STUDIA UBB. CHEMIA, LV, 3, 2010

THE TESTING AND CALIBRATION OF A ROTATING RHEOMETER

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ABSTRACT. The testing and calibration of the rotating viscometer were done by direct measurement of some standard liquids viscosity - aqueous glycerol or ethylene glycol solutions- and comparison with data measured on a standard viscometer (Rheotest). Formulas are presented for physical data that define the liquids rheological behavior: shear rate $\dot{\gamma}$, shear stress τ and viscosity η, particularized for this rheometer. Also, it was established the correction factor for the rotor end effect.

Keywords: *gap, rotating viscometer, shear rate, shear stress, viscosity*

INTRODUCTION

The rheometer (modified Couette type) subject to testing and calibration, consists of two coaxial glass cylinders. The inner cylinder is rotated and the fluid of whose rheological behavior is to be studied is placed in the ring-shaped space (gap) between the two cylinders. This will produce a certain torsional motion of the outer cylinder (Figure 1a, b and Figure 2) [1-4].

The functioning of the device is based on the following scheme (Figure 1b):

- the inner cylinder is rotated by means of an external drive with rotation speed n and angular velocity Ω, respectively;
- the outer cylinder, fixed to an elastic joint through a rubber muff, can be rotated by driving the liquid placed in the gap with a central angle $\Delta\theta$;
- the correlation between the shear stress τ and the torsion moment M, is done by using a calibration chart, $\Delta\theta = f(M)$;
- the prescribed variable is angular velocity Ω or revolution n;
- the measured variable is central angle $\Delta\theta$ proportional to shear stress τ.

In the calculations, instead of angular velocity Ω revolution n is used and central angle $\Delta\theta$ is expressed by the corresponding circle arc.

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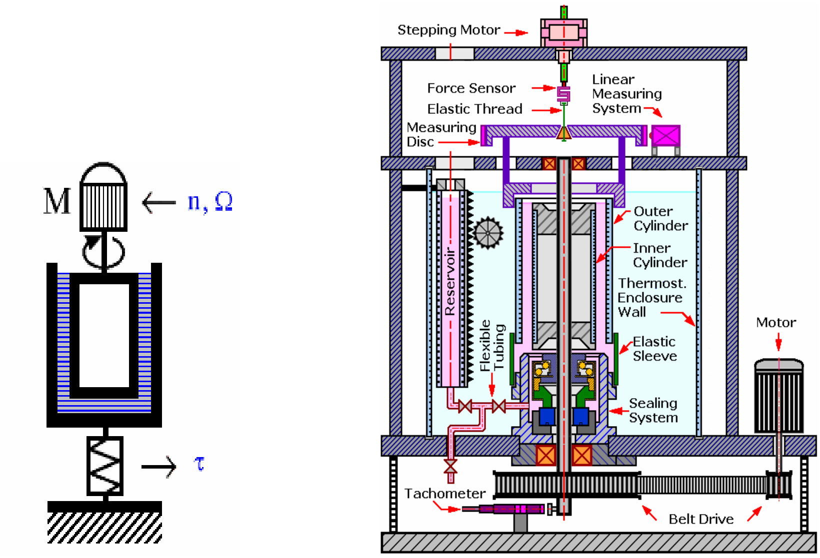

Figure 1 a,b. The Couette modified rheometer.

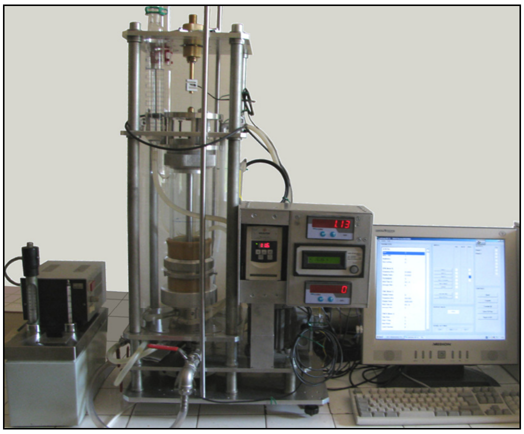

Figure 2. The achieved and tested experimental device.

RESULTS AND DISCUSSION

The calibration of the rheometer was done by comparing the rheometrical data obtained (measured or calculated) for aqueous glycerol solution (87%) or ethylene glycol with those measured for the same liquids, using a reference device (Rheotest-2), at different temperatures [5,6]. The rotation speeds at which measurements were made are within the range of $5 \div 50\ rpm$, which corresponds to the laminar flow regime of $0.86 \le Ta_{\mathrm{Re}} \le 8.58$ [4].

The main constructive sizes of the rheometer's cylinders: height $H = 290\ mm$; inner radius $r_i = 40\ mm$; outer radius $r_o = 42\ mm$; ratio of the radii $\delta = r_o / r_i = 1.05$; gap thickness $\Delta r = r_o - r_i = 2\ mm$ [4].

