RHEOMETRY OF CONCENTRATED CERAMIC SUSPENSIONS - STEPS FROM MEASURED TO RELEVANT DATA

PART 1. ROTATIONAL VISCOMETER WITH COAXIAL CYLINDERS - POWER-LAW MODEL

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The knowledge of flow behaviour of ceramic suspensions is important condition for control of ceramic wet processing. The flow behaviour can be stated by the rheological measurements. The paper deals with measurement of the concentrated ceramic suspensions rheological behaviour. The procedure of rotational rheometry experimental data evaluation from rotational viscometer with coaxial cylinders is presented. The correction of data obtained by direct measurement with non-Newtonian suspension is proposed with the aim to obtain the relevant flow curve, i.e. dependence of shear stress on shear rate.

INTRODUCTION

The rheological behavior of ceramic suspensions affects significantly wet ceramic processing. On the base of knowledge of rheological parameters the technological parameters of various processes (mixing, batching, spray drying, slip casting, injection molding, dip coating etc.) can be determined and controlled [1-9]. For measurement of rheological behavior a variety of rheometer types and sensors are available [9,10]. For correct determination of rheological parameters the selection of proper geometry and sensors must be done. From the data measured the flow curves must be designed and parameters of appropriate rheological models must be calculated. The aim of this paper is to show how the flow curves necessary for parameters of rheological model evaluation can be obtained from primary experimental data received from measurements on rotational viscometer with coaxial cylinders.

THEORETICAL

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 τ on shear rate $\dot{\gamma}$ can be expressed by the following relation:

$$\tau = K\,\dot{\gamma}^{n} \tag{1}$$

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

The rotational viscometer with coaxial cylinders is often used in rheological measurements. Its common configuration consists of an inner rotating cylinder with radius R_1 and the outer stationary cylinder with the radius R_2 - see figure 1. The values of shear stress τ_1 and Newtonian shear rate γ_{1N} on inner cylinder are usually obtained from rheological measurements.

However the shapes of velocity and shear rate profiles between cylinders for power-law fluids (calculated by equations presented in [11]) are strongly dependent on flow behavior index n as it is shown in figures 2 and 3 for cylinder radii ratio $R_1/R_2 = \kappa = 0.5$. In figure 2 the dimensionless tangential velocity $u^* = u/u_1$ is defined as ratio of tangential velocity u to velocity of rotating cylinder u_1 and dimensionless coordinate is defined by relation $y^* = y/(R_2 - R_1)$ where y is radial distance from rotating cylinder. In figure 3 the dimensionless shear rate $\dot{\gamma}^* = -\dot{\gamma}/\omega$ is defined as ratio of shear rate value to inner cylinder angular velocity ω. Similar plots for $\kappa = 0.9$ are shown in figures 4 and 5. From figures 2 and 4 it can be seen that u* decreases from value 1 at rotated cylinder $y^* = 0$ to zero at stationary cylinder $y^* = 1$. From figures 3 and 5 it is obvious that maximum shear rate is at inner cylinder ($y^* = 0$) and minimum at outer cylinder ($y^* = 1$). From above mentioned figures for both κ values it can be also seen that the effect of flow behavior index n on the shape of velocity and shear rate profiles is more pronounced at smaller κ value. At $\kappa \to 1$ (parallel plate asymptote) the flow behavior index n has no effect on velocity and shear rate profiles - velocity profile is linear and shear rate is constant.

As it was shown in [11] the equation

$$\dot{\gamma}_1 = \frac{2\omega}{n\left(1-\kappa^{2/n}\right)} \tag{2}$$

holds for value of shear rate on inner cylinder.

