

# RHEOMETRY OF CONCENTRATED CERAMIC SUSPENSIONS - STEPS FROM MEASURED TO RELEVANT DATA. PART 3: ROTATIONAL VISCOMETER WITH PARALLEL PLATES, POWER-LAW MODEL

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*The knowledge of flow behavior of ceramic suspensions is important condition for control of ceramic wet processing. The flow behavior can be stated by the rheological measurements. The aim of the measurements was to obtain the flow curve and to propose suitable and simple rheological model for the flow behavior description. The power-law is the simplest model mostly used for description of rheological behavior of non-Newtonian fluids. Using this model, the dependence of shear stress  $\tau$  on shear rate  $\dot{\gamma}$  can be expressed by the relation:  $\tau = K\dot{\gamma}^n$ , where  $K$  is the coefficient of consistency and  $n$  stands for the flow behavior index. The two arrangements of rotational viscometer method with parallel plates (discs) were used in rheological measurements for results checking. The procedure of experimental data evaluation from rotational viscometer with parallel plates to obtain parameters  $K$  and  $n$  is presented.*

## INTRODUCTION

Ceramics have been produced by wet processing for a long time. Many of traditional forming methods are used today including slip and tape casting method, filter pressing etc. The casting of ceramics done at room-temperature is a frequent operation for preparation of ceramics via net-shape forming processes. Ceramic particles suspended in a liquid are cast into porous mold that removes the liquid and leaves a particulate compact in the mold. There is a number of variations to this process, depending namely on the viscosity of the ceramic-liquid suspension and the procedure used. The principles and controls for slip casting are similar to those of the other particulate ceramics casting techniques. The clay-water system is the first colloidal system to be studied extensively. This system serves a basis for many traditional ceramic processes. Control of these processes is based on tailoring of ceramic suspensions flow behavior, i.e. on rheological properties of concentrated ceramic suspension. The rheology strongly controls the quality of the final product [1-7].

Rheological measurements monitor changes in flow behavior in response to applied stress (or strain) [5]. The critical parameters of interest include the shear stress-shear rate dependence. Various type of flow behavior can

be observed under steady shear depending on suspension composition and stability, as shown in Figure 1. Newtonian behavior is simplex flow response, where viscosity is independent of shear rate (see curve (1) in Figure 1). Pseudoplasticity of structurally viscous liquid behavior occurs when the viscosity decreases with shear rate (see curve (2)). This response can be accompanied by yield stress whose magnitude depends on the strength of the particle network (see curve (4) and (5)). If the flow curve is linear above yield stress, the system is referred to as Bingham plastic (curve (4)). Finally, dilatant liquids occur when the viscosity increases with shear rate (see curve (3)). The rheological properties of concentrated colloidal suspensions are often time dependent. Thixotropic (thixos = contacts, movement; trepo = change) systems exhibit an apparent viscosity that decreases with time under shear, but recovers to its original viscosity when flow ceases. The opposite behavior is referred to as rheopexy (rheoplectic liquids - anti-thixotropy) [2-4, 8].

As it was stated in [9-16] the rotational viscometer method with various sensors (coaxial cylinders, plate-plate, and cone-plate) is mostly used in rheological measurements. The aim of the measurement is to obtain the flow curve and to propose suitable and simple rheological model for the flow behavior description.

The power-law is the simplest model mostly used for description of rheological behavior of non-Newtonian fluids. Using this model, the dependence of shear stress  $\tau$  on shear rate  $\dot{\gamma}$  can be expressed by the following relation:

$$\tau = K \dot{\gamma}^n \quad (1)$$

where  $K$  is the coefficient of consistency and  $n$  stands for the flow behavior index.

In [13-17] the attention was focused to the rotational viscometer with coaxial cylinders. In this paper the rotational viscometer with parallel plates (discs) will be discussed. Its common configuration consists of an upper rotating disc with radius  $R$  in distance  $H$  from the lower stationary plate - see Figure 2. The values of Newtonian shear stress  $\tau_{RN}$  and shear rate  $\dot{\gamma}_R$  on radius  $R$  are usually obtained from rheological measurements. The aim of this paper is to show how the parameters of power-law model (coefficient of consistency  $K$  and the flow behavior index  $n$ ) can be obtained from measurements.

