 0.298675 | 0.060421 |
| 1 | 0.203592 | 10 | 16 | 8 | 4 | 0.282891 | 0.079299 |
| 2 | 0.203592 | 10 | 16 | 8 | 4 | 0.349613 | 0.146021 |
| 3 | 0.203592 | 10 | 16 | 8 | 4 | 0.306199 | 0.102607 |
| 1 | 0.258334 | 20 | 16 | 8 | 4 | 0.31163 | 0.053296 |
| 2 | 0.258334 | 20 | 16 | 8 | 4 | 0.360693 | 0.102359 |
| 3 | 0.258334 | 20 | 16 | 8 | 4 | 0.370916 | 0.112583 |
| 1 | 0.403354 | 5 | 8 | 4 | 4 | 1.047162 | 0.643808 |
| 2 | 0.403354 | 5 | 8 | 4 | 4 | 0.610618 | 0.207265 |
| 3 | 0.403354 | 5 | 8 | 4 | 4 | 0.632309 | 0.228955 |
| 1 | 0.390365 | 50 | 16 | 8 | 4 | 0.579263 | 0.188898 |
| 2 | 0.390365 | 50 | 16 | 8 | 4 | 0.503734 | 0.113368 |
| 3 | 0.390365 | 50 | 16 | 8 | 4 | 0.54459 | 0.154225 |
| 4 | 0.390365 | 50 | 16 | 8 | 4 | 0.483561 | 0.093196 |
| 1 | 0.459783 | 75 | 16 | 8 | 4 | 0.701875 | 0.242092 |
| 2 | 0.459783 | 75 | 16 | 8 | 4 | 0.532468 | 0.072685 |
| 3 | 0.459783 | 75 | 16 | 8 | 4 | 0.484444 | 0.024661 |
| 1 | 0.314578 | 10 | 8 | 4 | 4 | 0.607274 | 0.292696 |
| 2 | 0.314578 | 10 | 8 | 4 | 4 | 0.57542 | 0.260842 |
| 3 | 0.314578 | 10 | 8 | 4 | 4 | 0.595776 | 0.281199 |
| 1 | 0.71526 | 100 | 16 | 8 | 4 | 0.936294 | 0.221034 |
| 2 | 0.71526 | 100 | 16 | 8 | 4 | 1.494092 | 0.778831 |
| 3 | 0.71526 | 100 | 16 | 8 | 4 | 1.317475 | 0.602215 |
| 1 | 0.409962 | 20 | 8 | 4 | 4 | 0.638369 | 0.228407 |
| 2 | 0.409962 | 20 | 8 | 4 | 4 | 0.798001 | 0.388039 |
| 3 | 0.409962 | 20 | 8 | 4 | 4 | 0.610844 | 0.200882 |
| 1 | 0.981969 | 5 | 4 | 2 | 4 | 1.685535 | 0.703565 |
| 2 | 0.981969 | 5 | 4 | 2 | 4 | 1.71134 | 0.72937 |
| 3 | 0.981969 | 5 | 4 | 2 | 4 | 1.779285 | 0.797315 |
| 1 | 0.723424 | 50 | 8 | 4 | 4 | 1.331119 | 0.607695 |
| 2 | 0.723424 | 50 | 8 | 4 | 4 | 1.12408 | 0.400656 |
| 3 | 0.723424 | 50 | 8 | 4 | 4 | 1.238675 | 0.515251 |
| 1 | 0.954563 | 75 | 8 | 4 | 4 | 1.322521 | 0.367959 |
| 2 | 0.954563 | 75 | 8 | 4 | 4 | 1.630758 | 0.676196 |
| 3 | 0.954563 | 75 | 8 | 4 | 4 | 1.244327 | 0.289765 |
| 1 | 1.240846 | 10 | 4 | 2 | 4 | 1.97159 | 0.730744 |
| 2 | 1.240846 | 10 | 4 | 2 | 4 | 2.146518 | 0.905672 |
| 3 | 1.240846 | 10 | 4 | 2 | 4 | 1.988432 | 0.747586 |
| 1 | 1.236578 | 100 | 8 | 4 | 4 | 1.910342 | 0.673763 |
| 2 | 1.236578 | 100 | 8 | 4 | 4 | 1.470342 | 0.233763 |