Inserting $n = 1$, the following relation for Newtonian shear rate on inner cylinder $\dot{\gamma}_{1N}$ can be obtained

$$\dot{\gamma}_{1N} = \frac{2\omega}{\left(1-\kappa^2\right)} \tag{3}$$

Combining (2) and (3) we get

$$\dot{\gamma}_1 = \frac{1-\kappa^2}{n\left(1-\kappa^{2/n}\right)}\dot{\gamma}_{1N} \tag{4}$$

from which the value of real shear rate $\dot{\gamma}_1$ from the value of Newtonian shear rate $\dot{\gamma}_{1N}$ can be calculated. Dependencies of $\dot{\gamma}_1/\dot{\gamma}_{1N}$ ratio on flow behavior index n for values cylinder ratios $\kappa = 0.5$ and 0.9 are depicted in figure 6. From this figure it can be seen that values of this ratio (needed for Newtonian shear rate correction) increase with decreasing flow behavior index n and are significantly greater at $\kappa = 0.5$ than at $\kappa = 0.9$.

EXPERIMENTAL

The measurements were carried out on concentrated aqueous ceramic suspension (50 wt.%); the mass composition, density and mean diameter (median x_{50}) of solid components of suspension are listed in table 1. The particle size analyzer "analysette 22" (Fritsch) was used for the suspension particle size analysis. The particle size distributions of tabular alumina and the final mixture (x_{50} = 9.74 µm) are presented in figures 7 and 8.

Table 1. Composition of ceramic suspension.

component	(wt.%)	x_{50} (µm)	ρ(g/cm^3)
clay minerals	50	0.64	2.20
feldspar	25	4.10	2.62
tabular alumina	25	32.4	3.90

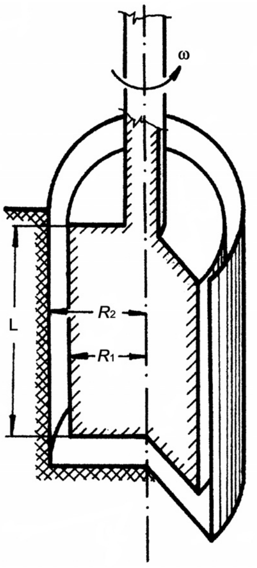

Figure 1. Viscometer with coaxial cylinders.

The rheological measurements were carried out with rotational viscometer with coaxial cylinders RV1 (ThermoHaake, Karlsruhe, Germany) equipped by sensors Z41 (κ = 0.954) and Z31 (κ = 0.725). The dependence of ratio of power law and Newtonian shear rates at inner cylinder on power law index n for both sensors used in experiments is shown in figure 9. From this figure it can be seen that the correction is much more pronounced for cylinder Z31 (with smaller R_1/R_2 ratio) in comparison with Z41 sensor. For example for Z31 cylinder and power law index $n = 0.2$ the real shear rate is for approximately 2.5 times greater than Newtonian shear rate; for cylinder Z41 the corresponding ratio of shear rates is 1.2. From figure 9 it also follows that at values $n > 0.5$ and cylinder Z41 correction is not significant but for cylinder Z31 and small n values the correction of Newtonian shear rate values cannot be omitted.

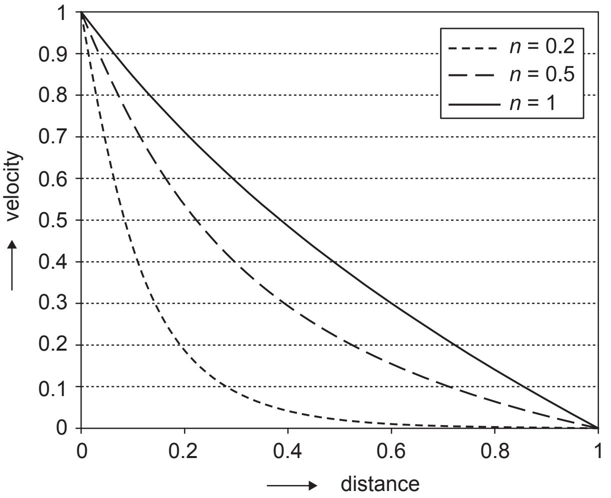

Figure 2. Dependence of dimensionless velocity u^* on dimensionless distance y^* for κ = 0.5 and selected flow behavior index values n.

