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Measurement of Rheology of Distiller's Grain Slurries Using a Helical Impeller Viscometer

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Abstract

Current research is focused on developing a process to convert the cellulose and hemicellulose in distiller's grains into fermentable sugars, increasing both ethanol yield and the amount of protein in the remaining solid product. The rheologic properties of distiller's grain slurries were determined for concentrations of 21, 23, and 25%. Distiller's grain slurries are non-Newtonian, heterogeneous fluids subject to particle settling. Traditional methods of viscosity measurement, such as cone-and-plate and concentric cylinder viscometers, are not adequate for these fluids. A helical impeller viscometer was employed to measure impeller torque over a range of rotational speeds. Newtonian and non-Newtonian calibration fluids were utilized to obtain constants that relate shear stresses and shear rates to the experimental data. The Newtonian impeller constant, *c*, was 151; the non-Newtonian shear rate constant, *k*, was 10.30. Regression analysis of experimental data was utilized for comparison to power law, Herschel-Bulkley, and Casson viscosity models with regression coefficients exceeding 0.99 in all cases.

Index Entries: Distiller's grain slurries; rheologic properties; wet grains; calibration fluids; helical impeller.

Introduction

During corn dry mill ethanol manufacturing, the most common method for ethanol production in North America, the primary byproduct is dried distiller's grain (DDG). As production increases to meet demand, the supply of DDG will significantly increase. Thus, ethanol producers need to modify their processes for the sake of profitability. Technological

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advances for alcohol producers will incorporate the improvement of ethanol yield and the creation of new products from DDG. Current research is focused on developing a process that converts the cellulose and hemicellulose in wet grains and fiber residues into fermentable sugars, increasing both the ethanol yield and the protein percentage in DDG.

To develop an optimum process, the rheologic properties of wet grains must be known. The rheologic properties of wet grain can be utilized to predict performance and behavior during the process, making it possible to design the process for optimal sugar conversion. Viscosity data will aid in the assessment of processability of wet grains when designing pumping and piping systems for the proposed process.

Wet grains are suspensions containing macroparticles. Because of this property, wet grains exhibit complex rheologic properties and are typically characterized as non-Newtonian fluids. Because of the complex relationship between shear rate and shear stress of wet grains, characterization of rheologic properties is more difficult than for simple Newtonian fluids. Conventional viscosity measurement devices, such as concentric cylinder, cone-and-plate, and pipeline viscometers, are prone to particle settling, slip velocity near the wall, and phase separation when measuring the rheology of suspensions. Therefore, an alternative method for measuring rheologic behavior must be used.

The impeller method is a technique commonly used to determine rheologic properties of fluids subject to particle settling. The impeller method utilizes a viscometer along with Newtonian and non-Newtonian calibration fluids to obtain constants that relate shear stresses and shear rates to experimentally measured values of torque and rotational speed. Newtonian calibration fluids are used to determine the impeller constant, c, and non-Newtonian calibration fluids are used to calculate the shear rate constant, k. These constants are then used to aid in the determination of rheologic properties of a selected non-Newtonian fluid, such as wet grains.

The aim of the present study was to determine rheologic characteristics of wet grain slurries using a helical impeller viscometer. The collected data were analyzed to develop a model describing the rheologic behavior of wet grain slurries.

Materials and Methods

Equipment

Two Brookfield viscometers were used to collect the data necessary for rheologic property studies of wet grains: a Brookfield RVDV III viscometer with a cone-and-plate spindle and a Brookfield HBDV III viscometer with a double helical ribbon impeller attachment. The Brookfield RVDV III had a full-scale torque of 7187 dyn-cm, and the HBDV III had a full-scale torque of 57,496 dyn-cm. Each viscometer had a maximum rotational speed of 250 rpm. Both viscometers had accuracy limits of ± 5% full-scale torque.

Howeever, inaccuracies were apparent for non-Newtonian calibration fluids and distiller's grain slurries up to ± 10% of the full-scale torque. Therefore, only measurements above 10% and below 90% torque were utilized for guar gum solutions and distiller's grain slurries. The experiments were conducted at a constant temperature of 25 ± 0.1°C.

The helical impeller was fashioned from nylon using selective laser sintering technology. The impeller had a diameter of 0.04 m and a pitch of 0.02 m. The impeller featured two helices, an ascending outer flight and a descending inner flight. The length of the impeller was 0.055 m, with an off-bottom clearance of 0.025 m.

