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Original

Rheological behavior of corn and soy mix as feed ingredients

Comportamento reológico de mistura de milho e soja para produção de rações

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Abstract

Foods behave as non-Newtonian fluids, but little is known about how corn and soybean mix behave under viscometric flow. In order to characterize the rheological behavior of animal feed under viscometric flow, a 70:30 (mass:mass) mixture of ground corn and soybean grains was submitted to a capillary rheometer at 3 different temperatures (80, 120, and 160 °C), different moisture levels (26.5 ± 0.08; 30.4 ± 0.31, and 33.4 ± 0.05%), and 4 shear rates (30.4; 72.9; 304.3, and 728.6/second). Different strain rates and die dimensions were used to obtain the target shear rates. The resulting data were fitted to Power Law, Casson, and Bingham models. Based on experimental data, water content, mass temperature, and the effects of shear rate on the apparent shear viscosity of corn-soy mix were fitted to a single expression ($p < 0.001$, $R^2 = 0.93$): $\eta = 18{,}769.7\ (\dot{\gamma})^{-0.86}\ e^{(-9.34\,U + 935\,T)}$, where $\dot{\gamma}$ is shear rate, U is sample moisture, and T is sample temperature in Kelvin scale. As expected, such mixture presented a pseudoplastic (shear-thinning) behavior.

Keywords: *viscometric flow; shear; shear-thinning; capillary rheometer.*

Resumo

Alimentos comportam-se como fluidos não newtonianos, porém pouco se sabe do comportamento de milho e soja sob escoamento viscométrico. Para caracterizar o comportamento reológico de alimentos para animais, uma mistura de grãos de milho e soja moídos na proporção 70:30 (massa:massa) foi submetida ao reômetro capilar sob 3 níveis de temperatura (80, 120 e 160 °C), umidade da massa (26,5 ± 0,08; 30,4 ± 0,31; e 33,4 ± 0,05%), e 4 taxas de cisalhamento aparente (30,4; 72,9; 304,3; e 728,6/second). Diferentes taxas de deformação e dimensões da matriz foram utilizadas para obtenção das taxas de cisalhamento acima. Os dados obtidos foram ajustados para os modelos da Lei da Potência, Casson e Bingham. Baseados nos dados experimentais, os efeitos de umidade, temperatura da massa e taxa de cisalhamento sobre a mistura de milho e soja foram ajustados para uma expressão única ($p < 0{,}001$, $R^2 = 0{,}93$): $\eta = 18.769{,}7\ (\dot{\gamma})^{-0{,}86}\ e^{(-9{,}34\,U + 935\,T)}$, onde $\dot{\gamma}$ é a taxa de cisalhamento, U é a de teor de água na amostra e T é a temperatura na massa, em escala Kelvin. Como esperado, a mistura de milho e soja moídos apresentou comportamento pseudoplástico.

Palavras-chave: *escoamento viscométrico; cisalhamento; pseudoplástico; reômetro capilar.*

1 Introduction

The current technological status of animal feed production justifies the use of ingredient and feed processing procedures, such as expansion, extrusion, and pelleting.

The mechanical behavior of those ingredients and their interaction with processing machinery are reflected in operating parameters such as: energy demand, operating capacity, and equipment use, as well as in the final properties of products, such as level of expansion and cooking, coloring, density, and texture.

It is known that extruded foods behave as non-Newtonian fluids, and their viscosity can be described by the Power Law model (CARVALHO; ASCHERI; MITCHELL, 2004; LI; CAMPANELLA; HARDACRE, 2004; SINGH; SMITH, 1999; EERIKAINEN; LINKO, 1998; RAO; ANANTHESWARAN 1982; REMSEN; CLARK, 1978). However, little is known about the rheological behavior of ingredients submitted to the characteristic processes in the feed industry.

In Brazil, corn and soy grains are the main ingredients in animal feed accounting for around 80% of its mass production (SINDIRAÇÕES, 2007). According to Steffe (1996), the shear rate generated during the cereal and animal feed extrusion process varies from 0 to 10^3/second, and the temperature and pressure may range from 80 to 160 °C and from 1,500 to 6,000 kPa, respectively.

Capillary rheometry (Figure 1) is a technique that allows one to measure the rheological properties of materials subjected to a broad range of shear rate and temperature, provided one may assume that flow is laminar and constant, entry and exit effects at the capillary die are negligible, fluid is incompressible, rheological properties are not affected by time and pressure, temperature is constant, and no fluid slippage at the capillary wall occurs (DARBY, 1979).