### THEORETICAL

When the shear flow between both discs occurs, the following relation holds for only non-zero tangential velocity component  $u_\varphi$

$$u_\varphi = r \Omega(z) \quad (2)$$

where  $\Omega(z)$  is angular velocity.

The  $\varphi$  component of the Cauchy's equation of motion for this case takes the form (see e.g. [18])

$$\frac{\partial \tau_{\varphi z}}{\partial z} = 0 \quad (3)$$

from which it follows that shear stress  $\tau_{\varphi z}$  and also shear rate

$$\dot{\gamma} = \frac{\partial u_\varphi}{\partial z} = r \frac{d\Omega}{dz} \quad (4)$$

do not depend on axial coordinate  $z$ . Integrating the equation

$$\frac{d\Omega}{dz} = C_1 \quad (5)$$

we receive

$$\Omega = C_1 z + C_2 \quad (6)$$

Using boundary condition

$$z = 0, \Omega = 0 \quad (7a)$$

$$z = H, \Omega = \omega \quad (7b)$$

we obtain

$$C_1 = \frac{\omega}{H}, \quad C_2 = 0 \quad (8a,b)$$

where  $\omega$  is angular velocity of upper rotating disc.

Substituting (8a) into (4) we obtain for shear rate the following relation

$$\dot{\gamma} = r \frac{d\Omega}{dz} = r C_1 = r \frac{\omega}{H} \quad (9)$$

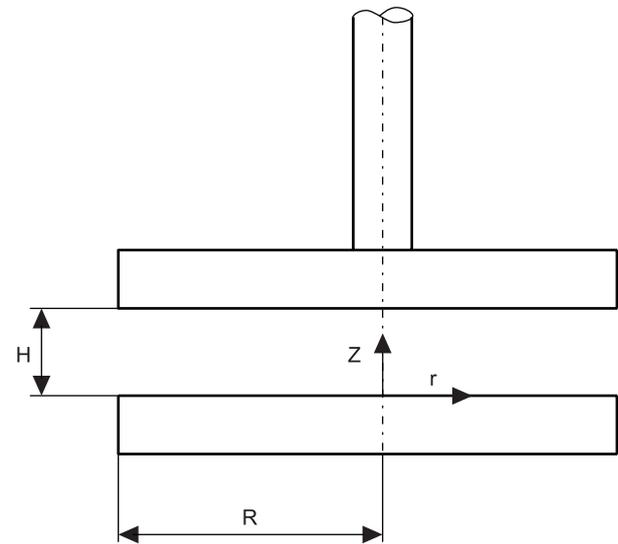
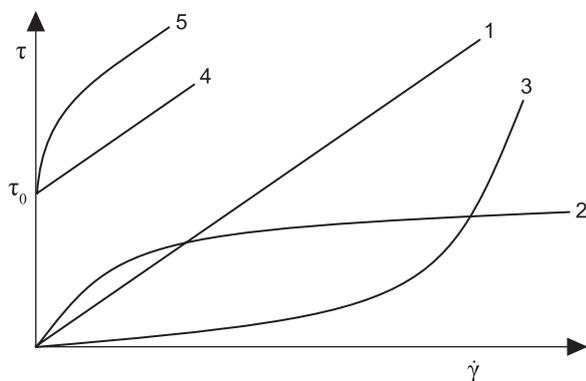
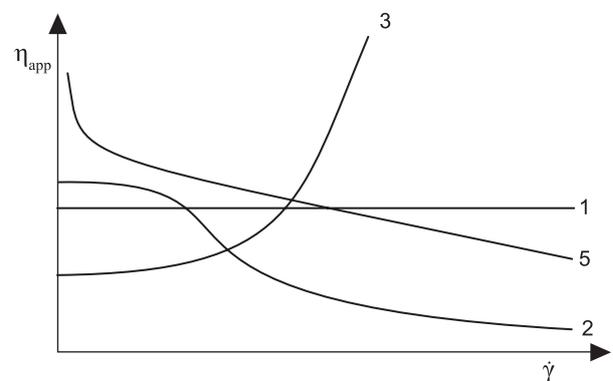


Figure 2. Configuration of parallel – plates discs.



a) stress - shear rate dependence:

- 1 - Newtonian behaviour; 2 - shear thinning behaviour; 3 - shear thickening behavior, 4 - Bingham dependence, 5 - Herschel-Bulkley dependence



b) viscosity dependence:

- 1 - Newtonian behavior; 2 - shear thinning behaviour 3 - shear thickening behaviour, 5 - Bingham dependence

Figure 1. Rheological behaviour of Newtonian and non-Newtonian suspensions.

