

A CORRECTION METHOD OF THE WALL-SLIP EFFECT IN A CONE-PLATE RHEOMETER

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The flow of the investigated fluid in a measuring system of a rheometer – a capillary or a slit between rotating parts – may be disturbed by anisotropic behavior of the fluid near the wall. This phenomenon, so-called wall slip, often takes place in concentrated suspensions and solutions of linear polymers and introduces experimental errors to measurement results. There are methods of correction of these errors in the case of capillary and coaxial cylinders measuring systems. In the cone and plate system the correction seems to be more difficult because the width of the gap between cone and plate changes along the radius and thus the influence of the wall slip on the shear stress varies along the radius in an unpredictable and complicated manner. This dependency of the shear stress on the distance from the axis underlies the presented method of correction of experimental results obtained in the cone and plate system. The method requires several series of measurements of shear stress vs. shear rate performed using one measuring set, at various degrees of filling the gap.

Keywords: rheometry, wall-slip, cone-plate rheometer

1. INTRODUCTION

During the flow of a fluid in the capillary or gap of a rheometer its wall gives the fluid a preferred direction which makes it anisotropic near the wall. For example, in the solution of a linear polymer the orientation of linear particles close enough to the wall cannot be arbitrary – it is limited by the orientation of the wall. This can influence the fluid behavior, namely it can reduce its viscosity near the wall (Barnes, 1995; Joshi, 2001). In non-homogeneous fluids a thin layer of the continuous phase may form at the wall. As viscosity of this layer is much lower than that of the fluid in the main stream, the velocity gradient at the wall increases significantly. This leads to an effect as if the fluid were “lubricated” at the wall by another fluid of a much lower viscosity, or as if it slipped at the wall with a velocity called the slip velocity (Ahuja and Singh, 2009; Ballesta et al., 2008; Barnes, 1995; Becu et al., 2005; Bertola et al., 2003; Caballero-Hernandez, 2017; Chen et al., 2009; Cloitre and Bonnecaze, 2017; Franco et al., 1998; Gulmus and Yilmazer, 2005; Isa et al., 2007; Kalyon, 2005; Meeker et al., 2004; Paredes et al., 2015; Soltani and Yilmazer, 1998). For this reason the effect is called the slip effect. It may exert a great influence on the results of rheometric measurements as it reduces the resistance to flow of the investigated fluid. The wall effect changes properties of the fluid and therefore influences results of the measurements and introduces experimental errors which are the higher the narrower the measuring gap or capillary of the rheometer. This underlies the methods of correction based on a comparison of experimental results obtained in two or more measuring systems of different dimensions in which wall effects disturb the flow to a different extent.

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The correction methods of the wall effect were first described by Mooney (1931) for a capillary and coaxial cylinder rheometer. They need the application of two capillaries of different diameters. Another method was proposed by Yeov et al. (2003). For a coaxial cylinder rheometer the Mooney (1931) method as well as its modification by Schlegel (1980) needs evaluation of three flow curves using three measuring sets of precisely adjusted diameters. The author (Kiljański, 1989) developed a method which required only two measuring sets of different width of the gap between the cylinders of any inner and outer diameters.

In both above mentioned measuring geometries – capillary and coaxial cylinder – the width of the narrow space which contains the fluid – a capillary or gap – is constant. For this reason, flow conditions, although influenced by the wall effect, are constant on the whole surface of the capillary or cylinder. So, shear stress and shear rate are also constant on the whole surface. This allows us to correct results.

However, in the most popular measuring system, the cone and plate, the situation is different (Fig. 1). The width of the gap varies in a wide range from the axis to the edge. So, the influence of the slip effect on the flow, the ‘lubrication’ effect, is different at each distance from the axis. The further from the axis the lower the lubrication effect and thus – the higher the shear stress. Thus, the flow conditions at the cone surface are different in each point. If the stress changes along the radius the slip velocity also changes along the radius, which complicates even more the description of the flow. That is why the cone-plate system is claimed to be undistinguished for the fluids which exhibit the wall slip because results are impossible to correct. As it will be shown below, this is not true.

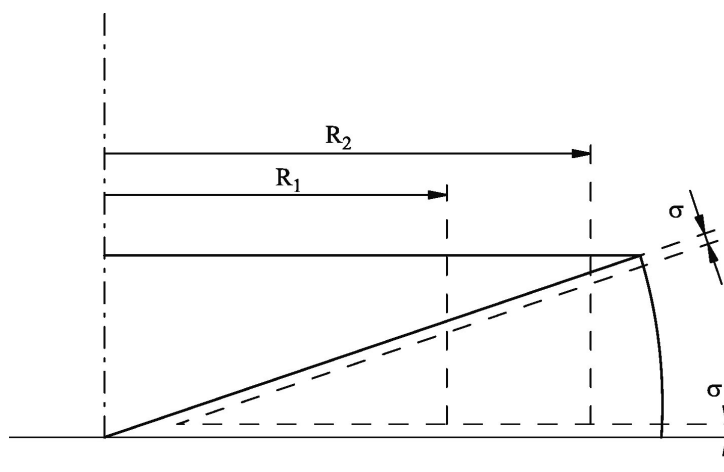


Fig. 1. Diagram of the gap between cone and plate filled with a liquid

2. DEVELOPMENT OF THE METHOD

Let us notice that in each axisymmetric part of the volume between cone and plate, of the radii, called R_1 and R_2 (Fig. 1), the gap width and thus the influence of the slip effects is different – like in two different gaps between the coaxial cylinders. What about performing two series of measurements using the same cone but at two different degrees of filling the gap? It is analogical to the two series of measurements using two different gaps between coaxial cylinders.

