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### Towards the development of a test-fluid for isothermal laboratory tests at low-shear rate in sugar and ethanol industry

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**Abstract.** *This manuscript presents the initial results of an ongoing project that have as goal the development and calibration of a test-fluid that could represent a set of molasses and massecoites from sugar and ethanol industry. The importance of viscous properties of complex fluids in that industry resides on the continuous need for increase in efficiency and maintenance of machinery. This paper presents a methodology to obtain a mixture of a Newtonian fluid and a solid granular material and to briefly characterize its viscous properties in low-shear rate for isothermal condition. Falling sphere viscometer was constructed to test glycerin-sugar mixtures at different concentrations and grain-size distribution, employing a set of different falling spheres. The results obtained show that the apparent viscosity of the mixture presents a classical behavior with the concentration and could be used to be associated to massecoites from industry. Further enhancements on this technique should be perform to better be able to predict friction coefficient.*

**Keywords:** *massecoite, rheology, non-Newtonian fluid, sugar and ethanol industry*

#### 1. INTRODUCTION

Food industry is one of the main sectors of capital production throughout the world. Since the beginning of human civilization, food and nutrition in large scales have been an important issue for societies development. Processes in food industry are in constant evolution to keep production efficient. Automation of food processing techniques have contributed to the increase in productivity. In this regard, one of the main products that have suffered continuous processes improvements is the sugar, combined with ethanol, in which Brazil stands out in the worldwide scenario being responsible for 40% of sugarcane production in 2016 (FAO, 2016).

In the process of obtaining sugar and alcohol, many different fluids are produced, presenting clear variations of consistencies (from liquid to slurry). They come from the industrial processing of the sugar juice, the first mixture of sucrose and water, originally extracted from sugarcane. At each stage of sugar processing (evaporation, boiling, crystallization, centrifugation, fermentation, distillation, etc.), the juice loses water content, thus increasing the concentration of sucrose, and change its properties. The fluids become increasingly rich in sucrose, process that facilitates the extraction of sugar as crystals in the final stage of the processing. The problem lies in the fact that these fluids are constantly pumped and driven from and to different sectors of the industry, demanding power and efficiency from the industrial machinery. The aim to industrial process designs is to guarantee that facilities work efficiently, ensuring process performance and infrastructure integrity.

Based on that problem, this manuscript presents preliminary results from an ongoing Scientific Initiation project in development at the Centro Universitário da Fundação Educacional de Barretos-SP. In this project, the research group aimed to constitute a non-Newtonian test-fluid that would have similar rheological behavior to the fluids present in the sugar extraction process under isothermal conditions. The elaboration of this fluid-type would facilitate assessments of the viscous characteristics of the mixtures. Consequently, their interaction with the devices that integrate the industrial process of sugar processing, such as conducts, accessories and pumps, could be evaluated.

The fluid-type was idealized as a mixture of solid granular material and viscous Newtonian fluid, both of easy access, aiming to reduce costs. A low-cost and expeditious method was used to estimate rheological properties at low-shear rate under laboratory conditions to reproduce the viscous characteristics of the industrial fluids.

## 1.1 Theoretical review

As presented by Rein (2007), water-based mixtures of sucrose present many different rheological behavior depending mainly on the temperature and on the sugar crystal concentration. The sugar juice, the first extract from sugarcane, presents a Newtonian behavior, as shows Eq. 1.

$$\tau = \mu \dot{\gamma} \quad (1)$$

where the relation between shear rate  $\dot{\gamma}$  and shear stress  $\tau$  is linear, given by the fluid dynamic viscosity  $\mu$ . As process advances, water content is lost, thus increasing dissolved sucrose proportion into the mixture. At this point, fluids are called molasses, and usually present Ostwald-de-Waele behavior, as point out Eq. 2 by many author (Rouillard and Koenig, 1980; Rein, 2007; Bofu *et al.*, 2016).

$$\tau = K \dot{\gamma}^n \quad (2)$$

where the consistency index  $K$  appears, analogous to the Newtonian viscosity, and a non-linear relationship between shear rate and shear stress is defined through the flow index  $n$ . As sugar crystals begin to form, the fluid, now called massequite, present a sharply increase in consistency, which is mainly dependent on the volume concentration of solid particles of sugar  $\phi$  in the mixture. Broadfoot *et al.* (1998) showed that such relation can be written as function of the consistency of the molasses  $K_0$  and concentration, as shows Eq. 3.