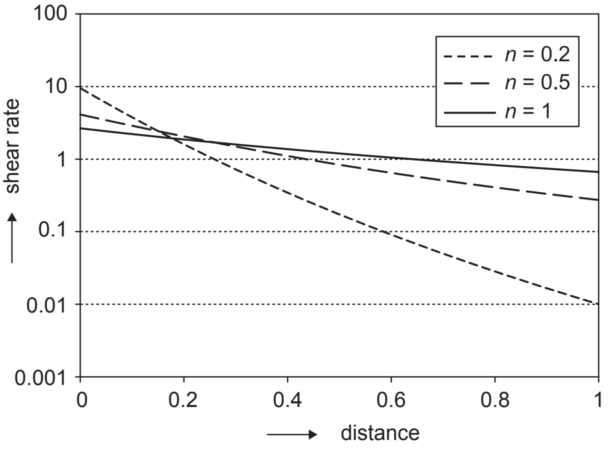

Figure 3. Dependence of dimensionless shear rate values $\dot{\gamma}^*$ on dimensionless distance y^* for κ = 0.5 and selected flow behavior index values n.

RESULTS AND DISCUSSION

The experimentally obtained dependences of τ_1 on Newtonian shear rate $\dot{\gamma}_{1N}$ measured by both sensors (calculated by standard viscometer software's) are shown in figure 10.

These same corrected dependences, i.e. dependences of τ_1 on real (non-Newtonian) shear rate $\dot{\gamma}_1$ (flow curves) are shown in figure 11. On the basis of comparison of dependences in figures 10 and 11, it is clear that from different dependences obtained from viscometer measurement shown in figure 10 we obtain dependence of shear stress on shear rate depicted in figure 11. From dependence in figure 11 the parameters of power law model *K* and *n* can be calculated.

On the basis of results presented above the following pro-cedure can be recommended for viscometer data evaluation:

1) Applying the power function regression on primary rheometric data ($\tau_1 = f(\dot{\gamma}_{1N})$), the exponent *n* can be obtained ($\tau_1 \approx \dot{\gamma}^{n}_{1N}$).
2) The values $\dot{\gamma}_1$ can be calculated by equation (4) from the corresponding values of Newtonian shear rate $\dot{\gamma}_{1N}$, real flow curve ($\tau_1 = f(\dot{\gamma}_1^{n})$) can be received and coefficient of consistency *K* can be obtained.

The use of Newtonian instead of real shear rates values can also lead to significant mistakes in the reference shear rate and apparent viscosity values.

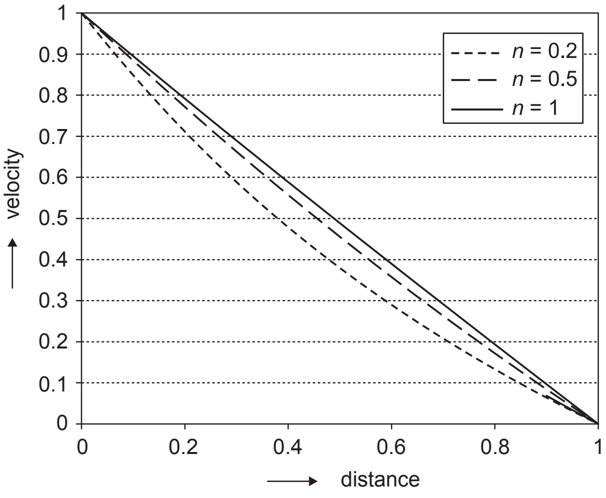

Figure 4. Dependence of dimensionless velocity u^* on dimensionless distance y^* for κ = 0.9 and selected flow behavior index values *n*.