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|---|---|---|---|---|---|---|---|
| 3 | 1.236578 | 100 | 8 | 4 | 4 | 1.711028 | 0.47445 |
| 1 | 1.59266 | 20 | 4 | 2 | 4 | 2.224107 | 0.631447 |
| 2 | 1.59266 | 20 | 4 | 2 | 4 | 2.448485 | 0.855824 |
| 3 | 1.59266 | 20 | 4 | 2 | 4 | 2.53575 | 0.94309 |
| 1 | 2.778175 | 50 | 4 | 2 | 4 | 3.575342 | 0.797167 |
| 2 | 2.778175 | 50 | 4 | 2 | 4 | 3.334848 | 0.556673 |
| 3 | 2.778175 | 50 | 4 | 2 | 4 | 3.914704 | 1.13653 |
| 4 | 2.778175 | 50 | 4 | 2 | 4 | 3.385104 | 0.606929 |
| 1 | 3.625396 | 75 | 4 | 2 | 4 | 5.118675 | 1.493279 |
| 2 | 3.625396 | 75 | 4 | 2 | 4 | 3.50044 | -0.12496 |
| 3 | 3.625396 | 75 | 4 | 2 | 4 | 3.219075 | -0.12496 |
| 1 | 4.387154 | 100 | 4 | 2 | 4 | 4.817675 | 0.430521 |
| 2 | 4.387154 | 100 | 4 | 2 | 4 | 7.813119 | 3.425966 |
| 3 | 4.387154 | 100 | 4 | 2 | 4 | 6.958675 | 2.571521 |
| 1 | 0.238254 | 5 | 32 | 8 | 8 | 0.817759 | 0.579505 |
| 2 | 0.238254 | 5 | 32 | 8 | 8 | 0.925183 | 0.686929 |
| 3 | 0.238254 | 5 | 32 | 8 | 8 | 0.827652 | 0.589398 |
| 1 | 0.203592 | 10 | 32 | 8 | 8 | 0.794249 | 0.590657 |
| 2 | 0.203592 | 10 | 32 | 8 | 8 | 0.817008 | 0.613416 |
| 3 | 0.203592 | 10 | 32 | 8 | 8 | 0.822177 | 0.618585 |
| 1 | 0.258334 | 20 | 32 | 8 | 8 | 0.830926 | 0.572592 |
| 2 | 0.258334 | 20 | 32 | 8 | 8 | 0.935756 | 0.677422 |
| 3 | 0.258334 | 20 | 32 | 8 | 8 | 0.913675 | 0.655341 |
| 1 | 0.403354 | 5 | 16 | 4 | 8 | 0.931649 | 0.528295 |
| 2 | 0.403354 | 5 | 16 | 4 | 8 | 0.92359 | 0.520236 |
| 3 | 0.403354 | 5 | 16 | 4 | 8 | 0.95174 | 0.548386 |
| 1 | 0.390365 | 50 | 32 | 8 | 8 | 1.259389 | 0.869024 |
| 2 | 0.390365 | 50 | 32 | 8 | 8 | 1.340726 | 0.950361 |
| 3 | 0.390365 | 50 | 32 | 8 | 8 | 0.996349 | 0.605984 |
| 1 | 0.459783 | 75 | 32 | 8 | 8 | 1.728305 | 1.268521 |
| 2 | 0.459783 | 75 | 32 | 8 | 8 | 1.602961 | 1.143177 |
| 3 | 0.459783 | 75 | 32 | 8 | 8 | 1.480156 | 1.020373 |
| 1 | 0.314578 | 10 | 16 | 4 | 8 | 0.899907 | 0.585329 |
| 2 | 0.314578 | 10 | 16 | 4 | 8 | 0.945564 | 0.630986 |
| 3 | 0.314578 | 10 | 16 | 4 | 8 | 0.956075 | 0.641497 |
| 1 | 0.71526 | 100 | 32 | 8 | 8 | 2.46677 | 1.75151 |
| 2 | 0.71526 | 100 | 32 | 8 | 8 | 1.375175 | 0.659915 |
| 3 | 0.71526 | 100 | 32 | 8 | 8 | 2.129786 | 1.414526 |