The vessels used for Newtonian and non-Newtonian calibration were 1000-mL glass beakers. Because of particle settling, the vessel size utilized for wet grain slurries was reduced to 200 mL. Reducing the ratio of the helical impeller diameter to the vessel diameter allowed for more accurate rheology measurement of grain slurries. Although the effect of vessel size on helical impeller readings was not investigated in this study, previous researchers have been reported that vessel size has little or no effect *(1)*.

Calibration Fluids and Distiller's Grains

The Newtonian calibration fluids used were silicone oil and glycerol. The non-Newtonian calibration fluids used were guar gum solutions at relatively low concentrations (0.75 and 1.0% by weight) in deionized water. Distiller's grain particles were provided by a commercial source and had an average particle size of 0.361 × 0.565 mm, measured by electron microscopy. The distiller's grains were used to formulate wet grain slurries in solutions of 0.1% (w/v) xanthan gum in deionized water. The xanthan gum was found to have no effect on the viscosity of water and was used to aid in the suspension of grain particles. Wet grain slurries of 21, 23, and 25% (w/v) solids concentration were analyzed.

Impeller Viscometer Technique

Newtonian and non-Newtonian calibration fluids were used to determine the necessary calibration constants for the impeller method. It has been previously determined that the impeller method is only valid for a Reynolds number (Re) <10. Impeller rotational speed and torque data from Newtonian calibration fluids of known viscosity were employed to determine the Newtonian calibration constant, *c*. Cone-and plate-viscometer data from non-Newtonian calibration fluids were used to determine a viscosity vs shear rate relationship. Impeller rotational speed and torque data of the non-Newtonian calibration fluids combined with a determined viscosity vs shear rate correlation were utilized to calculate the shear rate constant, *k*. The impeller method calibration constants allow the calculation of viscosity, shear rate, and shear stress data of non-Newtonian suspensions. Metz et al. *(2)* have thoroughly discussed the equations utilized in the impeller method.

The Newtonian calibration constant is a function of Re and the power number:

$$P_N = \frac{2\pi NM}{\rho N^3 D_i^5} \quad (1)$$

in which Re can be expressed as

$$\mathrm{Re} = \frac{\rho N D_i^2}{\eta_a} \quad (2)$$

Assuming a Newtonian fluid ($\mu = \eta_a$) and combining Eqs. 1 and 2 results in an equation for impeller torque:

$$M = \frac{c}{2\pi} \mu N D_i^3 \quad (3)$$

For a given rotational speed, the measured torque combined with fluid viscosity and impeller diameter can be used to determine the Newtonian calibration constant, *c*.

The viscosity of a non-Newtonian fluid can be calculated using the Newtonian calibration constant, impeller speed and torque, and impeller diameter:

$$\eta_a = \frac{2\pi M}{c N D_i^3} \quad (4)$$

To calculate the shear rate constant, *k*, a relationship must be established between shear rate and viscosity of a non-Newtonian calibration fluid. A cone-and-plate viscometer is used to determine a correlation between shear rate and viscosity that can be fit to a power law model. The power law correlation is then applied to viscosity data calculated from the impeller viscometer and Eq. 4. The shear rate constant can be calculated as follows:

$$k = \frac{\dot{\gamma}_{AV}}{N} \quad (5)$$

Viscosity, shear stress, and shear rate can be calculated for any non-Newtonian suspension using the impeller method calibration constants. Viscosity is determined using Eq. 4. The shear stress can be calculated for any impeller speed and measured torque:

$$\tau = \frac{2\pi MK}{c D_i^3} \quad (6)$$

The shear rate can then be calculated:

$$\tau = \eta_a \dot{\gamma} \quad (7)$$

Rheologic Models

Three empirical models were utilized to fit the rheologic characteristics of the wet grain slurries: power law, Herschel-Bulkley, and Casson. The power law and Casson models are two-parameter models and are ideal for

Table 1
Newtonian Calibration Fluids

Calibration solution	Average viscosity (Pa·s)	Re
Glycerol	0.8465	0.47–7.25
Silicone oil 1 *M*	0.9537	0.42–5.87

Table 2
Average Newtonian Calibration Constants

Calibration solution	c_{avg}	c_{max}	c_{min}	SD
Glycerol	151.96	156.27	148.83	2.34
Silicone oil 1 *M*	150.68	153.39	148.57	1.50
Overall	151.00			

non-Newtonian rheologic modeling. The Herschel-Bulkley model is a three-parameter model that combines the power law with yield stress.