Figure 1 illustrates the fluid flow through a capillary die of diameter D and length L. The total applied pressure is P_A and the capillary exit pressure is P_{EX} (atmospheric). Extensional and shear viscosity can be estimated from the pressure drop within the cylinder and within the capillary, respectively.

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The purpose of this work was to characterize the rheological behavior and the flow velocity profile of corn and soy mix under viscometric flow.

2 Materials and methods

Sample conditioning and preparation were carried out at Faculdade de Engenharia Agrícola, UNICAMP, Brazil. Corn and soy grains mixed at a proportion of 70:30 (mass) were ground in a hammermill, using a 1.5 mm mesh sieve. Samples were collected in order to determine the centesimal composition and particle size in triplicates. Aliquots of this material were conditioned by adding and mixing water at 15, 20, and 25% with an electric mixer, which were then placed in plastic bags and left to rest for 18 hours at 5 °C in order to bring mass moisture to equilibrium. The material was then vacuum-sealed and stored at 5 °C. Aliquots were collected to determine the water content using the gravimetric method. Two-gram aliquots in duplicate were weighed on an analytical scale and dried in a forced-air oven for 24 hours at 105 °C. The measurement of the average geometric diameter of the samples followed ANSI/ASAE standard S319.3 method (AMERICAN..., 2004). True density was determined using the Archimedes method (MOHSENIN, 1986) with the use of 98° ethanol instead of water to prevent particles to float. The centesimal composition of the sample was determined according to AOAC methodology (ASSOCIATION..., 1984).

The rheology tests were conducted at Centro de Tecnologia, UNICAMP, Brazil. The capillary rheometry method was used, as described by Rauwendaal (1994), using a capillary rheometer, as described by Fraiha et al. (2007), coupled to a universal testing machine (MTS, Sintech 5G). The data acquisition system, including the universal testing machine and the load cell, was calibrated for this experiment, and the expanded measurement uncertainty was ≤ 0.12%, coverage factor > 2.0 for a specific measurement range of 0 to 500 kgf (Calibration Certificate n. 241 06CT2007).

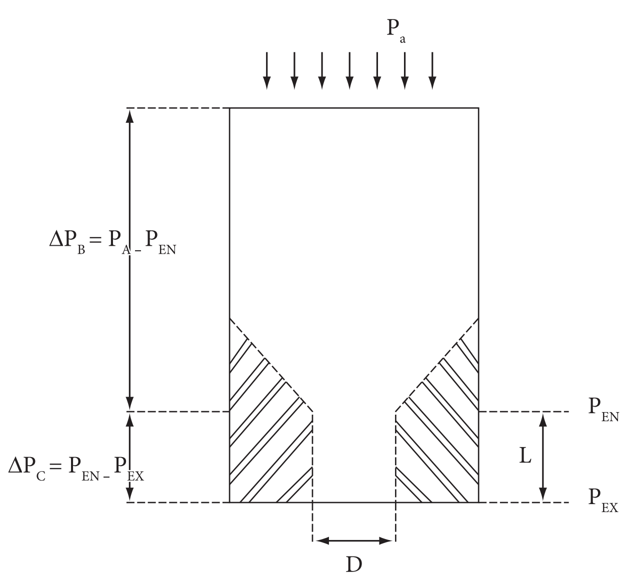

Figure 1. Illustration of fluid flow through a capillary die of diameter D and length L. The total applied pressure is P_A, pressure at die exit is P_{EX} (atmospheric), and P_{EN} is the pressure at capillary entrance. Extensional and shear viscosity can be determined from the pressure drop within the cylinder and within the capillary, respectively.

The experiment consisted of recording the force curve during extrusion of 50 g of the sample through the capillary rheometer, combining dies of different dimensions and piston displacement velocities, so as to obtain pre-defined shear rates. The die dimensions used ranged from 3 to 6 mm inner diameter (D) and had a length to diameter ratio of 10. Piston displacement velocities ranged from 0.4 to 4 mm/second.

The experimental design was completely randomized, in a 3×3×4 factorial scheme with 3 levels of temperature and moisture of the mass and 4 levels of apparent shear rate. Data were collected in four replicates.