| 1 | 0.409962 | 20 | 16 | 4 | 8 | 1.07984 | 0.669878 |
| 2 | 0.409962 | 20 | 16 | 4 | 8 | 1.188953 | 0.778991 |
| 3 | 0.409962 | 20 | 16 | 4 | 8 | 1.058585 | 0.648623 |
| 1 | 0.981969 | 5 | 8 | 2 | 8 | 2.324547 | 1.342578 |
| 2 | 0.981969 | 5 | 8 | 2 | 8 | 2.470263 | 1.488294 |
| 3 | 0.981969 | 5 | 8 | 2 | 8 | 2.342309 | 1.36034 |
| 1 | 0.723424 | 50 | 16 | 4 | 8 | 1.592961 | 0.869536 |
| 2 | 0.723424 | 50 | 16 | 4 | 8 | 1.622265 | 0.89884 |
| 3 | 0.723424 | 50 | 16 | 4 | 8 | 1.688425 | 0.965001 |
| 1 | 0.954563 | 75 | 16 | 4 | 8 | 2.039475 | 1.084912 |
| 2 | 0.954563 | 75 | 16 | 4 | 8 | 2.036675 | 1.082112 |
| 3 | 0.954563 | 75 | 16 | 4 | 8 | 1.952008 | 0.997446 |
| 1 | 1.240846 | 10 | 8 | 2 | 8 | 2.461846 | 1.221 |
| 2 | 1.240846 | 10 | 8 | 2 | 8 | 2.553119 | 1.312273 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 3 | 1.240846 | 10 | 8 | 2 | 8 | 2.687269 | 1.446423 |
| 1 | 1.236578 | 100 | 16 | 4 | 8 | 2.904444 | 1.667866 |
| 2 | 1.236578 | 100 | 16 | 4 | 8 | 2.756936 | 1.520358 |
| 3 | 1.236578 | 100 | 16 | 4 | 8 | 3.071075 | 1.834497 |
| 1 | 1.59266 | 20 | 8 | 2 | 8 | 3.080967 | 1.488307 |
| 2 | 1.59266 | 20 | 8 | 2 | 8 | 3.168378 | 1.575718 |
| 3 | 1.59266 | 20 | 8 | 2 | 8 | 3.00509 | 1.41243 |
| 1 | 2.778175 | 50 | 8 | 2 | 8 | 4.537766 | 1.759591 |
| 2 | 2.778175 | 50 | 8 | 2 | 8 | 4.505992 | 1.727817 |
| 3 | 2.778175 | 50 | 8 | 2 | 8 | 4.803087 | 2.024912 |
| 1 | 3.625396 | 75 | 8 | 2 | 8 | 5.969475 | 2.344079 |
| 2 | 3.625396 | 75 | 8 | 2 | 8 | 5.136402 | 1.511006 |
| 3 | 3.625396 | 75 | 8 | 2 | 8 | 5.461475 | 1.836079 |
| 1 | 4.387154 | 100 | 8 | 2 | 8 | 7.082961 | 2.695807 |
| 2 | 4.387154 | 100 | 8 | 2 | 8 | 6.583119 | 2.195966 |
| 3 | 4.387154 | 100 | 8 | 2 | 8 | 7.751175 | 3.364021 |

# 8. APPENDIX B

Data for 2.5% oil content added

| Run # | Bagley Correction | Speed mm/min | Length mm | Diameter mm | L/R | dP average | Corrected dP |
|---|---|---|---|---|---|---|---|
| 1 | 0.050386 | 5 | 0.25 | 8 | 0.0625 | 0.105373 | 0.054987 |
| 1 | -0.01781 | 10 | 0.25 | 8 | 0.0625 | 0.026441 | 0.044249 |
| 1 | 0.005701 | 20 | 0.25 | 8 | 0.0625 | 0.062611 | 0.05691 |
| 1 | #NUM! | 50 | 0.25 | 8 | 0.0625 | 0.237246 | #NUM! |
| 1 | 0.169523 | 75 | 0.25 | 8 | 0.0625 | 0.266094 | 0.096571 |
