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## 1 INTRODUCTION

A method for measuring the viscosity of cocoa and chocolate products (e.g. chocolate and coating masses, glazing mass, white chocolate) was developed in 1973. This method, published by the IOCCC (http://www.candy.net.au/IOCCC) has been revised in 2000 in order to provide reproducible results when comparing the measurements of different laboratories. The updated method ("Analytical Method 46") can be ordered at CAOBISCO (http://www.caobisco.be).

The method defines the rheometrical measurement procedure, i.e. for example the type of viscometer to be used, the preparation of the samples, the temperature and the shear rate or shear stress to be applied during the measurement. Additionally, the method describes how to correct the obtained apparent shear rate (Chapter 2) and provides a model function for fitting the flow curve (Chapter 3). A computer program "RheoCorr" was developed at the Laboratory of Food Process Engineering at the Swiss Federal Institute of Technology (http://www.vt.ilw.agrl.ethc.ch/rheoiccc) allowing to carry out these calculations (Chapter 4).

## **2** SHEAR RATE CORRECTION

In the concentric cylinder gap, the shear stress  $\tau$  is independent of the rheological properties of the measured fluid but it depends on the radius *r*:

$$\tau(r) = \frac{M_d}{2\pi \cdot H \cdot r^2} \tag{1}$$

where  $M_d$  denotes the applied or measured torque and H is the height of the cylinder. For non-Newtonian fluids this dependency will lead to a "viscosity distribution" in the shear gap. Most of the commercial rheometers only allow to display the apparent shear rate  $\dot{\gamma}_a$  calculated from the angular velocity  $\omega$  at the rotating inner cylinder (radius  $R_i$ ). The apparent shear rate is defined as follows:

$$\dot{\gamma}_a = \frac{2 \cdot \omega}{1 - \delta^2} \tag{2}$$

where  $\delta = R_i / R_o$  is the ratio of the radius of the inner  $(R_i)$  and outer  $(R_o)$  cylinders.

The apparent shear rate corresponds to the real shear rate in case of a Newtonian fluid. In order to allow for a more accurate result in case of a non-Newtonian fluid the corrected shear rate can be calculated as described in the next paragraph. Shear rate correction is only required for non-Newtonian fluids in cylinder gaps where  $\delta < 0.95$ .

Based on a stepwise power law approximation of the function the corrected shear rate  $\dot{\gamma}_c$  can be calculated as follows:

$$\dot{\gamma}_{c,j} = \frac{2 \cdot \omega_j}{n_j \cdot \left(1 - \delta^{2/n_j}\right)} \tag{3}$$

where  $n_j$  denotes the exponents of the N (number of measured points) stepwise power law functions. This spline approximation can be used for all types of non-Newtonian fluids (i.e. shear thickening and shear thinning).

$$n_{j} = \frac{\log(\tau_{j+1}) - \log(\tau_{j-1})}{\log(\omega_{j+1}) - \log(\omega_{j-1})}$$
(4)

The first and the last value of  $n_j$  can be extrapolated as follows:

$$n_1 = \frac{n_2^2}{n_3}$$
(5)

and

$$n_N = \frac{n_{N-1}^2}{n_{N-2}}$$
(6)

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## **3 MODEL FUNCTION**

The following model function has been proposed by Windhab to fit the flow curve of chocolates:

$$\tau = \tau_{o}^{*} + \eta_{\infty}^{*} \cdot \dot{\gamma} + \left(\tau_{1}^{*} - \tau_{o}^{*}\right) \cdot \left[1 - \exp\left\{-\frac{\dot{\gamma}}{\dot{\gamma}^{*}}\right\}\right]$$
(7)

 $\tau^*_{0}$  denotes the yield value which characterizes the "state of rest structure":

$$\tau_{o}^{*} = \tau_{1}^{*} - \left\{ \begin{bmatrix} \left( \frac{\tau_{2} - \tau_{1}}{\dot{\gamma}_{2} - \dot{\gamma}_{1}} \right)^{2} \\ \frac{\tau_{3} - \tau_{2}}{\dot{\gamma}_{3} - \dot{\gamma}_{2}} \end{bmatrix} \cdot \dot{\gamma}_{1} \right\}$$
(8)

 $\tau_1^*$  denotes the shear stress which leads to a maximum shear induced structuring:

$$\tau_{1}^{*} = \frac{\tau_{N} - \tau_{N-1}}{(\dot{\gamma}_{N} - \dot{\gamma}_{N-1}) \cdot \dot{\gamma}_{N}}$$
(9)

 $\eta^*_{\infty}$  is the gradient of the flow curve which is constant for most chocolates at  $\dot{\gamma} \ge$  60-100 s<sup>-1</sup>:

$$\eta_{\infty}^{*} = \frac{\tau_{N} - \tau_{N-1}}{\dot{\gamma}_{N} - \dot{\gamma}_{N-1}} \tag{10}$$

## One-parameter approximation model

A regression technique can be used to calculate  $\dot{\gamma}$  \*. In the computer program "RheoCorr" this feature is implemented as so called "best fit" approximation.

