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Rheological Characterization of Liver Paste with a New Capillary Rheometer Based on Direct Pressure Measurements in the Capillary

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ABSTRACT

The present article describes the application of a new capillary rheometer that allows direct measurements of pressure in the capillary[1]. Commercial canned Norwegian liver paste was used as a food material for testing the new instrument which is made as a fixture in a Lloyd LR50K Plus texture analyzer.

The tested rheometer seems to represent a new and feasible objective off-line method for texture characterization of liver paste (Herschel-Bulkley fluid). Further testing and optimization of the equipment is continuously going on.

INTRODUCTION

Rheology has become an increasingly important tool for food science as well as the food industry. It is used for a wide range of purposes, ranging from routine analysis in industry to more complex investigations of e.g. macromolecular interactions[2].

Food however, is often a complex material structurally. In many cases it consists of mixtures of solids as well as fluid structural components. There are several tests for rheological characterization of food. It is also well accepted that no single test provides all the information necessary to obtain a complete rheological description. Therefore, a careful selection among various tests is always recommended. The selection of such tests will depend upon the type of food, the application and the availability of suitable instrumentation[3].

Since special challenges are associated with rheological methods examining temperature sensitive biological products like liver paste, this study focuses on measurements of viscosity and yield stress. We intended to investigate and validate the feasibility of the new rheometer combining viscosity measurements with yield stress information. Special attention was dedicated to the performance of reliable shear viscosity measurements, since the construction of the new rheometer allows the use of flush mounted pressure measurements in the capillary. It is important to ensure that the positions of the pressure sensors do not alter the flow conditions.

In this study we have investigated the feasibility of the new rheometer developed by ourselves, concerning rheology measurements on liver paste. Liver paste is an oil-in-water emulsion where the fat is the dispersed phase. This study reports from an ongoing test of the application of the new capillary rheometer on highly viscous food emulsions. The objectives of this work were as follows;

- Test the performance of the new capillary rheometer on food like liver paste.
- Test feasibility of the new instrument measuring a Herschel -Bulkley fluid.
- Rheological characterization of liver paste.
- Evaluate improvements of the new developed capillary rheometer.

MATERIALS AND METHODS

Liver paste samples

Commercial canned liver paste from the Norwegian food company Stabburet was used in all the experiments. This sort of liver paste is used in Norway as sandwich spread. It is an oil in water emulsion consisting of a mixture of liver from pork, lamb and bovine animals (29%). In addition the product contain fat from pork- and beef meat, water, carbohydrates, wheat flour, salt and minor components as antioxidants (E300) and preserving agents (E250). The product has to be stored dry and frost proof.

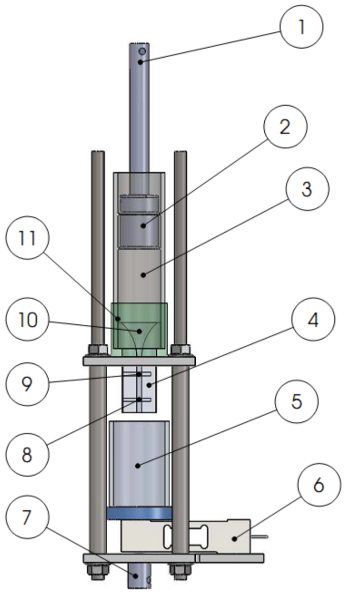

Figure 1. General description of the new capillary rheometer. Items are indicated by numbers: (1) RAM, (2) piston, (3) barrel, (4) capillary die, (5) feed collector, (6) load cell, (7) bottom connector, (8), (9) and (11) are the location of pressure measurements and (10) entry zone.

Table 1. Chemical composition of liver paste from "Stabburet"; g pr. 100 g product.

Constituent	g pr.100 g liver paste
Lipids	24.0
Protein	9.5
Carbohydrate	4.0
Salt	1.8

Instrumental analysis

The rheological measurements were all carried out on virgin (new sample for every measurement) liver paste samples at 22°C.