| 1 | 0.236453 | 100 | 0.25 | 8 | 0.0625 | 0.288175 | 0.051722 |
| 1 | 0.163054 | 5 | 0.25 | 4 | 0.125 | 0.246989 | 0.083935 |
| 1 | 0.10446 | 10 | 0.25 | 4 | 0.125 | 0.221428 | 0.116968 |
| 1 | 0.25012 | 20 | 0.25 | 4 | 0.125 | 0.372983 | 0.122863 |
| 1 | 0.575242 | 50 | 0.25 | 4 | 0.125 | 0.625614 | 0.050372 |
| 1 | 0.56815 | 75 | 0.25 | 4 | 0.125 | 0.727342 | 0.159192 |
| 1 | 0.842647 | 100 | 0.25 | 4 | 0.125 | 0.886402 | 0.043755 |
| 1 | 0.85514 | 5 | 0.25 | 2 | 0.25 | 0.874557 | 0.019418 |
| 1 | 1.682673 | 10 | 0.25 | 2 | 0.25 | 1.872258 | 0.189584 |
| 1 | 1.426056 | 20 | 0.25 | 2 | 0.25 | 1.426598 | 0.000542 |
| 1 | 4.003959 | 50 | 0.25 | 2 | 0.25 | 4.088448 | 0.084488 |
| 1 | 3.865801 | 75 | 0.25 | 2 | 0.25 | 4.107296 | 0.241494 |
| 1 | 5.64348 | 100 | 0.25 | 2 | 0.25 | 5.417499 | -0.22598 |
| 2 | 5.64348 | 100 | 0.25 | 2 | 0.25 | 6.000414 | 0.356934 |
| 1 | 0.050386 | 5 | 16 | 8 | 4 | 0.242623 | 0.192237 |
| 1 | -0.01781 | 10 | 16 | 8 | 4 | 0.204112 | 0.221921 |
| 1 | 0.005701 | 20 | 16 | 8 | 4 | 0.288132 | 0.282431 |
| 1 | 0.163054 | 5 | 8 | 4 | 4 | 0.482589 | 0.319535 |
| 1 | #NUM! | 50 | 16 | 8 | 4 | 0.447611 | #NUM! |
| 1 | 0.169523 | 75 | 16 | 8 | 4 | 0.44805 | 0.278527 |
| 1 | 0.10446 | 10 | 8 | 4 | 4 | 0.415456 | 0.310996 |
| 1 | 0.236453 | 100 | 16 | 8 | 4 | 0.685598 | 0.449145 |
| 1 | 0.25012 | 20 | 8 | 4 | 4 | 0.675264 | 0.425145 |
| 1 | 0.85514 | 5 | 4 | 2 | 4 | 1.383215 | 0.528075 |
| 1 | 0.575242 | 50 | 8 | 4 | 4 | 1.152008 | 0.576766 |
| 1 | 0.56815 | 75 | 8 | 4 | 4 | 1.092961 | 0.524811 |
| 1 | 1.682673 | 10 | 4 | 2 | 4 | 1.299725 | -0.38295 |
| 1 | 0.842647 | 100 | 8 | 4 | 4 | 1.590342 | 0.747694 |
| 1 | 1.426056 | 20 | 4 | 2 | 4 | 2.0618 | 0.635744 |
| 1 | 4.003959 | 50 | 4 | 2 | 4 | 3.633065 | -0.37089 |
| 1 | 3.865801 | 75 | 4 | 2 | 4 | 3.333503 | -0.5323 |
| 1 | 5.64348 | 100 | 4 | 2 | 4 | 4.921616 | -0.72186 |
| 1 | 0.050386 | 5 | 32 | 8 | 8 | 0.684598 | 0.634212 |
| 1 | -0.01781 | 10 | 32 | 8 | 8 | 0.621954 | 0.639762 |
| 1 | 0.005701 | 20 | 32 | 8 | 8 | 0.822769 | 0.817069 |
| 1 | 0.163054 | 5 | 16 | 4 | 8 | 1.143911 | 0.980857 |
| 1 | #NUM! | 50 | 32 | 8 | 8 | 1.214327 | #NUM! |
| 1 | 0.169523 | 75 | 32 | 8 | 8 | 1.169613 | 1.000089 |