$$K_m = K_0 \left[ 1 + \frac{3.8\phi}{1 - \phi/\phi_{max}} \right]^{1.4} \quad (3)$$

where  $K_m$  is the consistency of the massequite, and  $\phi_{max}$  is the maximum volume concentration for the solid granular material in the mixture. For cubic-shaped particles,  $\phi_{max} = 0.52$ . The relationship expressed by Eq. 3 follows the same type as other materials such as aqueous mixture of clay/sand particles (Bournonville and Nzihou, 2002; Alderman and Heywood, 2004; Mueller *et al.*, 2010). Senapati *et al.* (2010), for example, bring a non-linear relationship between apparent viscosity of slurry mixtures and the concentration of particles in suspension that follows Eq. 4, dependent on the shear rate.

$$K_m = K_0 S \left[ 1 + \frac{B}{\dot{\gamma}^n} \left( \frac{\phi}{\phi_{max} - \phi} \right) \right]^N \quad (4)$$

which presents a various of other parameters more suitable to non-Newtonian fluids.  $S$  represents the uniformity of the solid material,  $B$  represents the intrinsic viscosity, and approximates to the classical value once found by Einstein and Fürth (1956) of 2.5, and  $N$  is an exponent often calibrated empirically.

Fluid viscous properties should be known beforehand in order to estimate the friction factor that will be established and, therefore, the head loss. This must be an important part of the project and design of industry facilities and devices that are going to be used to drive the fluid through the process. Many kind of viscometers are present in the market that can be useful instruments to give an insight on the rheological properties of the fluid.

Simplified viscometers usually detect the apparent viscosity of a fluid  $\eta = \tau/\dot{\gamma}$ . At a given shear stress, the sensor detect the shear rate which the fluid is subdue, and thus is able to calculate  $\eta$ , or vice-versa. For a Newtonian fluid, using Eq. 1, it is direct that  $\eta = \mu$ . For a non-Newtonian fluid, however, a dependence on the shear ratio is still at stake, as shows Fig. 5.

$$\eta(\dot{\gamma}) = \frac{\tau}{\dot{\gamma}} = K \dot{\gamma}^{n-1} \quad (5)$$

One of the classical viscometers was designed after the derivation of the frictional force a fluid applies to a spherical object falling in a Newtonian test-fluid by George G. Stokes, in 1851. A sphere with known diameter  $D_e$  and known density  $\rho_e$  is let fall inside the test-fluid of known density  $\rho_f$ . As the sphere moves downward the fluid, it gains speed, and frictional force increase, contrary to the movement. At some point, balance between the forces that act on the submerging body (gravity, friction and buoyancy) achieve equilibrium, and the sphere submerges at constant velocity  $V_l$ . Measuring

the velocity  $V_l$  allows the estimative of the apparent viscosity of the fluid through Eq. 6.

$$\eta = \frac{1}{18} \frac{D^2 g (\rho_e - \rho_f)}{V_l} \quad (6)$$

The concept of this kind of flow was then idealized as Stokes flow, where the Reynolds number, as defined in Eq. 7, is much less than the unity ( $Re \ll 1$ ).

$$Re = \frac{\rho_f V_l D_e}{\eta} \quad (7)$$

## 2. METHODOLOGY

In order to properly develop a falling sphere viscometer, which is a practical and non-expensive method, some characteristics should be attended. The equipment used was a 1000 ml beaker, graduated, marked in its height with millimeters, and a chronometer. In this way, sphere vertical displacement in the fluid inside the beaker was timed, and the velocity found. Three metallic spheres were employed that presented different diameters  $D_e = [2.65, 3.52, 5.98]$  mm and were made from the same material (Lead,  $\rho_e = 11340 \text{ kg/m}^3$ ). General mounting is showed in Fig. 1.

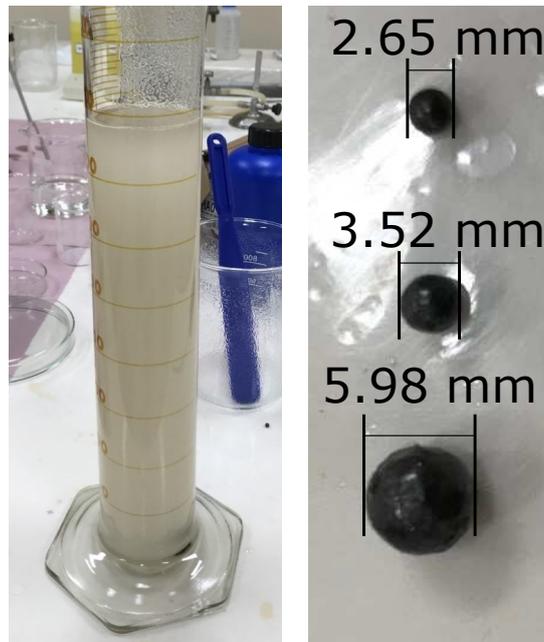


Figure 1. Beaker filled with mixture of glycerin and sugar crystals, and lead spheres used to measure apparent viscosity of the fluid.