The measuring and calculation of some functional data are presented in Table 1.

Table 1. The measure and calculation route

Crt.No.	The measured/calculated data	The obtained method/relation
1	Revolution, n	Experimentally measured, $[\min^{-1}]$
2	Shear rate, $\dot{\gamma}$	$\dot{\gamma} = 2.1467 \cdot n$, $[s^{-1}]$
3	Torsion circle arc, α	Experimentally measured, $[div.]$
4	Central angle, $\Delta\theta$	$\Delta\theta = 0.028 \cdot \alpha$, $[rad]$
5	Moment, M	$M = 16.78 \cdot (\Delta\theta - 0.056)$, $[N \cdot m]$
6	Shear stress, τ	$\tau = 343.18 \cdot M$, $[Pa]$
7	Viscosity, η	$\eta = \frac{\tau}{\dot{\gamma}} = \frac{159.86 \cdot M}{n}$, $[Pa \cdot s]$

The values of these functional parameters for different measured rotation speeds are presented in Table 2.

Table 2. Functional data for Couette modified rheometer in the case of glycerol aqueous solution (87%), at 298K and different revolution values

Revolution n	Shear rate $\dot{\gamma} = 2.1467 \cdot n$	Cylinder torsion: Motion value, α	Cylinder torsion: Central angle, $\Delta\theta = 0.028 \cdot \alpha$	Torsion moment, M	Shear stress $\tau = 343.18 \cdot M$
$\min^{-1}$	s^{-1}	$div \times 10^2$	$rad \times 10^3$	$[N \cdot m] \times 10^3$	Pa
0	0	0	0	0	0
5	10.73	1.00	0.28	4.0	1.37
10	21.47	1.50	0.42	6.5	2.23
15	32.20	2.25	0.63	10.0	3.43
20	42.93	2.75	0.77	12.0	4.12
25	53.67	3.25	0.91	15.0	5.15
30	64.40	4.00	1.12	18.5	6.35
35	75.13	4.50	1.26	20.5	7.03
40	85.87	5.00	1.40	23.5	8.06
45	96.60	5.75	1.61	27.0	9.26
50	107.33	6.00	1.68	27.5	9.44

By graphically representing the relation of $\tau = f(\dot{\gamma})$ a linear variation is obtained, which is mathematically expressed by equation (1), its slope being the dynamic viscosity:

$$\tau = 0.0948 \cdot \dot{\gamma} + 0.0395\,; \quad r^2 = 0.9986 \qquad (1)$$

Thus, the dynamic viscosity obtained from the data measured with the modified Couette rheometer is 94.8 $mPa \cdot s$.

Also, through the rheological characterization of the same solution (standard) using the viscometer Rheotest-2, the relations were obtained: $\tau = f(\dot{\gamma})$ (as shown in Figure 3a) at different temperatures and $\ln\eta = f(1/T)$ (Figure 3b), respectively [6].

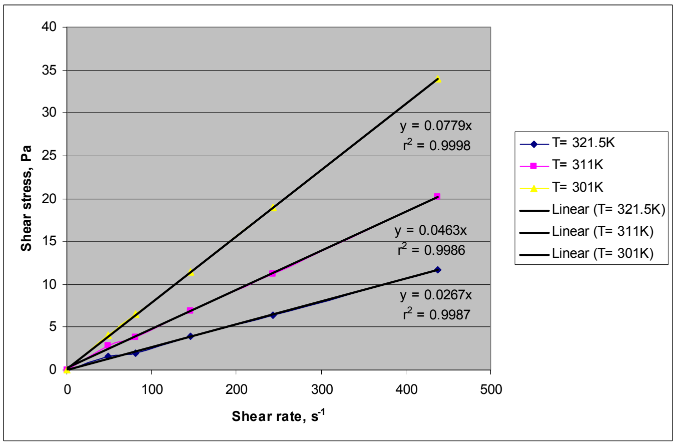

Figure 3a. $\tau = f(\dot{\gamma})$ for standard glycerol solution at different temperatures.

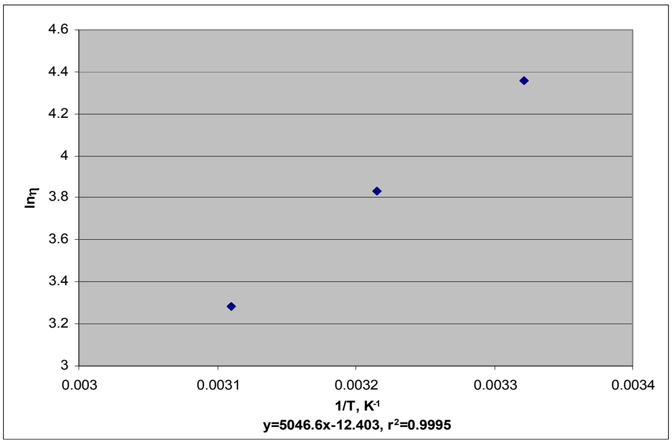

Figure 3b. $\ln\eta = f(1/T)$ for standard glycerol solution.