| 1 | 0.10446 | 10 | 16 | 4 | 8 | 1.222145 | 1.117685 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1 | 0.236453 | 100 | 32 | 8 | 8 | 1.349508 | 1.113055 |
| 1 | 0.25012 | 20 | 16 | 4 | 8 | 1.606864 | 1.356744 |
| 1 | 0.85514 | 5 | 8 | 2 | 8 | 1.852966 | 0.997827 |
| 1 | 0.575242 | 50 | 16 | 4 | 8 | 1.878283 | 1.303041 |
| 1 | 0.56815 | 75 | 16 | 4 | 8 | 2.277738 | 1.709587 |
| 1 | 1.682673 | 10 | 8 | 2 | 8 | 1.833344 | 0.150671 |
| 1 | 0.842647 | 100 | 16 | 4 | 8 | 2.432275 | 1.589628 |
| 1 | 1.426056 | 20 | 8 | 2 | 8 | 2.529305 | 1.103249 |
| 1 | 4.003959 | 50 | 8 | 2 | 8 | 3.724361 | -0.2796 |
| 1 | 3.865801 | 75 | 8 | 2 | 8 | 3.980675 | 0.114874 |
| 1 | 5.64348 | 100 | 8 | 2 | 8 | 5.056675 | -0.5868 |

# 9. APPENDIX C

Data for 5% oil content added

| Run # | Bagley Correction | Speed mm/min | Length mm | Diameter mm | L/R | dP average |
|---|---|---|---|---|---|---|
| 1 | 0.397802 | 75 | 0.25 | 8 | 0.0625 | 0.314845 |
| 3 | 0.081153 | 10 | 0.25 | 8 | 0.0625 | 0.069842 |
| 1 | 0.081153 | 10 | 0.25 | 8 | 0.0625 | 0.072113 |
| 2 | 0.081153 | 10 | 0.25 | 8 | 0.0625 | 0.082503 |
| 3 | 0.163192 | 5 | 0.25 | 8 | 0.0625 | 0.181254 |
| 2 | 0.163192 | 5 | 0.25 | 8 | 0.0625 | 0.191349 |
| 3 | 0.1479 | 100 | 0.25 | 8 | 0.0625 | 0.178104 |
| 1 | 0.145562 | 50 | 0.25 | 8 | 0.0625 | 0.185093 |
| 3 | 0.397802 | 75 | 0.25 | 8 | 0.0625 | 0.444845 |
| 2 | 0.1479 | 100 | 0.25 | 8 | 0.0625 | 0.201496 |
| 2 | 0.397802 | 75 | 0.25 | 8 | 0.0625 | 0.451694 |
| 3 | 0.145562 | 50 | 0.25 | 8 | 0.0625 | 0.217649 |
| 2 | 0.145562 | 50 | 0.25 | 8 | 0.0625 | 0.220081 |
| 1 | 0.1479 | 100 | 0.25 | 8 | 0.0625 | 0.230342 |
| 1 | 0.163192 | 5 | 0.25 | 8 | 0.0625 | 0.334257 |
| 2 | #NUM! | 20 | 0.25 | 8 | 0.0625 | 0.18942 |
| 1 | #NUM! | 20 | 0.25 | 8 | 0.0625 | 0.259088 |
| 3 | #NUM! | 20 | 0.25 | 8 | 0.0625 | #NUM! |
| 3 | 0.866576 | 100 | 0.25 | 4 | 0.125 | 0.847053 |
| 3 | 0.803141 | 10 | 0.25 | 4 | 0.125 | 0.807938 |
| 3 | 0.387259 | 50 | 0.25 | 4 | 0.125 | 0.408137 |
| 2 | 0.866576 | 100 | 0.25 | 4 | 0.125 | 0.937906 |
| 1 | 0.866576 | 100 | 0.25 | 4 | 0.125 | 0.959508 |
| 2 | 0.803141 | 10 | 0.25 | 4 | 0.125 | 0.905779 |
| 1 | 0.29814 | 75 | 0.25 | 4 | 0.125 | 0.461554 |