The material moisture levels were obtained via the previously described conditioning procedure and were 26.5 ± 0.08; 30.4 ± 0.31, and 33.4 ± 0.05% (mean ± standard deviation). The mass temperature in the rheometer was controlled by an electrical resistance-based heating system, at 80, 120, and 160 °C, allowing 3-minute come-up time for temperature equilibrium. Preliminary observations indicated that moisture loss at the highest temperature values were negligible. Apparent shear rate ($\dot{\gamma}_{ap}$) was estimated using Equation 1 and was fixed at 30.4; 72.9; 304.3; and 728.6/second.

$$\dot{\gamma}_{ap} = \frac{32Q_c}{\pi D^3} \quad (1)$$

where Q_c is the volumetric flow in the capillary, determined by piston area (πD_p^2) and displacement velocity (v_p) (Equation 2):

$$Q_c = \frac{v_p \pi D_p^2}{4} \quad (2)$$

Shear tension on the capillary wall, τ_{pc}, can be associated to a pressure drop along the capillary (ΔP_c) using the flowing Equation 3:

$$\tau_{pc} = \frac{\Delta P_c D_c}{4L_c} \quad (3)$$

Pressure loss due to convergence of the capillary walls and loss of kinetic energy at the capillary entrance were corrected by measuring the force required for the material to flow through dies of same diameter as the test dies, but zero length ($L_c = 0$), and then subtracted from force measured with test dies. Thus, ΔP_c can be calculated (Equation 4):

$$\Delta P_c = \frac{4\left[F_p\left(L_c\right) - F_p\left(L_c = 0\right)\right]}{\pi D_p^2} \quad (4)$$

where D_p is the piston diameter and F_p is the force applied to the piston.

Figure 2 shows a characteristic force-strain curve obtained by displacement of the rheometer piston through time.

To calculate ΔP_c, a mean force value (F) is obtained from the force-strain curves generated during the experiment, considering only the data from region β, as exemplified in Figure 2. Fmax represents the necessary force to initiate the material flow through the capillary die, whereas β represents

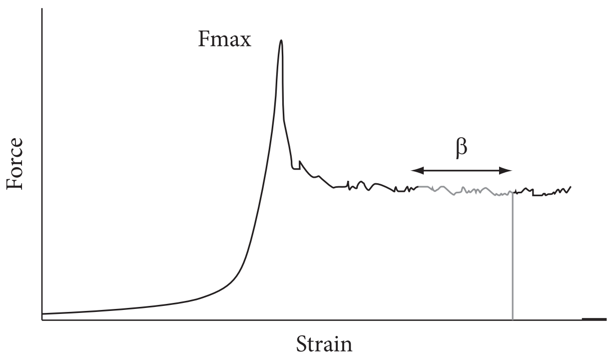

Figure 2. Force as a function of the strain imposed to the sample in the capillary; Fmax - force necessary to initiate flow; β - portion of the curve where data is extracted for calculations.

the piston displacement required to flow a quantity of mass that is equivalent to the internal volume of the test die. The distance between Fmax and the start of β is equal to β, meaning that a volume of mass is flowed before data collection; therefore, the data obtained for the calculations coincides with the stable force response to imposed strain. This procedure enables one to acquire average values with variation coefficients of about 5%.

The estimated shear rate on the capillary wall (γ'_{pc}) was obtained by applying the Rabinowitsch correction to the apparent shear rate (Equation 5):

$$\dot{\gamma}_{pc} = \left(\frac{3n+1}{4n}\right)\dot{\gamma}_{ap} \tag{5}$$

where *n* is the flow behavior index (Equation 6):

$$n = \frac{\left(d \log \dot{\gamma}_{ap}\right)}{\left(d \log \tau_{pc}\right)} \tag{6}$$

Apparent viscosity (η) could then be determined (Equation 7):

$$\eta = \frac{\tau_{pc}}{\dot{\gamma}_{pc}} \tag{7}$$

The rheological behavior of the samples was represented by the Power Law (lp) and Bingham (bg) and Casson (cs) models, according to respective Equations 8, 9 and 10:

$$\eta_{lp} = K_{lp}\left(\dot{\gamma}_{pc}\right)^{n-1} \tag{8}$$

$$\eta_{bg} = K_{bg} + \frac{\tau_0}{\dot{\gamma}_{pc}} \tag{9}$$

$$\eta_{cs} 0.5 = K_{cs} + \left(\frac{\tau_0}{\gamma_{pc}}\right)^{0.5} \tag{10}$$

where K_{lp}, K_{bg} and K_{cs} are the consistency indexes of the samples, τ_0 is the minimum force to initiate the flow through the capillary, as determined by the extrapolation method when $\dot{\gamma}_{pc} = 0$ (STEFFE, 1996).