These relations allowed to establish rheological relations for different temperatures, as well as the general relationship between viscosity and temperature (Arrhenius type relation) [4,6]:

$$\eta = A \cdot e^{E_a/R \cdot T} = 4.106 \cdot 10^{-6} \cdot e^{5046/T} \tag{2a}$$

Particularizing this for glycerol at operating temperature (T=298K), the value was obtained of standard solution dynamic viscosity $\eta_R = 92.93\ mPa \cdot s$ and the activation energy of viscous flow $E_a = 41.94\ kJ \cdot mol^{-1}$.

Similar results were obtained also for ethylene glycol (activation energy $E_a = 25.35\ kJ \cdot mol^{-1}$).

$$\eta = A \cdot e^{E_a/R \cdot T} = 4.98 \cdot 10^{-4} \cdot e^{3050/T} \tag{2b}$$

The calculations did not take into account the additional effect introduced by the rotor rheometer terminal part (end effect). By correlating the values obtained experimentally with this device η_C, and the reference value η_R (standard), the correction coefficient was defined for the end effect:

$$CI = \frac{\eta_C}{\eta_R} = \frac{94.8}{92.93} = 1.02 \tag{3}$$

The correction of viscosity calculated value η_C is done by multiplying it by the factor $f = 1/CI$.

CONCLUSIONS

A modified Couette-type rotating viscometer was made in which the measurement part consists of glass cylinders. It gives the advantage of viewing the hydraulic phenomena that occur, especially in the field of transitional flow regime;

The methodology for measurement (revolution, torsion angle) was established and verified, as well as for the calculation of functional parameters (torsion moment, shear rate, shear stress) so that the rheological characterization and calculation of liquids viscosity can be realized;

Comparing the obtained viscosity value with the standard liquids viscosity, the end effect and the adequate correction factor were calculated. This coefficient is not significantly different from 1.

EXPERIMENTAL SECTION

The torsion of the measuring disk (Figure 1b) by which the central angle $\Delta\theta$ is assessed, was experimentally determined by calibration [6]. To a complete rotation, ($2 \cdot \pi$, rad.) 224.41 divisions correspond, and to a division correspond 0.028 rad., respectively.

The measurement of the dependence between central angle $\Delta\theta$ and the torsion moment M was experimentally done using a torsion balance. The torsion force was applied to the upper side of the outer cylinder on the measuring disk, the force arm being $b = 72.5\ mm$. The calibration chart was made for the muff thickness $1.5\ mm$, at room temperature (298K) and is plotted in Figure 4a, b. For work domain, the dependence is practically linear, with a slope of $0.06\ rad/N\cdot m$ (Figure 4a), but for the start domain ($M < 50\cdot 10^{-3}\ N\cdot m$) it shows a slight hysteresis phenomenon (Figure 4b).

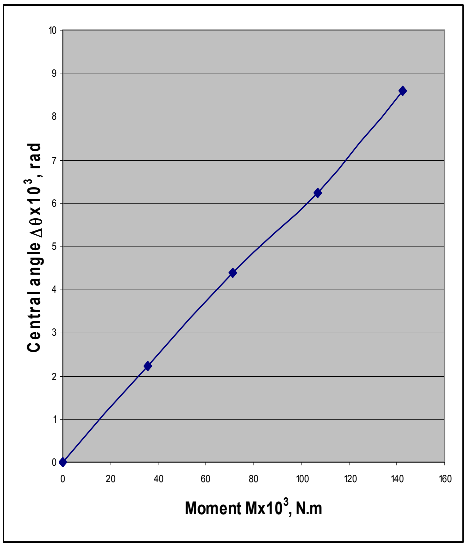

Figure 4a. Calibration chart Δθ = f (M).

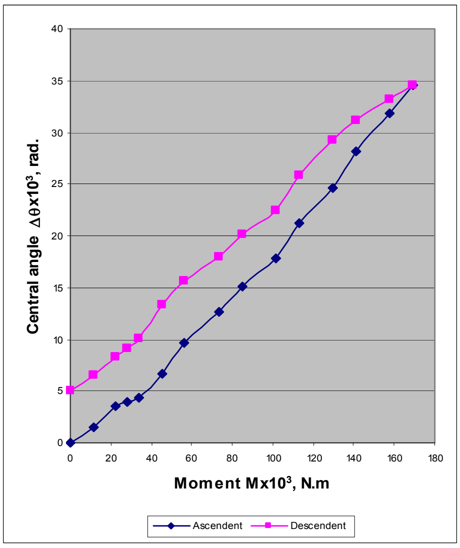

Figure 4b. Hysteresis curve Δθ = f (M).

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