However, the main above-mentioned difficulty remains. In the measuring gap, both fully and partially filled, shear stress, due to the wall slip, changes along the radius in an unpredictable and complicated manner. The rheometer does not measure its local values. Here, mathematics will help us to calculate what we cannot measure. Let us start with the description of stress which changes along the radius. Between

shear stress at the cone surface $\tau(r)$ and torque M which is measured by the rheometer there is a simple correlation

$$M = \int_0^R 2\pi \cdot r^2 \cdot \tau(r) \cdot dr \tag{1}$$

where R is the radius of the zone filled with the fluid and $\tau(r)$ is the variable, unknown and unmeasurable stress inside this zone. The torque at a given angular speed is therefore a function of radius R . It is the so-called function of the upper integration limit which can be written in the general form as

$$M = f(R) \tag{2}$$

If measurements are performed at a constant angular speed but at variable R , we can obtain a particular form of Equation (2), from which derivative dM/dR can be calculated as a function of R . This derivative can be connected with stress at distance R from the axis by differentiation of Equation (1)

$$\frac{dM}{dR} = 2\pi \cdot R^2 \cdot \tau(R) \tag{3}$$

from which shear stress $\tau(R)$ for various R may be calculated. This must be repeated for other angular speeds to obtain for each speed function (2) and next the dependence of $\tau(R)$ on R .

Now, let us consider shear rate. The velocity of a given point at the cone surface may be expressed as (Fig. 2)

$$u = \frac{du}{dy} \cdot h + 2u_s \tag{4}$$

$$\frac{u}{h} = \frac{du}{dy} + \frac{2u_s}{h} \tag{5}$$

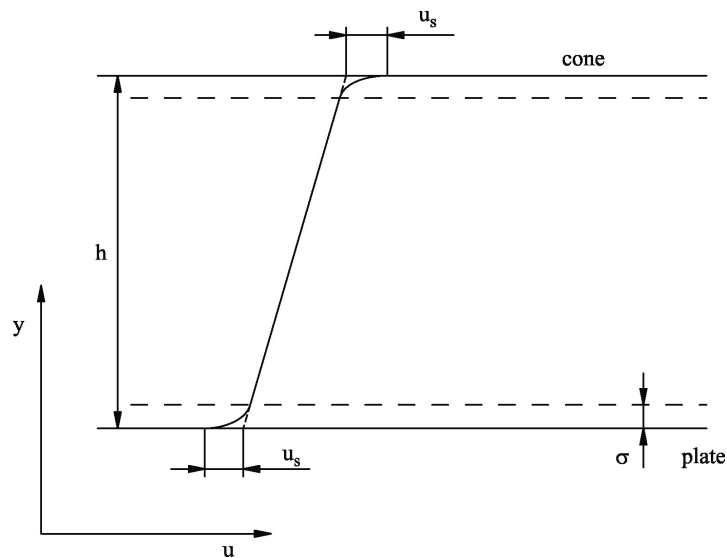


Fig. 2. Velocity distribution inside the gap. Dashed line – the limit of the slip zone

The left side of Eq. (5) is constant for a given measuring system rotating at a constant speed – it assumes the same value in each point of the cone surface and is given by the rheometer as the value of shear rate $\dot{\gamma}$.

$$u = r \cdot \omega = \frac{h}{\tan \alpha} \cdot \omega \tag{6}$$

$$\frac{u}{h} = \dot{\gamma} = \frac{\omega}{\tan \alpha} = const \tag{7}$$

The undisturbed shear rate du/dy outside the slip zone may be calculated from the set of two equations based on Eq. (5) written for two sets of data: angular speed + radius R of the zone filled with the fluid, for which shear stress at the distance R has the same value, Fig. 3. One set of data taken from Fig. 3 is: angular speed ω_1 and radius R_1 (and the gap width $h_1 = R_1 \tan \alpha$), the other is: angular speed ω_2 and radius R_2 (and the gap width h_2).