The assessment and correction of slip at the capillary wall followed the methodology described by Darby (1979) using dies of different inner diameters and same length to determine the slip coefficient δ in order to estimate slip-corrected shear rate, $\dot{\gamma}_{ap,c}$ (Equation 11):

$$\gamma_{ap,c} = \gamma_{ap} - \frac{8\delta\tau_{pc}}{D} \tag{11}$$

The mass temperature and moisture also influence the viscosity of a material. Shear rate, temperature and moisture of the mass can be combined into a single expression (HARPER; RHODES; WANNINGER, 1971) (Equation 12):

$$\eta = f\left(\gamma', U, T\right) = K_{\gamma'} U T \left(\gamma'\right)^{n-1} . e^{\left[B(U)+A(T)\right]} \tag{12}$$

where the influence of shear rate is represented by the Power Law model, U is the material moisture (percentage), and T is the inverse of the testing temperature, in Kelvin scale.

The assumption of laminar flow in the capillary is a theoretical fundament in this discussion, and it is assessed by calculating the Reynolds number (N_{Re}) (RAO; ANANTHESWARAN, 1982). A fluid governed by the Power Law (lp) presents laminar flow if (Equation 13):

$$N_{Re,lp} < 6464n\left(2+n\right)^{\frac{(2+n)}{(1+n)}}\left(1+3n\right)^{-2} \tag{13}$$

where *n* is the flow behavior index (*n* < 1 for pseudoplastic fluids and *n* = 1 for Newtonian fluids).

The Reynolds number for Power Law fluids is given as (Equation 14):

$$N_{Re,lp} = \left[D^n \left(v_m\right)^{2-n} \frac{\rho}{\left(8^{(n-1)}K\right)}\right]\left(\frac{4n}{3n+1}\right)^n \tag{14}$$

where *D* is the capillary diameter, *vm* is the mean velocity flow through the capillary die, *ρ* is the true density of sample, and *K* is the consistency coefficient of the fluid ($K = K_{lp}$) (STEFFE, 1996).

The velocity profile of laminar flow in a cylinder can be estimated via Equation 15 (RAO; ANANTHESWARAN, 1982):

$$\frac{v}{v_m} = \left(\frac{3n+1}{n+1}\right)\left[1-\left(\frac{r}{R}\right)^{n+1/n}\right] \tag{15}$$

where *v* is the flow velocity at radial position *r* and *vm* is the average flow velocity in the capillary, estimated from the volume of material flow due to piston displacement.

3 Results and discussion

The ash, ether extract, and crude protein content were 2.66 ± 0.09; 7.61 ± 0.07; and 19.7 ± 0.81% (mean ± standard error), respectively (dry matter basis).

The average geometric diameter was 542.39 ± 42.98 µm. The particle size may influence the sample viscosity (SERVAIS; JONES; ROBERTS, 2002). However, only very few authors provided particle size and the centesimal composition profile of the material studied; therefore a perfect comparison of results is difficult.

Based on the original force and strain data, it was possible to estimate the apparent viscosity (η) of the samples at different moisture, temperature, and shear rate levels. The consistency coefficient, *K*, the flow behavior index, *n*, as obtained using the Power Law model, and *K*, the minimum shear force required to initiate flow, τ_0, as obtained through the Casson and Bingham models for the different experimental conditions, are given in Table 1.

The coefficients of determination for the Casson and Bingham models, which takes τ_0 into account, were not better than those for the Power Law model. Alavi, Chen and Rizvi (2002) also recorded the same observation while modeling the rheological behavior of wheat starch. The existence of a true yielding behavior has been the subject of prolonged debate among rheologists since yield stress values are typically calculated by extrapolation of data (STICKEL; POWELL, 2005; TIN; GUO; UHLHERR, 2006)

Under the conditions of this experiment, the mixture of ground corn and soy grains presented a pseudoplastic behavior with a strong decline in apparent viscosity as shear rate increased, as indicated by the value of *n* in the Power Law model (Table 1). Sandoval and Barreiro (2007) found similar behavior for corn grits when using capillary rheometry and experimental conditions very similar to the present experiment

The utilization of the Power Law model to describe the flow behavior of cereal grains has been confirmed by several authors (LI; CAMPANELLA; HARDACRE, 2004; CARVALHO; ASCHERI; MITCHELL, 2004; ALAVI; CHEN; RIZVI, 2002; SINGH; SMITH, 1999; BAGLEY; DINTZIS; CHAKRABARTI, 1998; PADMANABHAN; BHATTACHARYA, 1991; SENOUCI; SMITH, 1988).