The shear stress in viscometer is calculated from torque M that can be obtained from the following integral

$$M = 2\pi \int_0^R r^2 \tau_{\varphi z} dr = 2\pi K \left(\frac{\omega}{H}\right)^n \int_0^R r^{2+n} dr = \frac{2\pi}{3+n} K \left(\frac{\omega}{H}\right)^n R^{3+n} \quad (10)$$

where relation for shear stress  $\tau_{\varphi z}$  was obtained by substitution (9) into (1).

For Newtonian fluids with dynamic viscosity  $\mu$

$$\tau = \mu \dot{\gamma} \quad (11)$$

Equation (10) transforms to

$$M = 2\pi \int_0^R r^2 \tau_{\varphi z} dr = 2\pi \mu \left(\frac{\omega}{H}\right)^n \int_0^R r^{3+n} dr = \frac{\pi}{2} \mu \left(\frac{\omega}{H}\right)^n R^4 \quad (12)$$

For radius R the equation (9) takes the form

$$\dot{\gamma}_R = R \frac{\omega}{H} \quad (13)$$

and from equation (1) we obtain

$$\tau_R = K \dot{\gamma}_R^n = K \left(\frac{R\omega}{H}\right)^n \quad (14)$$

and from equation (11)

$$\tau_{RN} = \mu \dot{\gamma}_R = \mu \frac{R\omega}{H} \quad (15)$$

Using (14) in (10) we can write

$$M = \frac{2\pi}{3+n} \tau_R R^3 \quad (16)$$

and similarly using (15) in (12) we receive

$$M = \frac{\pi}{2} \tau_{RN} R^3 \quad (17)$$

Substituting (17) and (13) into (10) we get

$$\tau_{RN} = \frac{4}{3+n} K \dot{\gamma}_R^n = K_N \dot{\gamma}_R^n \quad (18)$$

Combining (16) and (17) we obtain the following relation

$$\tau_R = \frac{3+n}{4} \tau_{RN} \quad (19)$$

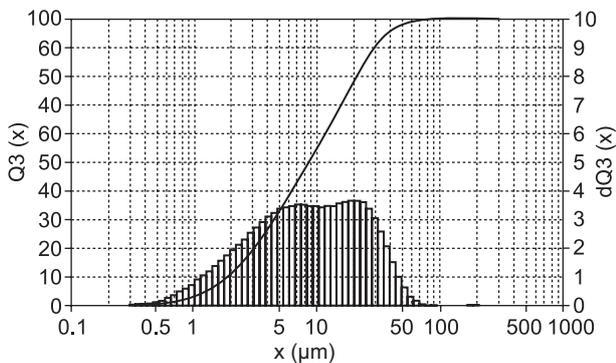


Figure 3. Particle size distribution of clay - materials for preparation of aqueous suspensions.

## EXPERIMENTAL

Water suspension of clay with mean volumetric concentration 30% of solid phase was used in rheological experiments. The particle size distribution of clay obtained from measurements with particle size analyzer A22 Compact (Fritsch) is shown in Figure 3. Mean particle diameter  $x_{50} = 8.4 \mu\text{m}$  was also obtained from this analysis.

The experiments were carried out using rheometer RV1 (Haake) by usage of two parallel plates with diameters 35 mm (PP35) and 60 mm (PP60), distance of plates was 1 mm.

