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HOW TO DRAW A RHEOGRAM WITH A MFI-TYPE DEVICE?

MFI-type devices (M3350) are in most cases used for quality control to validate the stability of the melt index during production or to check the conformity of the ordered polymer before processing. However it may be often necessary to draw at least once rheogram of the polymer used to know its behavior and optimize the processing conditions (if necessary). Indeed, the rheogram represents the change in viscosity versus the shear rate. Depending on the process (rotational molding, calendering, extrusion, injection, etc...), it becomes easier to optimize the processing conditions.

A. CALCULATIONS

The rheogram is the curve of variation of the dynamic viscosity μ (in Pa.s) versus the shear rate γ (in s^{-1}). How to go from the MFI (in g/10 min) to the viscosity? Simply by using the equations governing the flow of fluids inside a capillary (also called die).

1) Calculation of the shear rate

The **shear rate γ** depends on the **volumic flow rate Q** (in $m^3.s^{-1}$) and the die radius r (in m).

$$\gamma = \frac{4 \times Q}{\pi \times r^3}$$

The volume flow rate Q can be determined by dividing the volume V extruded during the time t. To switch from the MFI to the volume, it is sufficient to know the hot density ρ of the melt polymer (it can be determined by dividing the MFI by the MVI).

$$V = \frac{600 \times MFI}{\rho}$$

Or :

$$Q = \frac{600 \times MFI}{\rho \times t}$$

Ultimately :

$$\boldsymbol{\gamma = \frac{2400 \times MFI}{\pi \times r^3 \times \rho \times t}}$$

2) Calcul of the viscosity

The **viscosity μ** is the **shear stress τ divided by the shear rate γ.**

The shear stress τ depends on the pressure P exerted at the die inlet, that is to say, the force F exerted by the weight (mass M) placed on the MFI, the radius R of the heating cylinder and the die length L.

$$P = \frac{F}{\pi \times R^2} = \frac{M \times g}{\pi \times R^2}$$

Hence :

$$\tau = \frac{P \times r}{2 \times L} = \frac{M \times g \times r}{\pi \times R^2 \times 2 \times L}$$

So the viscosity expressed in terms of MFI is written:

$$\boldsymbol{\mu = \frac{M \times g \times r^4 \times \rho \times t}{4800 \times R^2 \times L \times MFI}}$$

3) Rabinowitsch shear rate correction

The calculations presented above are for Newtonian fluids. Thermoplastics have (in the overwhelming majority of cases) a non-Newtonian behavior, which requires correcting the shear rate. A first realistic approach to the rheological behavior is the power law symbolized by the Oswald law:

$$\tau = k \times \gamma^n$$

With k a constant of the fluid and n the flow index (also called pseudoplasticity index). When the fluid is Newtonian, n is equal to 1. To determine n, we plotted ln τ = γ + ln ln n k, n being the slope and *ln k* the intercept. If n is not 1, it is necessary to correct the apparent shear rate γ_a using the following equation:

$$\gamma = \frac{(3n+1)}{4n} \times \gamma_a$$

The **true shear rate γ** is then used to calculate the **true viscosity μ.**

B. METHOD

Tests were performed with a MFI M3350 (R = 4,95 mm) at 190°C. The die dimensions are:

- r = 0,5 mm ;
- L = 25 mm.

The mass series (in kg) is the following:

2,16 – 5 – 6,89 – 10 – 12,5 – 14,75 – 17,25 – 19,5 – 22,25 – 24 – 27 – 28,9 – 31,7 – 33,5

For each mass used, five values of MFI were measured.

C. RESULTS

At first, stresses and apparent shear rates were calculated using data from the MFI tests.

The curve Ln τ = f(Ln γ_a) has been plotted in order to determine the pseudoplasticity index *n* and perform the Rabinowitsch correction (*Figure 1*).

One can then calculate the true viscosity and the true shear rate. The rheogram thus obtained (*Figure 2*) represents the rheological behavior of the polymer, its Newtonian viscosity μ_0 and the critical shear rate γ_c. The latter gives an indication of when the rheological behavior changes from Newtonian to shear thinning (pseudoplastic).

According to the rheogram, in the Newtonian zone, Ln $\mu_0 \approx 8.65$ so $\mu_0 \approx 5700$ Pa and the change in rheological behavior occurs towards Ln $\gamma_c = 0.8$ so $\gamma_c \approx 2.23\ s^{-1}$.