Recently, Viamajala et al. (2009) utilized Casson and Bingham models to describe the rheological behavior of corn stover slurry at high solid concentration. The Herschel-Buckley model is also used for some authors and predicts a yield stress like the Casson and Bingham models, but its evaluation is less certain (NGUYEN; BOGER, 1992; PIMENOVA; HANLEY, 2004)

Carvalho, Ascheri and Mitchell (2004), in their work with corn grits submitted to capillary rheometry under test temperatures from 90 to 120°C and shear rates from 10 to 500/second, determined values of *n* ranging from 0.16 to 0.52, and of *Klp* from 2.6 to 97.8 kPa.s^n. Singh and Smith (1999) obtained values of *n* and *Klp* ranging from 0.17 to 0.51 and 3 to 93 kPa.s^n, respectively, for wheat flour with a water content from 21 to 28% and at temperatures of 100 to 125 °C.

The negative *n* index obtained for the samples with 33.4% water content at 120 °C (Table 1) is difficult to explain although the negative flow behavior indexes had already been reported in the past. Padmanabhan and Bhattacharya (1991), studying the behavior of ground corn with moisture levels ranging from 25 to 45% at temperatures ranging from 150 to 180 °C, obtained $-0.193 \le n \le 0.811$ and $1.42\times10^2 \le Klp \le 7.52\times10^5$. These authors varied the apparent shear rate from 163 to 652/second, and thereby obtained an apparent viscosity ranging from 98 to 1276 Pa.s, values which are close to the ones obtained in the present study. Their hypotheses to explain the negative values of *n* include molecular degradation of sample, viscous dissipation, fluid slip along the capillary wall, and the influence of τ_0 although the contribution of each of these factors was not clear. In the present experiment, sample slip may be involved, which could explain the negative *n*.

Few authors assessed the presence of slip on the capillary wall. In the present experiment, the corrections resulted in inconsistent data attributing negative values to $\dot{\gamma}_{ap,c}$. Other authors have obtained similar results and could not proceed with the correction (MACKEY et al., 1989; SINGH; SMITH, 1999).

Based on the experimental data, excluding those with a negative *n*, the constants $K_{\gamma' UT}$, $\overline{n}$, B, and A (Equation 12) were determined by multiple regression ($p < 0.001$, $R^2 = 0.93$), resulting in the expression (Equation 16):

$$\eta = 18769.7(\dot{\gamma})^{-0.86}\, e^{(-9.34U + 935T)} \tag{16}$$

A comparison of the observed data and the estimated data from the general model, including the effects of shear rate, based on the Power Law model, water content, and temperature (Equation 16), is shown in Figure 3.

Under the conditions of this experiment, with n = 0.14, the flow is considered laminar if $N_{Re,\,lp} < 1876.2$.

In this experiment, the capillary flow could be estimated by considering the maximum piston displacement velocity as

Table 1. Consistency coefficients (K), flow behavior indexes (n) as determined by the Power Law model, and (K) and minimum shear forces required to initiate flow (τ_0), as determined by the Casson and Bingham models for the different experimental conditions.

Experimental conditions	Power Law			Casson			Bingham		
U% - T °C	K_{lp}(kPa.s^n)	n	R^2	K_{cs}(kPa.s^n)	$(\tau_0)^{0.5}$(kPa)	R^2	K_{bg}(kPa.s^n)	τ_0 (kPa)	R^2
26.5 - 80	17.60	0.20	0.96	0.85	0.195	0.95	−13.90	44.4	0.97
26.5 - 120	22.54	0.13	0.99	1.17	0.180	0.98	8.52	36.3	0.99
26.5 - 160	18.95	0.14	0.96	1.86	0.155	0.96	21.37	27.7	0.98
30.4 - 80	6.71	0.31	0.91	1.47	0.148	0.86	−14.84	29.0	0.90
30.4 - 120	20.17	0.06	0.99	0.42	0.151	0.99	2.22	24.2	0.99
30.4 - 160	9.00	0.15	0.95	1.81	0.101	0.92	21.04	11.5	0.87
33.4 - 80	7.81	0.28	0.96	2.09	0.138	0.97	10.44	23.9	0.98
33.4 - 120	22.11	−0.05	0.99	−0.23	0.141	0.99	0.95	19.2	0.99
33.4 - 160	10.01	0.12	0.92	1.93	0.095	0.82	24.80	9.8	0.70

U – mass moisture (bu); T – mass temperature during the experiment, R^2- coefficient of determination, total of four replicates.