## RESULTS

Dependence of  $\tau_{RN}$  on  $\dot{\gamma}_R$  measured on parallel plates PP35 is shown in Figure 4. From this figure it can be seen that dependence in logarithmic coordinate is a straight line and for this reason power-law can be used for data evaluation. From the figure it can be seen that  $K_N = 11.34 \text{ Pa}\cdot\text{s}^n$  and  $n = 0.262$ . The consistency coefficient K can be calculated from Equation (18)

$$K = K_N \frac{3+n}{4} = 11.34 \frac{3+0.262}{4} = 9.25 \text{ Pa}\cdot\text{s}^n \quad (20)$$

The corresponding results obtained on parallel plates PP60 are shown in Figure 5 and values  $K_N = 11.72 \text{ Pa}\cdot\text{s}^n$  and  $n = 0.258$  were obtained and  $K = 9.55 \text{ Pa}\cdot\text{s}^n$  was calculated.

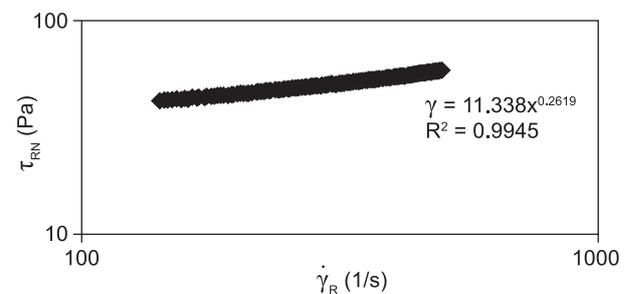


Figure 4. Dependence of  $\tau_{RN}$  on  $\dot{\gamma}_R$  measured on parallel plates PP35.

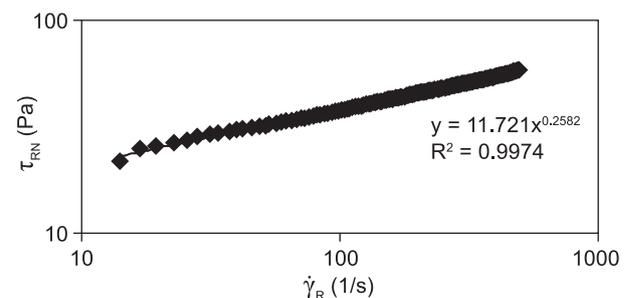


Figure 5. Dependence of  $\tau_{RN}$  on  $\dot{\gamma}_R$  measured on parallel plates PP35.

The values  $\tau_R$  must be calculated from Equation (19) and the value of  $\dot{\gamma}_1$  must be calculated from Equation (4) of [13] for comparison of results obtained with parallel plates and coaxial cylinders arrangements. From Figure 6 where the comparison is depicted, the relative good agreement of all results can be seen. On the basis of data from all sensors (parallel plates PP35 and PP60 and coaxial cylinders Z31 and Z41) the parameters of power-law describing the behaviour of measured suspension are shown in Figure 6.

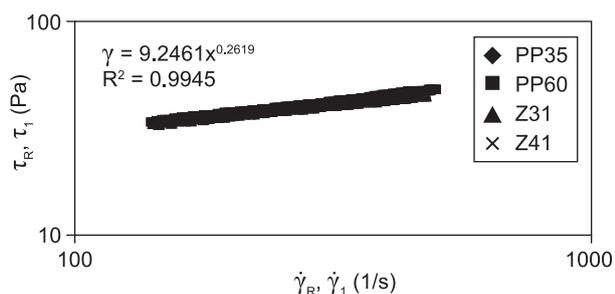


Figure 6. Comparison of corrected results from all sensors (parallel plates PP35 and PP60 and coaxial cylinders Z31 and Z41) with power-law parameters describing the behaviour of measured suspension.

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#### Symbols

$H$	distance of plates (discs)
$K$	consistency coefficient
$M$	torque
$n$	flow index
$r$	radial coordinate
$R$	radius of discs
$u$	velocity
$z$	axial coordinate
$\dot{\gamma}$	shear rate
$\varphi$	tangential coordinate
$\omega$	angular velocity of rotating plate

$\Omega$	local angular velocity
$\mu$	dynamic viscosity
$\tau$	shear stress

#### Subscripts

$R$	on radius R
$N$	Newtonian
$z$	in direction of z
$\varphi$	in direction of $\varphi$
$l$	on radius of inner cylinder

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