The power law, or Ostwald-de Waele, model is the simplest and most widely used rheologic empiricism. The power law states

$$\tau = K_{pl} |\dot{\gamma}|^{n_{pl}} \tag{8}$$

The Herschel-Bulkley model is shown as

$$\tau = \tau_0 + K_{HB} \dot{\gamma}^{HB} \tag{9}$$

The Casson model incorporates yield stress and is written as

$$\tau^{1/2} = \tau_0^{1/2} + K_c \dot{\gamma}^{1/2} \tag{10}$$

Results

Calibration

Average viscosity and helical impeller data were combined to determine the Newtonian calibration constant, *c*. Table 1 presents the measured average viscosities and R_e ranges for silicone oil and glycerol. Table 2 provides the average Newtonian calibration constants for each calibration fluid, standard deviation (SD) for each data set, and recorded c_{max} and c_{min} values. The overall average Newtonian calibration constant was 151. Figure 1 presents the power law fits for the guar gum solutions. Table 3 summarizes power law parameters calculated from these graphs. The average overall shear rate constant was calculated to be 10.3; and Table 4 summarizes shear rate constants.

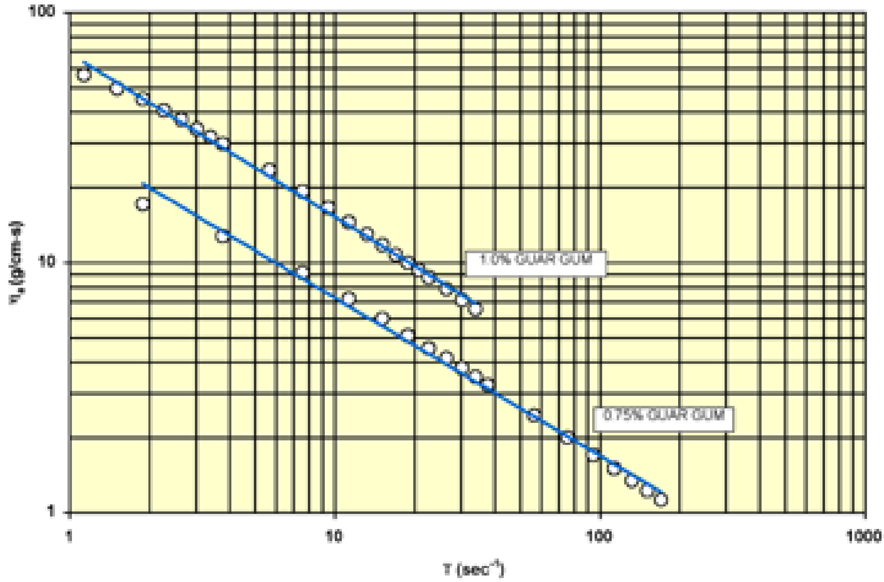

Fig. 1. Guar gum suspendsions: viscosity vs shear rate.

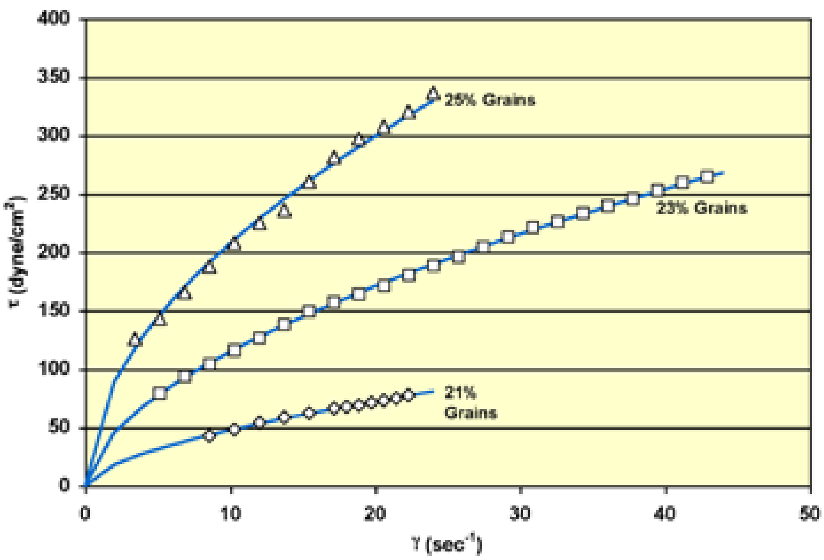

Fig. 2. Power Law model fit for distiller's grain slurries.

Wet Grain Slurries

Figure 2 shows the power law fits for the wet grain slurries. Table 5 summarizes the power law parameters for each slurry concentration. Figure 3 presents the experimental data fit to the Herschel-Bulkley model.