The experiment firstly focused on the qualitative observations about possible combinations of solid granular materials and highly viscous Newtonian or non-Newtonian fluids to set the material that should be used. The characteristics that were observed was:

- mixture transparency at various concentrations - it is of utmost importance that the spherical object that will be launched into the test-fluid can be easily followed by the observer;
- settling velocity of granular material in suspension - to reduce possible changes in mixture rheological behavior, the settling velocity of the solid granular material should be low, so no segregation would occur;
- solubility - the packing phenomena is the effect responsible for the relationship between apparent viscosity and the concentration, previous showed. In this case, granular material should not be soluble within the fluid, otherwise the geometric effect of its volume in the mixture will be compromised.

Employing the qualitative method explained before, the granular material chosen was the commercial sugar ( $\rho_s = 1587 \text{ kg/m}^3$ ) which have a regular cubic shape ( $\phi_{max} = 0.52$ ), and the fluid was glycerin ( $\rho_l = 1260 \text{ kg/m}^3$ ,  $\mu_0 =$

0.72 Pa.s), a Newtonian fluid. The sugar was sorted based on its diameter size  $D$ , or grain-size distribution, following NBR-7217. Two characteristic sizes were selected: 0.223 mm and 0.112 mm. Properties of both granular and fluid are summarized in Tab. 1.

Table 1. Properties of the materials employed on the constitution of the the test-fluid (mixtures).

Sugar (crystal grains)		Glycerin	
$D$ (mm)	[0.223; 0.112]	$\mu_0$ (Pa.s)	0.72
$\rho_s$ (kg/m <sup>3</sup> )	1587	$\rho_l$ (kg/m <sup>3</sup> )	1260

Finally, tests with the viscometer were conducted, following the guidelines below:

1. measure the apparent viscosity of the fluid;
2. prepare mixture of solid granular material with the fluid at a given volume concentration  $\phi$ .
3. conduct quantitative measurements of settling velocity of metallic spheres of different diameters  $D_e$  for the prepared mixture.
4. calculate apparent viscosity for each test and perform statistical analysis to find mean value and uncertainties related to the measurements.

### 3. RESULTS AND DISCUSSIONS

In this first stage of project, 28 measurements were conducted, for different concentrations, ranging from 5% to 40%. The measured viscosity for the pure glycerin ( $\phi = 0\%$ ) was  $\mu_0 = 0.72$  Pa.s. For mixtures, measured apparent viscosity  $\eta$  ranged from 1.14 to 9.41 Pa.s. Uncertainties of measurement ranged from 1.0% to 6.2%, being the greater values those where the Reynolds number was higher than the expected for a Stokes flow ( $Re > 0.1$ ), where Stokes classical law was predicted not to capture all the physics behind the phenomenon (Oseen, 1910; Goldstein, 1929).

Plotting  $\eta$  against  $\phi$ , Figure 2 is obtained. The interpolated curve that represents the function  $\eta(\phi)$  was generated aiming to obtain a similar curve to the Eq. 3. Equation 8 presents the interpolated function, with  $R^2 = 0.88$ .

$$\eta = \mu_0 \left[ 1 + A \left( \frac{\phi}{1 - \phi/\phi_{max}} \right) \right]^B \quad (8)$$

where  $\mu_0 = 0.72$  Pa.s,  $A = 12.79$  and  $B = 0.85$ .

The coefficients found (Eq. 8) stand apart from the ones calculated/interpolated by Broadfoot *et al.* (1998). Nevertheless, differences in the constitutions of the test-fluid (nature of fluid and solid materials) could have causes the discrepancies observed. The aim in this initial work was to detect the general behavior  $\eta(\phi)$ , which was found to be acceptable and in agreement with the literature. Many other relationships can be tested to find the best interpolation.