#### Zero-parameter approximation model Alternatively $\dot{\gamma}^*$ can be approximated as follows:

$$\dot{\gamma}^* = 2 \cdot \dot{\gamma} (\tau^*)$$
(11)

where

$$\tau^{*} = \tau_{o}^{*} + \left(\tau_{1}^{*} - \tau_{o}^{*}\right) \cdot \left(1 - \frac{1}{e}\right)$$
(12)

 $\tau^*$  denotes the shear stress where about 63%  $\approx$  1-1/e of the shear induced structure has been built up.

) 🖯 🖯		RheoCorr Document				
• F	ile Settings					
		Input Data Output Data		Output Graph	Fit Parameters	
###	A		В	С	D	E
1	Meas. Pts.	Shear Ra	te She	ar Stress	Viscosity	Temperature
2	{-}	[1/s]	[Pa	L	[Pa·s]	[°C]
3	1	50	85.	5	1.71	40
4	2	47.5	82		1.73	40
5	3	44.9	78.	4	1.74	40
6	4	42.4	74.	8	1.76	40
7	5	39.9	71.	3	1.79	40
8	6	37.4	67.	7	1.81	40
9	7	34.8	64.	2	1.84	40
10	8	32.3	60.	6	1.88	40
11	9	29.8	57		1.91	40
12	10	27.3	53.	5	1.96	40
13	11	24.7	49.	9	2.02	40
14	12	22.2	46.	2	2.08	40
15	13	19.7	42.	6	2.16	40
16	14	17.2	38.	9	2.27	40
17	15	14.6	35.	1	2.4	40
18	16	12.1	31.	3	2.59	40
19	17	9.58	27.	4	2.86	40
20	18	7.05	23.	3	3.31	40
21	19	4.53	19		4.2	40
22	20	2	14.	1	7.04	40

Figure 1: Input data spreadsheet.

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Figure 2 (above): Settings dialog.

#### Figure 3 (below): Corrected flow curve.

Kneecon settings	Knebco	RheoCorr Settings		
Input Data Output Data Output Graph Save Info	Input Data Output Data	Output Graph Save Info		
Geometry Data	X Axis:	Y Axis:		
Radius of Inner Cylinder: 13.0 mm	Logarithmic	€ Logarithmic ◯ Linear		
Radius of Outer Cylinder: 15.0 mm	🔘 Linear			
Height of Inner Cylinder: 45.0 mm	Shear Rate	<ul> <li>Shear Rate</li> <li>Shear Stress</li> <li>Viscosity</li> </ul>		
Shear Stress / Torque	Shear Stress Viscosity			
• Use Shear Stress Use Torque Column: C				
Shear Rate / Angular Velocity				
● Use Shear Rate O Use Angular Velocity Column: B	Approximation:			
First/Last Data Row	Show Approximation Curve Show "Best Fit" Approximation Curve			
First Data Row: 3 + Last Data Row: 22 +				

# 4 COMPUTER PROGRAM

"RheoCorr" is a Java application running on all major computer platforms (Windows, MacOS, Linux) given that the Java Runtime Environment (JRE 1.1.8 or higher) is installed. "RheoCorr" is freeware and can be downloaded on the internet at http://www.vt.ilw.agrl.ethc.ch/rheoiccc.

"RheoCorr" allows to open the raw rheometric data which must have been saved as tabdelimited text in the rheometer program (Fig. 1).

After opening the data table, the user must select the columns to be used for the cal-

culations (i.e. either shear stress or torque and either shear rate or angular velocity). Furthermore the geometrical data of the used cylinder geometry has to be specified (Fig. 2).

The corrected data can be displayed, saved and printed. "RheoCorr" also generates a graphical representation of the data points. The user can choose which data to show and whether to use a linear or a logarithmic scale for this flow curve (Fig. 3).





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