| 3 | 0.29814 | 75 | 0.25 | 4 | 0.125 | 0.484185 |
| 1 | 0.079938 | 5 | 0.25 | 4 | 0.125 | 0.271169 |
| 1 | 0.803141 | 10 | 0.25 | 4 | 0.125 | 1.016464 |
| 2 | 0.079938 | 5 | 0.25 | 4 | 0.125 | 0.298825 |
| 3 | 0.079938 | 5 | 0.25 | 4 | 0.125 | 0.31984 |
| 2 | 0.29814 | 75 | 0.25 | 4 | 0.125 | 0.579613 |
| 3 | -0.07166 | 20 | 0.25 | 4 | 0.125 | 0.249369 |
| 1 | 0.387259 | 50 | 0.25 | 4 | 0.125 | 0.721687 |
| 2 | 0.387259 | 50 | 0.25 | 4 | 0.125 | 0.723149 |
| 1 | -0.07166 | 20 | 0.25 | 4 | 0.125 | 0.277721 |
| 2 | -0.07166 | 20 | 0.25 | 4 | 0.125 | 0.322922 |
| 3 | 2.326032 | 75 | 0.25 | 2 | 0.25 | 1.018675 |
| 1 | 3.6848 | 100 | 0.25 | 2 | 0.25 | 3.696066 |
| 3 | 2.817673 | 50 | 0.25 | 2 | 0.25 | 2.923564 |
| 1 | 2.817673 | 50 | 0.25 | 2 | 0.25 | 2.940133 |
| 2 | 0.557885 | 5 | 0.25 | 2 | 0.25 | 0.779432 |
| 2 | 2.817673 | 50 | 0.25 | 2 | 0.25 | 3.10677 |
| 2 | 3.6848 | 100 | 0.25 | 2 | 0.25 | 4.050342 |
| 3 | 3.6848 | 100 | 0.25 | 2 | 0.25 | 4.081342 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 1.214544 | 20 | 0.25 | 2 | 0.25 | 1.710835 |
| 3 | 1.214544 | 20 | 0.25 | 2 | 0.25 | 1.759856 |
| 2 | 1.214544 | 20 | 0.25 | 2 | 0.25 | 1.839078 |
| 3 | 0.557885 | 5 | 0.25 | 2 | 0.25 | 1.251085 |
| 1 | 0.834283 | 10 | 0.25 | 2 | 0.25 | 1.589227 |
| 3 | 0.834283 | 10 | 0.25 | 2 | 0.25 | 1.607584 |
| 2 | 0.834283 | 10 | 0.25 | 2 | 0.25 | 1.61699 |
| 1 | 0.557885 | 5 | 0.25 | 2 | 0.25 | 1.431906 |
| 1 | 2.326032 | 75 | 0.25 | 2 | 0.25 | 3.286367 |
| 2 | 2.326032 | 75 | 0.25 | 2 | 0.25 | 3.636417 |
| 3 | 3.6848 | 100 | 4 | 2 | 4 | 2.895624 |
| 3 | 2.817673 | 50 | 4 | 2 | 4 | 2.265557 |
| 1 | 3.6848 | 100 | 4 | 2 | 4 | 3.243906 |
| 3 | 0.803141 | 10 | 8 | 4 | 4 | 0.427039 |
| 2 | 2.817673 | 50 | 4 | 2 | 4 | 2.454369 |
| 2 | 0.803141 | 10 | 8 | 4 | 4 | 0.452213 |
| 1 | 0.803141 | 10 | 8 | 4 | 4 | 0.461303 |
| 1 | 2.817673 | 50 | 4 | 2 | 4 | 2.633976 |
| 2 | 3.6848 | 100 | 4 | 2 | 4 | 3.501104 |
| 2 | 0.163192 | 5 | 16 | 8 | 4 | 0.243226 |
| 1 | 0.163192 | 5 | 16 | 8 | 4 | 0.246941 |
| 3 | 0.163192 | 5 | 16 | 8 | 4 | 0.247911 |
| 2 | 0.397802 | 75 | 16 | 8 | 4 | 0.497008 |
| 3 | 0.397802 | 75 | 16 | 8 | 4 | 0.550575 |
| 1 | 0.866576 | 100 | 8 | 4 | 4 | 1.034762 |
| 3 | 0.29814 | 75 | 8 | 4 | 4 | 0.507961 |