However, one particular observation that has been made is that the shear rate could slightly vary from one test to another. Using an approximation for the shear rate as presents Eq. 9, Fig. 3 can be constructed that shows the variations for  $\dot{\gamma}_m$  for a each fixed value for  $\phi$ .

$$\dot{\gamma}_m = \frac{V_l}{D_e} \quad (9)$$

It was noted that a possible linear relationship with negative coefficient exists between both variables, as shows Eq. 10, with  $R^2 = 0.57$ . The high discrepancy found relates to the single point far away from interpolated curve which represent a test where  $Re = 0.6$ . Ignoring that test,  $R^2 = 0.76$ . This relationship, however, could be the result of the increase on apparent viscosity, which would lead to an increase in the frictional force, decreasing sphere velocity of submersion. Nevertheless, both are not linearly combined, since a rational function for  $\eta(\phi)$  was found and a first-order polynomial function was found for  $\dot{\gamma}_m(\phi)$ .

$$\dot{\gamma}_m = A + B\phi \quad (10)$$

with  $A = 14.67$  and  $B = -32.96$ .

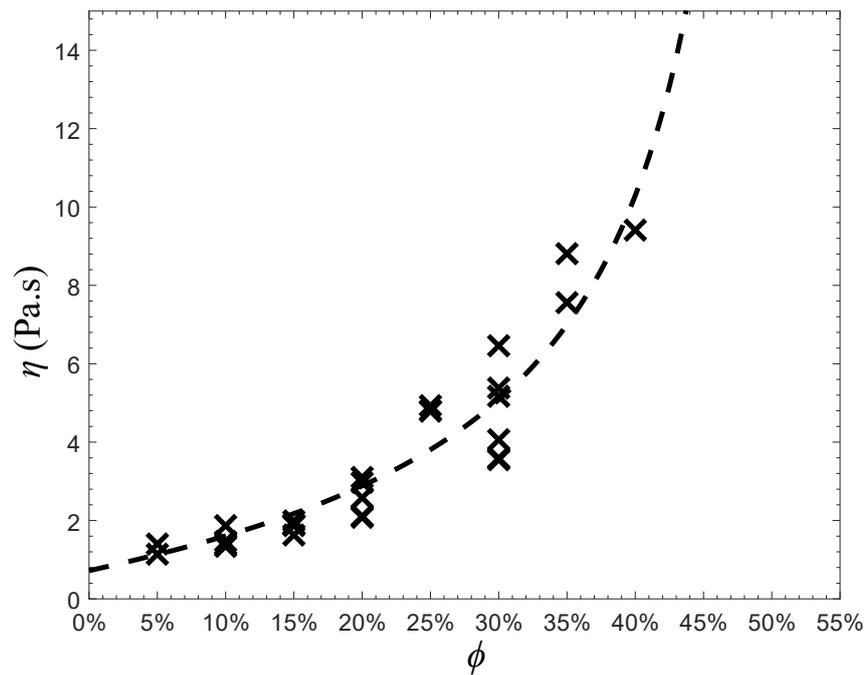


Figure 2. Apparent viscosity  $\eta$  against volume concentration of sugar  $\phi$ . Crosses indicate measurements and dashed line the interpolated curve (Eq. 8).

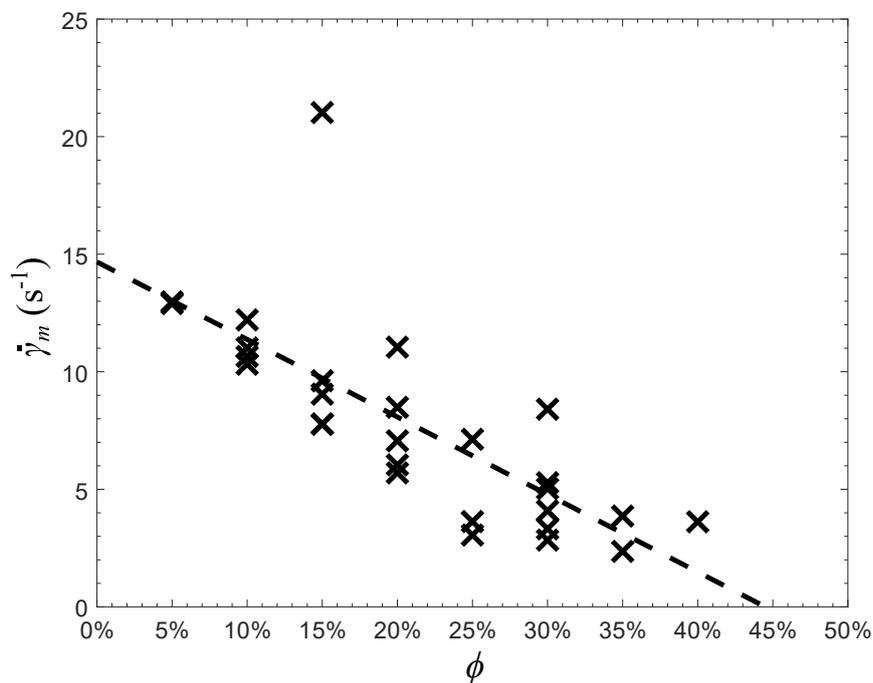


Figure 3. Estimated shear rate  $\dot{\gamma}_m$  against volume concentration of sugar  $\phi$ . Crosses indicate measurements and dashed line the interpolated curve (Eq. 10).