4 mm/second, and the inner diameter of the rheometer cylinder as 24.3 mm. Hence, the average velocity flow (*vm*) through the capillary with 3 mm diameter was estimated as 262.4 mm/second. The true density (ρ) was previously determined as 1300 kg.m^{-3}. Based on these data, the Reynolds Number was estimated as 0.37, indicating that flow is laminar, thereby fulfilling a basic theoretical assumption made in this experiment.

The flow velocity profile of an incompressible fluid through a cylindrical canal may be estimated. Under steady state flow, a ring-shaped portion of the fluid next to the canal wall presents a velocity gradient, where parts of the fluid move in relation to their adjacent areas (Figure 4). In the central region of the canal, delimited by *r*, the fluid flows freely without forming a velocity gradient.

Figure 5 shows the velocity profile of a fluid submitted to viscometric flow as a function of radial position in relation to the capillary wall.

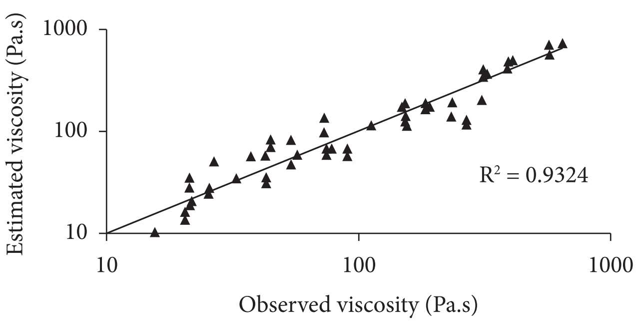

Figure 3. Modeled apparent viscosity versus observed viscosity.

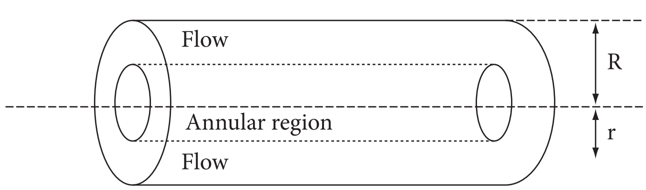

Figure 4. Poiseuille flow, R - radius; r - distance from the annular center to a given point. In the annular region, theoretically, there is no gradient of velocity between a given point and its adjacencies. In the flow region, such gradient of velocity is expected to occur.

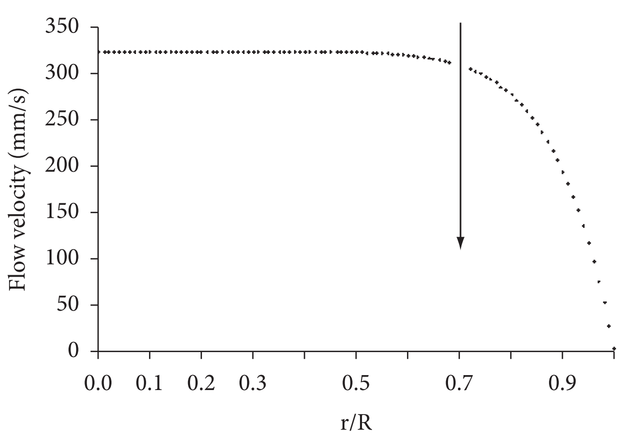

Figure 5. Flow velocity profile as a function of r/R ratio; arrow indicates the end of the constant flow velocity.

By analogy, the zero value of r/R in Figure 5 represents the center, and the 1 value represents the capillary wall. The maximum flow velocity was estimated as 323 mm/second at the capillary center. It can be observed that most of the mass is not subjected to a velocity gradient while flowing. The marked region under the curve in Figure 5, between 0.7 and 1 r/R, represents the portion of mass under shear while flowing, and its volume is estimated as 50% of the mass, approximately. It is worth to note that such volume depends on the flow behavior index *n* only, as implied by Equation 9, regardless of the average flow velocity.

4 Conclusions

Ground corn and soy grain exhibit a pseudoplastic behavior under viscometric flow, represented by the Power Law model.

The velocity profile of such mixture under viscometric flow indicates that a large portion is not submitted to a velocity gradient.

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