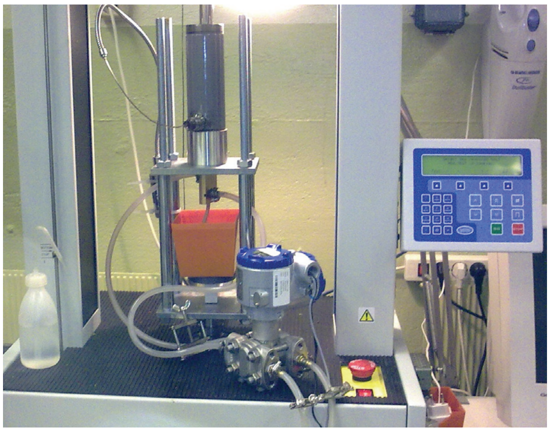

Figure 2. The capillary rheometer mounted in a Lloyd LR50K*Plus* 50KN texture analyser equipped with a differential pressure sensor (Fuji FCX-CII).

Experimental set-up

Figs. 1-4 illustrate the rheometer set-up. This set-up was used to test three teoretical equations (models) for volumetric flow rate in pipes given in literature for Herschel-Bulkley fluids according to the method decribed by Salas Bringas et. al[1]. A more detailed description of the system can be found in[1]. The pressure differential set up used the pin configuration described in literature[1].

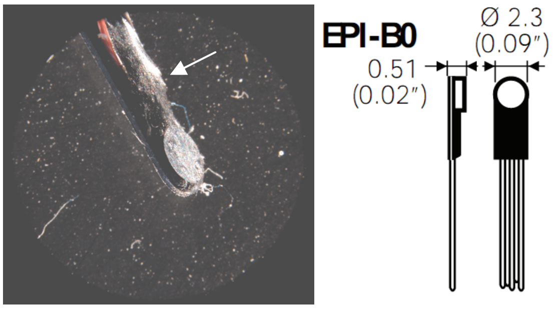

Figure 3. Subminiature pressure sensor (EPI-B0, Entran Ltd, Northants, Eng.); Dimensions in millimeters and (inches).

Part of the following nomenclature will be referred to Fig. 4.

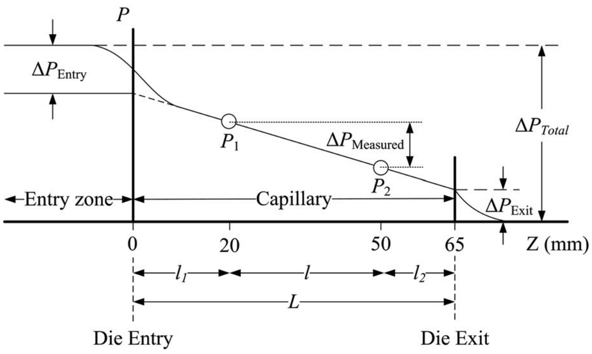

Figure 4. Sketch of pressure distribution in the capillary. *L,* length of capillary and *l*, distance between sensors in the capillary.

The three equations that are shown below for volumetric flow rate of a Herschel-Bulkley fluids in pipes, were used to estimate the consistency index (*K*) and the flow behavior index (*n*). Liver paste was used in all tests as an example of a real food emulsion. The equations are:

$$Q=\pi R^3 n\left(\frac{\tau_w}{K}\right)^{1/n}\left(1-\frac{\tau_0}{\tau_w}\right)^{n+1/n}\left[\frac{\left(1-\tau_0/\tau_w\right)^2}{3n+1}+\frac{2\left(\tau_0/\tau_w\right)\left(1-\tau_0/\tau_w\right)}{2n+1}+\frac{\left(\tau_0/\tau_w\right)^2}{n+1}\right] \qquad (1)$$

$$Q=\pi R^3 K^{-1/n}\left(\frac{R\Delta P}{2l}\right)^{-3}\left(\frac{R\Delta P}{2l}-\tau_0\right)^{(n+1)/n}\left[\left(\frac{R\Delta P}{2l}-\tau_0\right)^2\frac{n}{3n+1}+2\tau_0\left(\frac{R\Delta P}{2l}-\tau_0\right)\frac{n}{2n+1}+\tau_0^{2}\frac{n}{n+1}\right] \qquad (2)$$