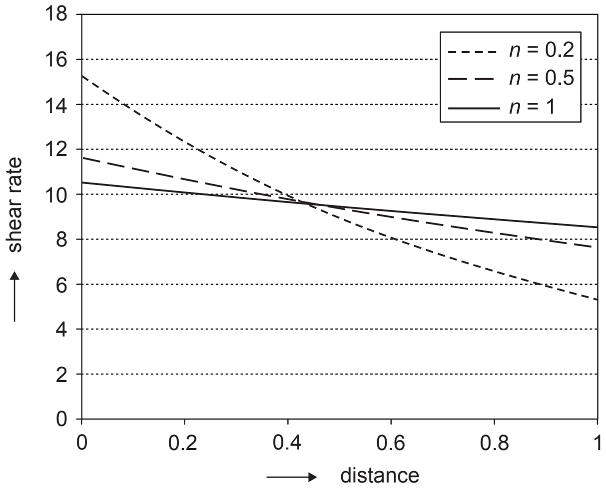

Figure 5. Dependence of dimensionless shear rate values $\dot{\gamma}^*$ on dimensionless distance y^* for κ = 0.9 and selected flow behavior index values *n*.

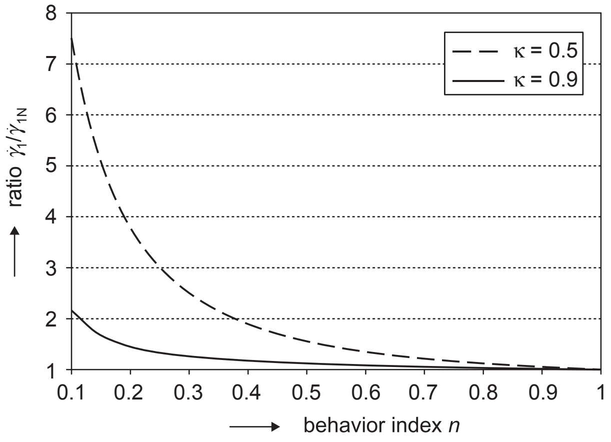

Figure 6. Dependence of ratio $\dot{\gamma}_1/\dot{\gamma}_{1N}$ on flow behavior index *n* for selected κ values.

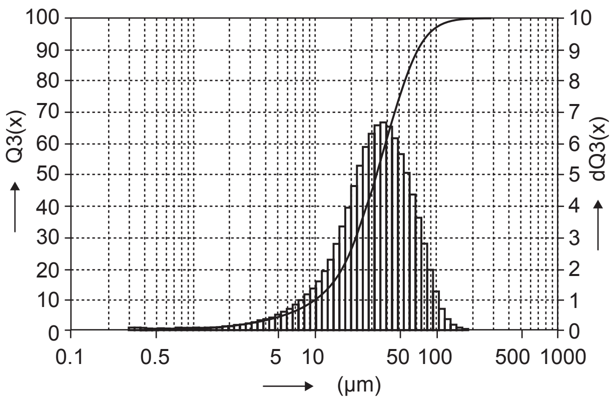

Figure 7. Particle distribution of tabular alumina.

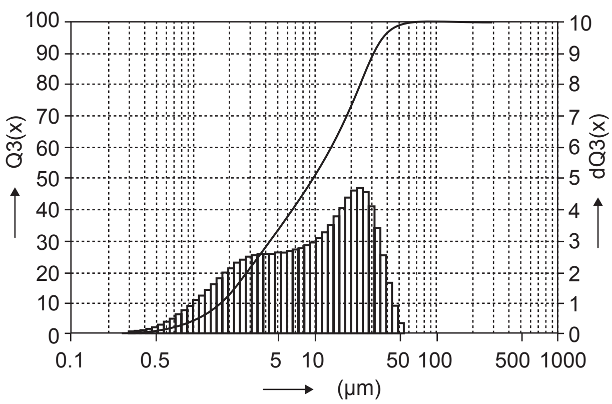

Figure 8. Particle distribution of clay–feldspar–alumina mixture.