#### 4. FINAL REMARKS

As an ongoing project, the results presented so far indicate that the method employed can give some insights of the test-fluid composed, and how they could be used to represent masseccites from sugar and ethanol industry. Figure 4 shows the results here found combined to temperature-dependent measurements of apparent viscosities reported by Rein (2007). Figure 4 demonstrates (dashed box) that the fluid here developed could be used to represent masseccite (A type) in high

temperatures as well as molasses (A, B, and C types) in different temperature conditions.

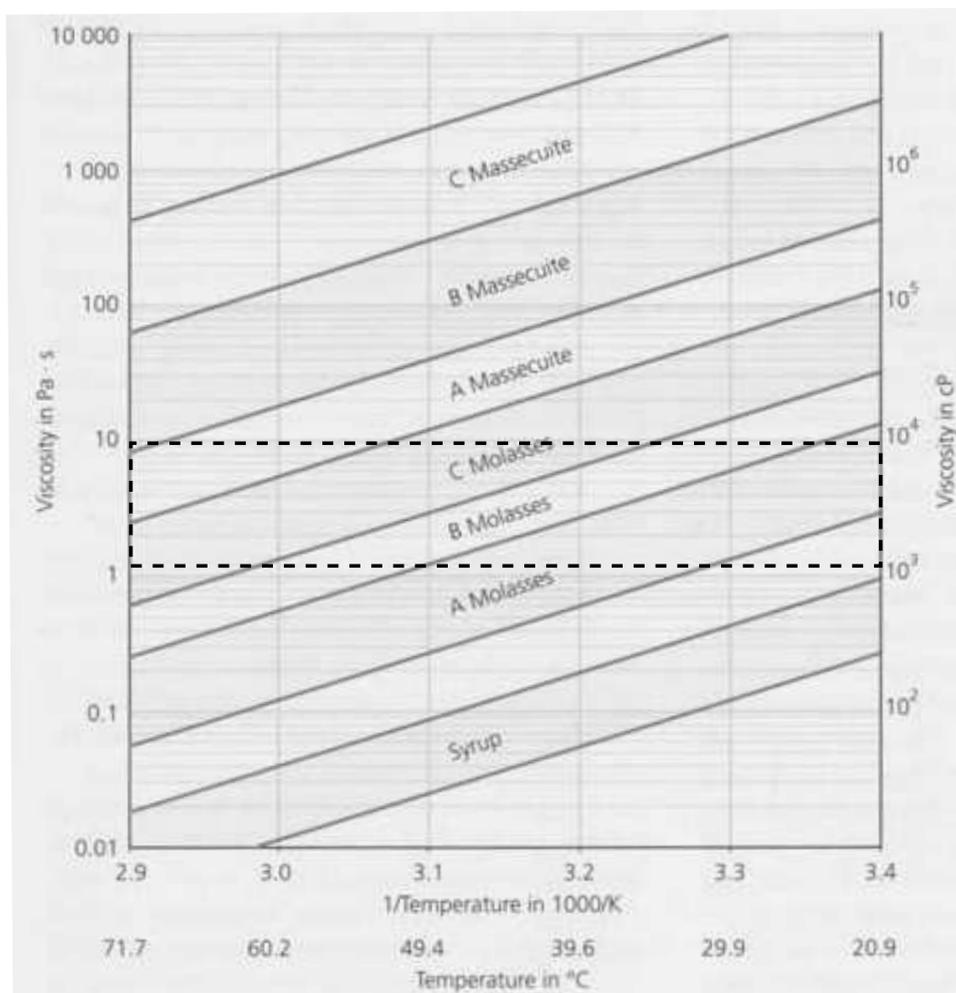


Figure 4. Correspondence of the test-fluid developed to the rheological properties measured for massecuites. The dashed box represent possible zone of utilization of the test-fluid.

However, further experimental tests should be performed to verify the variability of shear ratio based on different falling spheres. This could help estimate further properties of power-law fluids such as the consistency index and flow index which are important to calculate head loss. The relations that may represent the goal of this research could be further used to pinpoint the composition of a specific fluid to perform a pump-test, for example, without need for thermal exchanges, at any shear ratio.

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