Beyond the tested polymer has a shear thinning behavior. The slope *n* is equal to -0.45 and the intercept *K* is equal to Ln 9. Rheological equation on this section of the curve being Oswald law-type, the variation of viscosity as a function of velocity gradient is thus written:

$$\mu = 8310 \times \gamma^{-0,45}$$

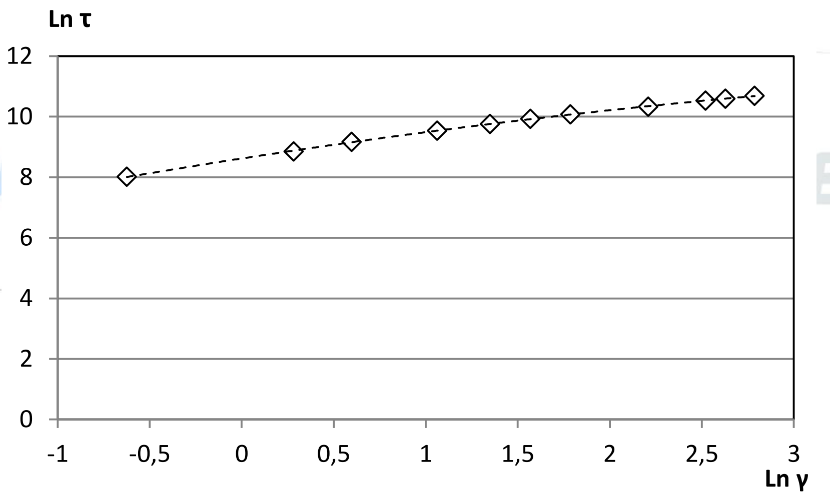

Figure 1: Ln τ = f(Ln γ) curve for the Rabinowitsch correction.

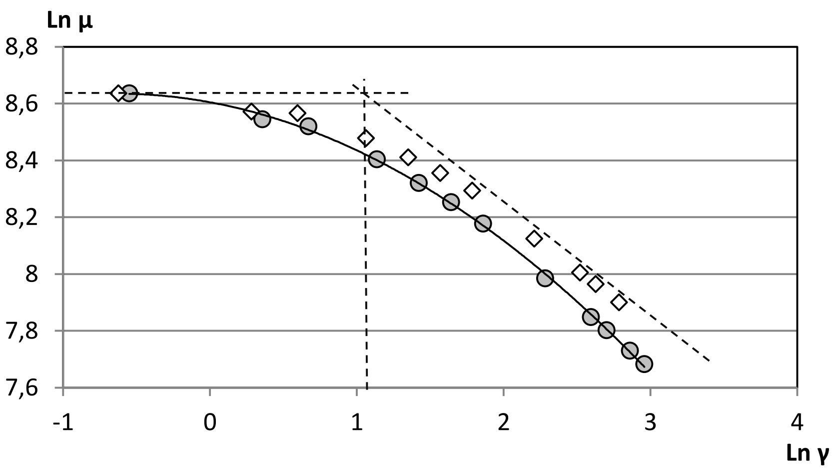

Figure 2: Apparent (white diamonds) & true (grey diamonds) rheograms.

D. CONCLUSION

As you can see, it is actually quite easy to draw a flow chart without having sophisticated rheometer. Here, with an MFI and a set of 4 different masses, we were able to vary the shear stress (we also could play on the dimensions of the die that have a huge influence on the shear stress). Then applying simple rheological equations, we were able to trace the rheogram of the polymer and thus know its rheological behavior at 190°C. The process optimization is much easier for the operator who may adjust its processing conditions in order to reduce costs (reduced cycle time) and/or improve the quality of its production (waste reduction, improved appearance, etc..).

In the second article devoted to the study of the behavior of plastic rheology with an MFI, we study the influence of the temperature on viscosity.

Suggested bibliographic reference

[1] A. V. Shenoy, S. Chattopadhyay & V. M. Nadkarni, *From melt flow index to rheogram,* Rheol. Acta 22 (1983) 90-101

[2] ISO 1133 : Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics.

[3] ASTM D1238: Standard Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer

[4] ASTM D3364: Standard Test Method for Flow Rates for Poly(Vinyl Chloride) with Molecular Structural Implications

[Autres] BS 2782, JIS K7210, etc…