| 1 | 0.145562 | 50 | 16 | 8 | 4 | 0.357917 |
| 1 | 0.397802 | 75 | 16 | 8 | 4 | 0.618831 |
| 1 | 2.326032 | 75 | 4 | 2 | 4 | 2.55044 |
| 1 | 0.081153 | 10 | 16 | 8 | 4 | 0.330648 |
| 2 | 0.145562 | 50 | 16 | 8 | 4 | 0.404182 |
| 3 | 0.866576 | 100 | 8 | 4 | 4 | 1.125197 |
| 3 | 0.081153 | 10 | 16 | 8 | 4 | 0.350558 |
| 3 | 0.145562 | 50 | 16 | 8 | 4 | 0.423675 |
| 2 | 0.866576 | 100 | 8 | 4 | 4 | 1.152906 |
| 3 | 0.079938 | 5 | 8 | 4 | 4 | 0.37152 |
| 3 | 2.326032 | 75 | 4 | 2 | 4 | 2.635606 |
| 3 | 1.214544 | 20 | 4 | 2 | 4 | 1.533736 |
| 2 | 0.081153 | 10 | 16 | 8 | 4 | 0.403692 |
| 1 | 0.079938 | 5 | 8 | 4 | 4 | 0.416203 |
| 2 | 0.079938 | 5 | 8 | 4 | 4 | 0.435245 |
| 3 | 0.387259 | 50 | 8 | 4 | 4 | 0.771501 |
| 2 | 0.387259 | 50 | 8 | 4 | 4 | 0.79821 |
| 1 | 0.387259 | 50 | 8 | 4 | 4 | 0.854092 |
| 1 | 1.214544 | 20 | 4 | 2 | 4 | 1.691175 |
| 2 | 2.326032 | 75 | 4 | 2 | 4 | 2.858675 |
| 1 | -0.07166 | 20 | 8 | 4 | 4 | 0.461578 |
| 3 | 0.1479 | 100 | 16 | 8 | 4 | 0.685288 |
| 3 | -0.07166 | 20 | 8 | 4 | 4 | 0.486077 |
| 2 | -0.07166 | 20 | 8 | 4 | 4 | 0.527842 |
| 1 | 0.29814 | 75 | 8 | 4 | 4 | 0.899008 |
| 2 | 1.214544 | 20 | 4 | 2 | 4 | 1.824966 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 2 | 0.834283 | 10 | 4 | 2 | 4 | 1.444939 |
| 3 | 0.834283 | 10 | 4 | 2 | 4 | 1.447332 |
| 1 | 0.1479 | 100 | 16 | 8 | 4 | 0.769065 |
| 2 | 0.1479 | 100 | 16 | 8 | 4 | 0.784572 |
| 2 | 0.29814 | 75 | 8 | 4 | 4 | 1.01803 |
| 1 | 0.834283 | 10 | 4 | 2 | 4 | 1.598494 |
| 1 | 0.557885 | 5 | 4 | 2 | 4 | 1.377308 |
| 3 | 0.557885 | 5 | 4 | 2 | 4 | 1.493962 |
| 2 | 0.557885 | 5 | 4 | 2 | 4 | 1.578725 |
| 3 | #NUM! | 20 | 16 | 8 | 4 | 0.249385 |
| 2 | #NUM! | 20 | 16 | 8 | 4 | 0.258841 |
| 1 | #NUM! | 20 | 16 | 8 | 4 | 0.297223 |
| 3 | 0.803141 | 10 | 16 | 4 | 8 | 0.598187 |
| 2 | 0.803141 | 10 | 16 | 4 | 8 | 0.634974 |
| 1 | 0.803141 | 10 | 16 | 4 | 8 | 0.675422 |
| 1 | 2.817673 | 50 | 8 | 2 | 8 | 2.825988 |
| 3 | 2.817673 | 50 | 8 | 2 | 8 | 2.843827 |
| 2 | 3.6848 | 100 | 8 | 2 | 8 | 3.771212 |
| 3 | 0.397802 | 75 | 32 | 8 | 8 | 0.650313 |
| 2 | 2.817673 | 50 | 8 | 2 | 8 | 3.101099 |
| 2 | 0.397802 | 75 | 32 | 8 | 8 | 0.740758 |
| 3 | 3.6848 | 100 | 8 | 2 | 8 | 4.050359 |