$$Q=\left(\frac{\pi R^3}{256}\right)\left(\frac{4n}{3n+1}\right)\left(\frac{\tau_w}{K}\right)^{1/n}\left(1-\frac{\tau_0}{\tau_w}\right)^{1/n}\left[1-\frac{(\tau_0/\tau_w)}{2n+1}\left[1+\frac{2n}{n+1}\left(\frac{\tau_0}{\tau_w}\right)\left(1+\frac{n\tau_0}{\tau_w}\right)\right]\right] \qquad (3)$$

where Q is the volumetric flow rate (m^3/s), R the radius (m), n the flow behavior index (dimensionless), K the Consistency index (Pa s^n), τ_0 the yield stress (Pa), τ_w the shear stress at the capillary wall (Pa) and ΔP the pressure drop in the capillary (Pa). Eq. (1) was obtained from Chhabra & Richardson[4], Eq. (2) from Wildson[5] and Eq. (3) from Steffe[6].

RESULTS

Fig. 5 illustrates the results using different *L/D* ratios having no pressure measurements in the capillary.

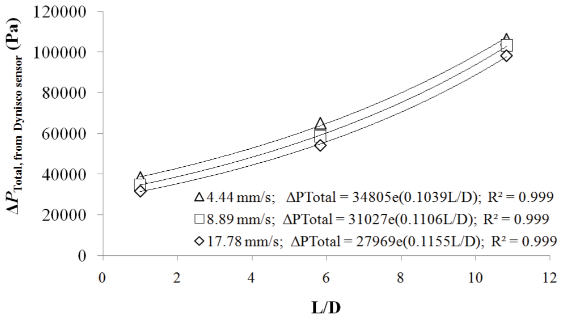

Figure 5. Measured pressures inside the barrel using Dynisco sensor for three different die speeds and three different *L/D* ratios. *L/D* = 1, *L/D* = 5.83 and *L/D* = 10.83, with a *D* = 0.006 m for all cases.

Figs. 6-11 show results with pressure measurements in the capillary.

The diameter of the plug formed in the center of a pipe when the shear stress (τ) is lower than the yield stress (τ_0), can be estimated by the following equation:

$$D_p=\frac{2\tau_0 R}{\tau_w} \qquad (4)$$

where D_p is the diameter of the plug.

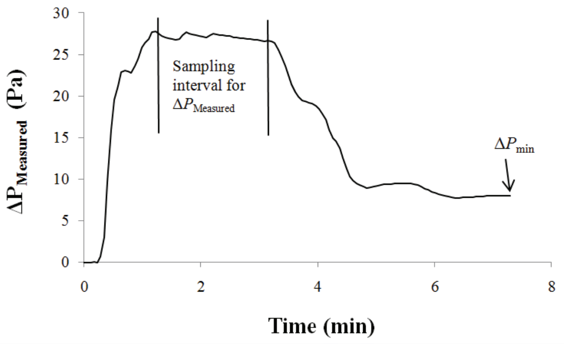

Figure 6. Raw data of the differential pressure obtained from the capillary measurements. The figure indicates the sampling interval used to estimate an average of $\Delta P_{\text{Measured}}$ and from where $\Delta P_{\min}$ is taken to calculate τ_0.

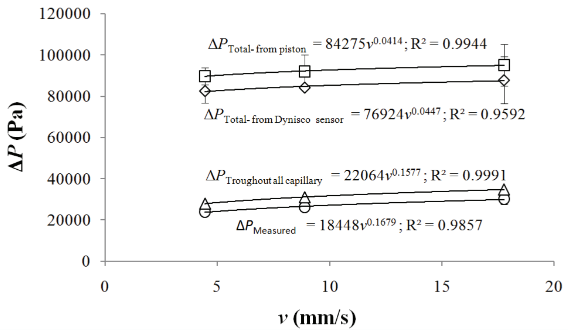

Figure 7. Average pressures (Pa) at three different die speed (mm/s). Averages come from two experiments at each speed. Error bars indicate the location of the two experimental values.