CONCLUSION

On the basis of the theoretical analysis of non-Newtonian fluids (power law model) flow between coaxial cylinders of rotational viscometer the correction of experimental data was recommended. The use of the proposed method is illustrated on rheological measurement of concentrated ceramic suspension with the components of different particle diameter. The rheological measurements were carried out by rotational rheometry method on viscometer RV1 (ThermoHaake) equipped with coaxial cylinders sensors of different diameter (Z31 and Z41). On the basis of presented results the procedure for data evaluation is recommended.

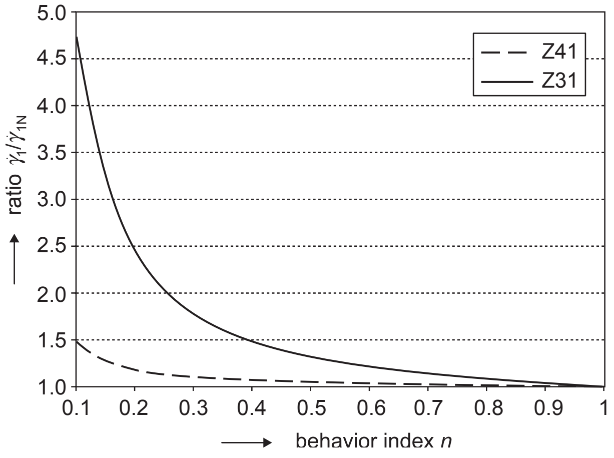

Figure 9. Dependence of ratio $\dot{\gamma}_1/\dot{\gamma}_{1N}$ on flow behavior index *n* for sensors Z41 and Z31.

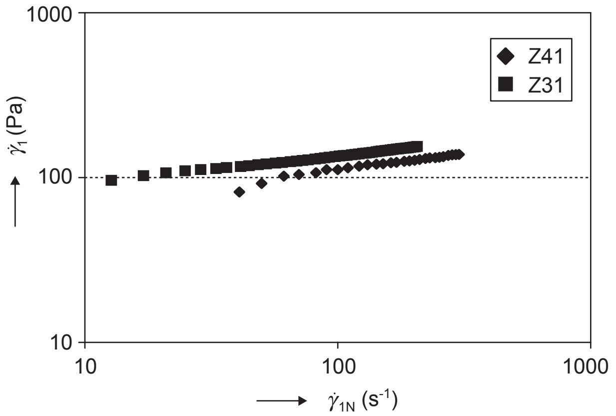

Figure 10. Dependence of shear stress τ_1 on Newtonian shear rate $\dot{\gamma}_{1N}$ measured with sensors Z41 and Z31.

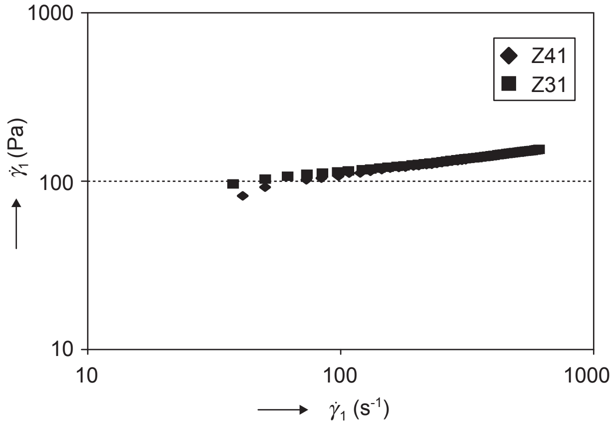

Figure 11. Dependence of shear stress τ_1 on shear rate $\dot{\gamma}_1$ (flow curve) measured with sensors Z41 and Z31.

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REOMETRIE KONCENTROVANÝCH KERAMICKÝCH SUSPENZÍ - OD MĚŘENÝCH K RELEVANTNÍM HODNOTÁM - ČÁST 1. ROTAČNÍ VISKOZIMETR SE SOUOSÝMI VÁLCI; MOCNINOVÝ MODEL

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