| 1 | 0.397802 | 75 | 32 | 8 | 8 | 0.799212 |
| 1 | 3.6848 | 100 | 8 | 2 | 8 | 4.104347 |
| 1 | 0.163192 | 5 | 32 | 8 | 8 | 0.591549 |
| 2 | 0.081153 | 10 | 32 | 8 | 8 | 0.562771 |
| 3 | 0.081153 | 10 | 32 | 8 | 8 | 0.576521 |
| 2 | 0.163192 | 5 | 32 | 8 | 8 | 0.708471 |
| 3 | 0.163192 | 5 | 32 | 8 | 8 | 0.711772 |
| 1 | 0.081153 | 10 | 32 | 8 | 8 | 0.632592 |
| 2 | 0.866576 | 100 | 16 | 4 | 8 | 1.431351 |
| 1 | 0.866576 | 100 | 16 | 4 | 8 | 1.535052 |
| 3 | 0.866576 | 100 | 16 | 4 | 8 | 1.625562 |
| 1 | 0.145562 | 50 | 32 | 8 | 8 | 0.913372 |
| 3 | 0.145562 | 50 | 32 | 8 | 8 | 0.91882 |
| 2 | 0.145562 | 50 | 32 | 8 | 8 | 0.940675 |
| 4 | -0.07166 | 20 | 16 | 4 | 8 | 0.767948 |
| 1 | 0.1479 | 100 | 32 | 8 | 8 | 1.436256 |
| 3 | 0.1479 | 100 | 32 | 8 | 8 | 1.624218 |
| 2 | 0.1479 | 100 | 32 | 8 | 8 | 1.637564 |
| 2 | 0.079938 | 5 | 16 | 4 | 8 | 1.575707 |
| 3 | 0.079938 | 5 | 16 | 4 | 8 | 1.73244 |
| 2 | 0.387259 | 50 | 16 | 4 | 8 | 2.046322 |
| 2 | 2.326032 | 75 | 8 | 2 | 8 | 3.999925 |
| 3 | 0.387259 | 50 | 16 | 4 | 8 | 2.064045 |
| 1 | 0.079938 | 5 | 16 | 4 | 8 | 1.760298 |
| 3 | 0.29814 | 75 | 16 | 4 | 8 | 2.055675 |
| 2 | 0.29814 | 75 | 16 | 4 | 8 | 2.188455 |
| 3 | 2.326032 | 75 | 8 | 2 | 8 | 4.354733 |
| 3 | 1.214544 | 20 | 8 | 2 | 8 | 3.260794 |
| 1 | 0.29814 | 75 | 16 | 4 | 8 | 2.406483 |
| 1 | 0.387259 | 50 | 16 | 4 | 8 | 2.587824 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 1 | 2.326032 | 75 | 8 | 2 | 8 | 4.605933 |
| 3 | -0.07166 | 20 | 16 | 4 | 8 | 2.426414 |
| 2 | 1.214544 | 20 | 8 | 2 | 8 | 3.762061 |
| 31 | -0.07166 | 20 | 16 | 4 | 8 | 2.485293 |
| 1 | 1.214544 | 20 | 8 | 2 | 8 | 4.061388 |
| 1 | -0.07166 | 20 | 16 | 4 | 8 | 3.292579 |
| 3 | 0.557885 | 5 | 8 | 2 | 8 | 4.109753 |
| 2 | -0.07166 | 20 | 16 | 4 | 8 | 3.485521 |
| 2 | 0.557885 | 5 | 8 | 2 | 8 | 4.380672 |
| 3 | 0.834283 | 10 | 8 | 2 | 8 | 4.859156 |
| 2 | 0.834283 | 10 | 8 | 2 | 8 | 5.332109 |
| 1 | 0.834283 | 10 | 8 | 2 | 8 | 5.674253 |
| 31 | 1.214544 | 20 | 8 | 2 | 8 | 6.117204 |
| 1 | 0.557885 | 5 | 8 | 2 | 8 | 5.669651 |
| 4 | #NUM! | 20 | 32 | 8 | 8 | 0.65022 |
| 2 | #NUM! | 20 | 32 | 8 | 8 | 0.74657 |
| 3 | #NUM! | 20 | 32 | 8 | 8 | 0.755066 |
| 1 | #NUM! | 20 | 32 | 8 | 8 | 0.884187 |