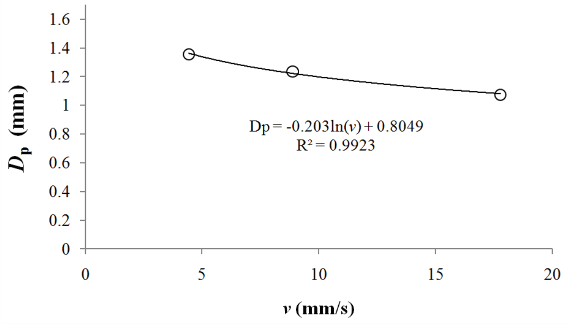

Figure 8. Diameter of the plug (mm) inside the capillary formed at different die speed (mm/s)

Fig. 9 shows an example of how the K and n values are obtained for Eq. (1). A full review of this method is given in Salas-Bringas et al[1]. A similar procedure is done for Eq. (2) and (3).

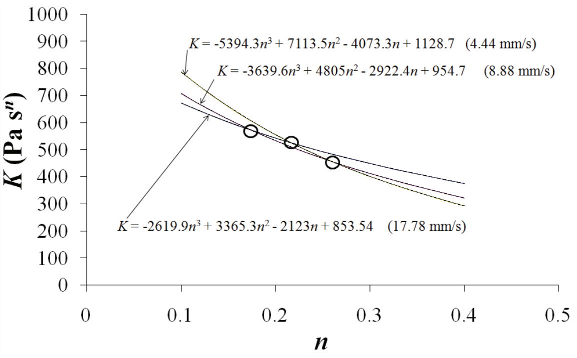

Figure 9. Plot that shows the intersections generated by Eq. (1) with data at three different die speeds.

The three intersections shown in Fig. 9 for Eq. (1) are presented as a single average in Fig. 10. Similar procedure is done for Eq. (2) and (3).

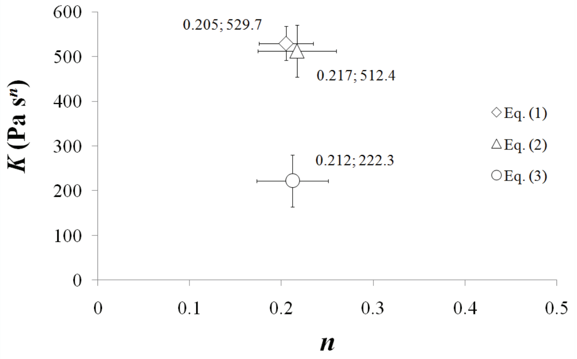

Figure 10. Average K and n values for the three eqautions. The error bars represent the standard deviation, number of samples 3.

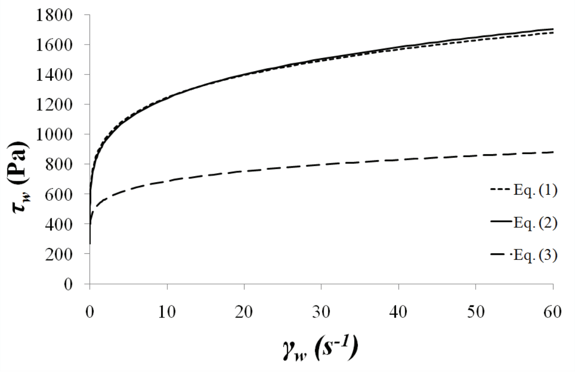

Figure 11. Herschel-Bulkley curves from the three equations.

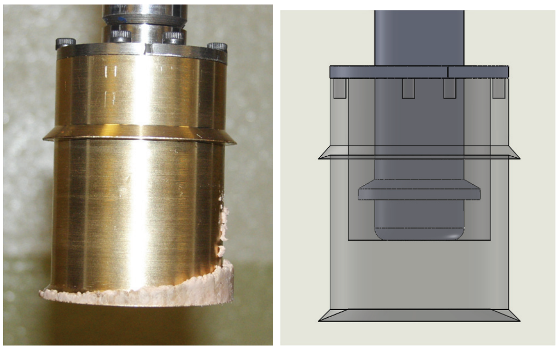

Figure 12. Accumulated material in the piston after one run (9 cm displacement). The picture shows the largest accumulation of material found.