$$\dot{\gamma}_1 = \frac{du}{dy} + \frac{2u_s}{h_1} \quad (8a)$$

$$\dot{\gamma}_2 = \frac{du}{dy} + \frac{2u_s}{h_2} \quad (8b)$$

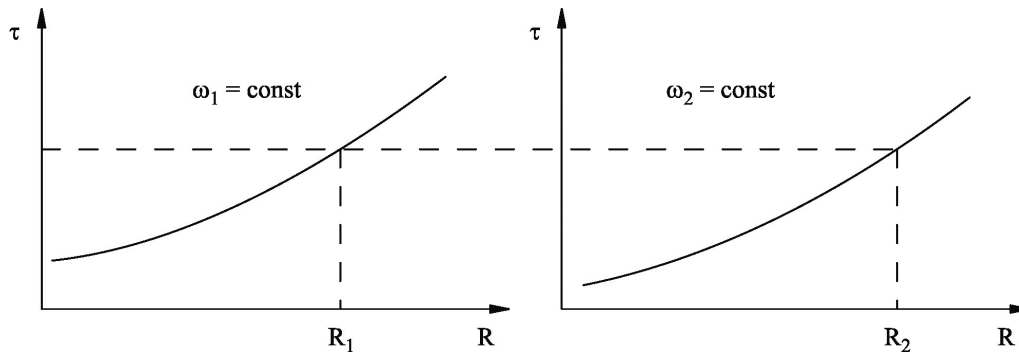


Fig. 3. Evaluation of R_1 and R_2 values for which the equation set (8) is written

Because both equations refer to the same value of shear stress, shear rate du/dy and slip velocity u_s assume the same values in each of these two equations. The values of $\dot{\gamma}_1$ and $\dot{\gamma}_2$ are the disturbed values of shear rate given by the rheometer at rotational speeds ω_1 and ω_2 , (see Eq. 7), the gap widths h_1 and h_2 are defined by R_1 and R_2 , respectively. The value of shear rate du/dy calculated from Eqs. (8) is the corrected value at shear stress for which the values of R_1 and R_2 were obtained from Fig. 3. Thus, we have one point on the corrected flow curve.

In this manner the main defect of the cone-plate system, i.e. variability of the slip effect with axial distance, was applied to correct results. Furthermore, we did not need two different measuring sets which were necessary in the case of capillaries and coaxial cylinders. One should be aware of an important fact – to be able to find in the results of measurements two different values of angular speed for which the same value of shear stress can be found (see Fig. 3), one should perform measurements at many angular speeds which do not differ much from the previous and the next one. The procedure is then as follows.

1. Perform measurements of the flow curve at various filling degrees of the gap, measuring each time the radius of the circular zone filled with the liquid. Besides the normal values measured by the rheometer, such as shear stress and shear rate (both influenced by the wall slip) record also the value of torque M and angular speed ω of the cone. If the rheometer does not give these values, it is easy to calculate them using the measured values of shear stress τ

$$M = \frac{2}{3} \pi \cdot R_c^3 \cdot \tau \quad (9)$$

and the shear rate $\dot{\gamma}$

$$\omega = \dot{\gamma} \cdot \tan \alpha \quad (10)$$

2. Choose a set of measurement data obtained at the same angular speed of the cone but at various gap filling.

3. Make a plot of the torque measured by the rheometer versus radius R of the zone filled with the liquid

$$M = f(R) \quad (11)$$

and determine the dependence of derivative dM/dR on R

4. From Eq. (3)

$$\frac{dM}{dR} = 2\pi \cdot R^2 \cdot \tau(R) \quad (12)$$

one can now calculate shear stress at the distance R from the axis $\tau(R)$ for any R value. Thus, we have the dependence of shear stress on axial distance R for the given speed of rotation of the cone.

5. Repeat the operations 2–4 for other investigated angular speeds, which give us the dependence of shear stress on axial distance R for each investigated angular speed.
6. Write the set of Eq. (8)

$$\dot{\gamma}_1 = \frac{du}{dy} + \frac{2u_s}{h_1} \quad (13a)$$

$$\dot{\gamma}_2 = \frac{du}{dy} + \frac{2u_s}{h_2} \quad (13b)$$

for a chosen value of shear stress obtained at two different angular speeds (Fig. 3). The value of the corrected shear rate du/dy which is the solution of the equations, together with the value of the stress for which this set of equations was formulated, gives us one point on the corrected flow curve.

7. Repeat point 6 for the succeeding values of shear stress, and thus obtain the corrected flow curve.

Of course, performing measurements with another cone of a different cone angle α will improve the accuracy of final results, but is not necessary.

3. SUMMARY

A method for correction of the wall-slip effect in a cone-plate rheometer was developed. It is based on the dependency of shear stress on the distance from the axis. The method requires several series of measurements performed in one measuring set, at various degrees of filling the gap.

SYMBOLS

h	local gap width, m
du/dy	undisturbed shear rate in the fluid outside the slip zones, s^{-1}
M	torque, Nm
r	distance from the axis, m
R	radius of the zone filled with the fluid, m
R_c	radius of the cone, m
u	velocity, m/s
u_s	slip velocity, m/s
α	the angle between the cone and the plate
$\dot{\gamma}$	the disturbed, incorrect value of shear rate, obtained as a result of the measurement performed using the rheometer
ω	angular speed of rotation, rad/s
σ	width of the slip zone, m
τ	shear stress, Pa

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Received 1 February 2018

Received in revised form 13 November 2018

Accepted 6 May 2019