Table 3
Power Law Parameters for Guar Gum Solutions

Calibration solution	K_{pl}	n_{pl}	R^2
0.75% Guar gum	32.17	0.3534	0.9946
1.0% Guar gum	68.48	0.3469	0.9964

Table 4
Average Shear Rate Constants

Calibration solution	k_{avg}	k_{max}	k_{min}	SD
0.75% Guar gum	10.24	11.17	9.59	0.55
1.0% Guar gum	10.36	11.16	9.94	0.27
Overall	10.30			

Table 5
Power Law Parameters for Distiller's Grain Slurries

Slurry concentration	K_{pl}	n_{pl}	R^2
21% Solids	11.877	0.6019	0.9972
23% Solids	30.696	0.5727	0.9995
25% Solids	61.358	0.5291	0.9925

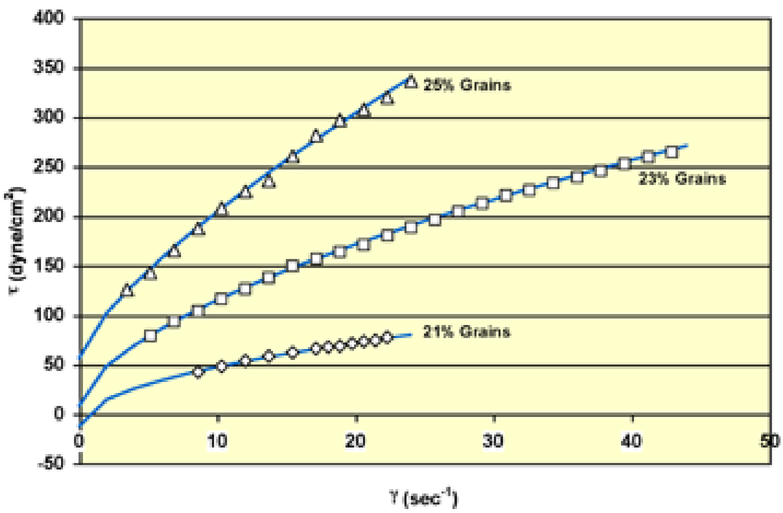

Fig. 3. Herschel-Bulkley model fit for distiller's grain slurries.

Table 6 summarizes the Herschel-Bulkley model parameters for each slurry concentration. Figure 4 represents the Casson model regression for 21, 23, and 25% grain slurries. Table 7 summarizes the Casson model parameters for each slurry concentration.

Table 6
Herschel-Bulkley Parameters for Distiller's Grain Slurries

Slurry concentration	K_{HB}	τ_{0HB}	n_{HB}	R^2
21% Solids	31.9580	–12.005	0.3919	0.9982
23% Solids	26.4580	8.306	0.6068	0.9996
25% Solids	26.9215	56.346	0.7409	0.9977

Table 7
Casson Parameters for Distiller's Grain Slurries

Slurry concentration	K_C	τ_{0C}	R^2
21% Solids	1.211	9.467	0.9972
23% Solids	1.697	28.478	0.9970
25% Solids	2.404	44.035	0.9977

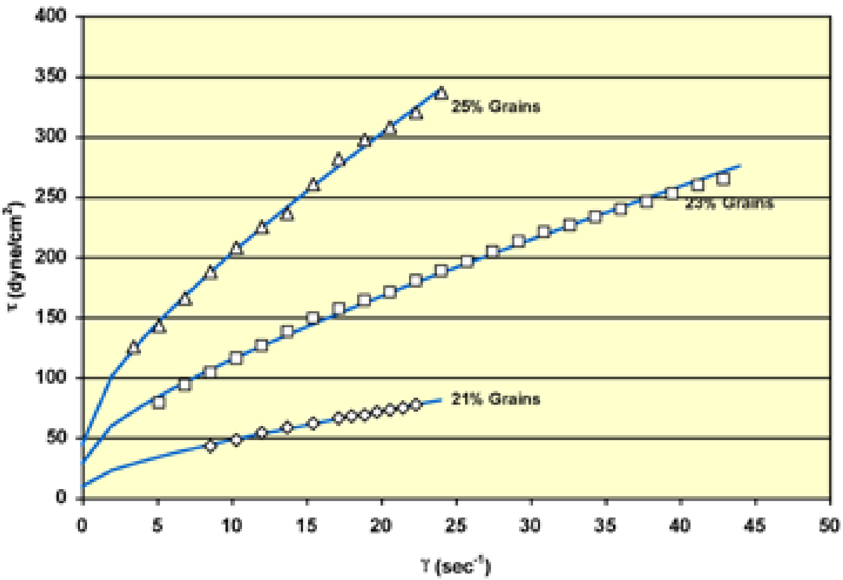

Fig. 4. Casson model fit for distiller's grain slurries.