DISCUSSION

Regarding the subminiature pressure sensors showed in Fig. 3, these sensors gave a very unstable signal and it was not possible to record their data (P_1 and P_2) simultaneously.

The arrow in Fig. 3 shows the sensor's connecting housing which was not flat as it is indicated in the drawing of the right side from supplier. This non-uniformity made the sensor installation very difficult to perform.

Fig. 5 is used to estimate the pressure drop through the entire capillary ($\Delta P_{\text{Throughout the capillary}}$) by projecting the curves made at different speeds to a *L/D* = 0. In Fig. 7 $\Delta P_{\text{Throughout the capillary}}$ is plotted in order to compare these values with the ones obtained using pressure measurements in the capillary ($\Delta P_{\text{Measured}}$).

When plotting $\Delta P_{\text{Throughout the capillary}}$ together with $\Delta P_{\text{Measured}}$ illustrated in Fig. 7, it is possible to observe that both curves present a similar exponent in the power law curve (0.1577 and 0.1679 respectively). This indicates that a good agreement can exist between the *n* values that could be obtained by using *L/D* method, and the ones obtained by direct pressure measurements in the capillary.

When comparing $\Delta P_{\text{Total- from Dynisco sensor}}$ using a die without pressure measurement in the capillary (*L/D* =10.83 in Fig. 5) with the ones with pressure measurements in the capillary ($\Delta P_{\text{Total- from Dynisco sensor}}$ in Fig. 7), it is possible to observe a small reduction in pressure when performing measurements in the capillary. It is likely that this pressure decrease is caused by a small amount of water from the pin system used to perform the pressure measurements in the capillary. This can be solved by changing the pin diameter or by changing this system by a membrane. Further improvements will be taken. Detailed view of this arrangement can be found in Salas-Bringas et. al[1].

From Fig. 10 it is possible to observe that all equations give relatively similar *n* values. Unfortunately this is not the case for the *K* value from Eq. (3) that differs greately from the other two equations. This can also be observed clearly when plotting the Herschel-Bulkley equations based on the three equations (see Fig. 11). The constant 256 on Eq. (3) is causing this disagreement.

Using *K* and *n* values given by Eq. (3) at 23.7 s^{-1}, resulted in a $\Delta P_{P1\text{-}P2}$ of 14433.1 Pa (using $\Delta P = \tau_w \, 2 \, l / R$) that is below any registered value ($\Delta P_{\text{Measured}}$, on Fig. 7). 23.7 s^{-1} is the shear rate at the wall for a Newtonian fluid at 17.78 mm/s in the capillary.

Since the *K* value for Eq. (3) differs so largely from the other two equations, and also due to the low values of $\Delta P_{P1\text{-}P2}$ in the back calculation, we believe that Eq. (3) underestimates the *K* values.

Fig. 12 demonstrates that the use of pressure from the piston can slightly overestimate the pressure in the barrel. When comparing the pressure measured by the sensor ((item 11) in Fig. 1) and the estimated pressure from the piston for all tests, it was found an average pressure difference of 12.14 x 10^3 Pa. This is a much smaller difference to the ones described in literature (290 x 10^3 Pa) when using a cylindrical piston without sharp flights on cement based materials[7].

CONCLUSIONS

The conclusions from this study can be summarized as follows,

- The new capillary rheometer was able to characterize a food emulsion like liver paste according to a Herschel-Bulkley fluid.
- The rheological characterization of liver paste with the new rheometer was performed by direct measurements of pressure in the capillary.
- The new capillary rheometer avoids the need of entry- and exit corrections.
- Installed subminiature pressure sensors gave a very unstable signal due to the non-uniformity of the sensors caused by inaccuracy during production. This made the sensor installation very difficult to perform. Further developments using different sensors set-up are continuously going on.

ACKNOWLEDGMENTS

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