Discussion

Calibration

The overall Newtonian calibration constant was 151, the same calibration constant obtained by Rieth *(3)* for the same helical impeller. Previously reported data by Donnelly *(4)* concluded that c was a function of R_e for vane and turbine impeller viscometers. It was concluded by Rieth *(3)* and confirmed in our study that c is a constant in the laminar region of flow (Re < 10) for the helical impeller viscometer. Table 2 shows that the SD from the average Newtonian calibration constant was 2.34 for glycerol and 1.50 for silicone oil 1M.

The regression analysis of the power law models for 0.75 and 1.0% guar gum suspensions returned R^2 values exceeding 0.99 and are shown in Table 3. The power law model was an accurate representation of guar gum rheologic properties.

Table 4 lists calculated average shear rate constants and SDs of 10.24 ± 0.55 (0.75% guar gum solution) and 10.36 ± 0.27 (1.0% guar gum solution). The SDs are minimal. The average shear rate constant range calculated by Rieth *(3)* was 10.45–10.91 for the same helical impeller. The calculated overall average shear rate constant of 10.30 falls below the range reported by Rieth *(3)*.

Limitations of Grain Slurry Rheology Measurement

Grain slurries of 21, 23, and 25% solids were tested using the helical impeller viscometer. The grain slurry concentration range was selected based on trial-and-error impeller experimentation. The lower limit of grain slurry concentration (21%) was bound by R_e restrictions. The impeller method required that the R_e be held at values <10. Grain slurries of concentrations <21% resulted in Reynolds numbers >10 for suitable impeller speeds. The upper limit of grain slurry concentration (25%) was bound by the mixing capabilities of the helical impeller viscometer. Grain slurries of concentrations >25% were not mixed sufficiently by the helical impeller viscometer and therefore did not return accurate impeller torque readings.

Figures 2–4 show that no experimental data were recorded at low impeller shear rates. Experimental data began at $\gamma = 8.53\ s^{-1}$ for 21% solids, 5.15 s^{-1} for 23% solids, and 3.43 s^{-1} for 25% solids. The reason for the missing data is that the helical impeller viscometer has limitations. Owing to possible viscometer error, data were not recorded until the impeller torque was >10% of the full-scale torque. Therefore, no experimental data were recorded at low impeller rotational speeds. The lack of experimental data at low shear rates made comparison of rheologic models at low shear rates and the prediction of yield stress impossible.

Grain Slurry Rheologic Modeling

Experimental rheologic data were fit to the power law, Herschel-Bulkley, and Casson models. The power law model does not predict yield stress. Yield stress for 21% grain slurries predicted by the Herschel-Bulkley model was a negative value, as shown in Table 6. Yield stress values predicted by the Herschel Bulkley model for 23 and 25% solids were 8.31 and 56.3 dyn/cm^2, respectively. Predicted yield stress values from the Casson model were 9.47 dyn/cm^2 for 21% solids, 28.5 dyn/cm^2 for 23% solids, and 44.0 dyn/cm^2 for 25% solids.

Grain Slurry Rheology Predictions

Overall, each model accurately represented the experimental data for shear rates >5 s^{-1}. At shear rates between 0 and 5 s^{-1}, the empirical models disagree. Since no experimental data exist at low shear rates, it is impossible to determine which empirical model best represents the actual properties of the grain slurries. Further, all of the empirical models have comparable regression coefficients, as shown in Tables 5–7. Regression coefficient (R^2) ranges were 0.992–1.000 (power law), 0.998–1.000 (Herschel-Bulkley), and 0.997–0.998 (Casson).

Conclusion

The helical impeller viscometer method is an accurate technique for approximating rheologic behavior of distiller's grain slurries of concentrations between 21 and 25% solids. Power law and Casson empirical models are adequate methods for modeling rheologic behavior of distiller's grain slurries of concentrations between 21 and 25% solids. These data should help in the design and operation of the agitator, especially for the start of the fermentation.

Nomenclature

c = Newtonian calibration constant
D_i = impeller diameter
k = non-Newtonian calibration constant
M = torque
n_{pl}, K_{pl} = power law parameters
N = rotational speed
P_N = dimensionless power number
Re = dimensionless Reynolds number
η_a = apparent viscosity
γ = shear rate
μ = Newtonian viscosity
ρ = density
τ = shear stress
τ_0, K_C = Casson parameters
τ_0, K_{HB} = Herschel-Bulkley parameters

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