

Influence of Dispersed Phase Characteristics on Rheological Behavior of Suspensions

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The rheological behavior of clay and kaolin suspensions has been investigated in order to find possible effects of dispersed phase characteristics. Those effects were described using property functions. Clay particles were dispersed in water at four different solid volume fractions (6.4 %, 8.8 %, 11.4 %, and 14.2 %). At each solid volume fraction, six clay samples were used having different particle size distribution. Particle size distribution was described using LN distribution function where the measure of particle size range varied from 5.07 to 8.48 μm , and the measure of spread from 0.448 to 0.513. The same solid volume fractions were used for dispersing kaolin particles in the water. LN distribution function was also used for defining particle size distribution of six different kaolin samples. Measure of particle size range varied from 4.24 to 4.94 μm , while the measure of spread was ranging from 0.446 to 0.512. Rheological measurements using rotational rheometer showed that all used kaolin suspensions can be described using Herschel-Bulkley model while all clay suspensions showed Bingham behavior at any concentration. The obtained rheological parameters were correlated with solid volume fraction and external specific surface what resulted with two parameter models. Those models were joined into one equation that describes suspension plastic viscosity and enables viscosity estimations when solid volume fraction and external specific surface are known particle characteristics. Very good results were obtained for highly concentrated suspensions mostly used in process industry.

Keywords:

Bingham model, external specific surface, Herschel-Bulkley model, property function, specific pore volume, suspension rheology

Introduction

Knowledge about effects of physical properties of dispersed phase on technological properties is defined with property functions. Importance of property functions in chemical process engineering is very significant nowadays. Finding a model that is joining rheological parameters, dispersed phase characteristics and solid volume fraction, and is valid for all suspensions, is a great challenge.

Suspensions are very common engineering products or byproducts in many industries. Good definition of conveying conditions, mixing parameters, pump selection, valve selection etc. is possible only if suspension characteristics are known.¹ The most important process characteristic of suspension is its viscosity. Therefore, it is necessary to find connection between dispersed phase and continuous phase. Most authors have described influence of solid volume fraction on viscosity. For example, the Einstein equation describes the solid volume fraction for highly diluted suspension ($\Phi = 10$ % and less solid phase volume)^{2,3}

$$\eta = \eta_s \cdot (1 + 2.5 \Phi_V) \quad (1)$$

where η_s is viscosity of continuous phase and Φ_V is solid volume fraction.

Equation (1) shows that only solid volume fraction influence is included while particle size distribution and particle interactions are neglected. Batchelor did some modifications of Einstein equation and introduced the square of solid volume fraction to include particle interactions.^{2,3}

$$\eta = \eta_s \cdot (1 + 2.5 \Phi_V + 6.2 \Phi_V^2) \quad (2)$$

Therefore, equation (2) includes particle particle interactions and particle medium interactions beside the characteristics of dispersed phase.

When talking about high solid content suspensions it is necessary to define packing fraction because particles in suspension form certain packing, so called three dimensional structures.^{2,4,5,6} Maximum packing fraction is solid volume fraction where solid content is so high that three dimensional structures make suspension flow impossible, and viscosity tends to infinity. Particle size distribu-

tion and shape are parameters that define maximum packing fraction. Often used equation for viscosity of concentrated suspensions is Krieger-Dougherty model:⁷

$$\eta_r = \left(1 - \frac{\Phi_V}{\Phi_{V, \max}}\right)^{-[\eta]\Phi_{V, \max}} \quad (3)$$

and the Quemada model

$$\eta_r = \left(1 - \frac{\Phi_V}{\Phi_{V, \max}}\right)^{-2} \quad (4)$$

where η_r is relative viscosity, $\Phi_{V, \max}$ is the shear dependent maximum packing fraction and $[\eta]$ is intrinsic viscosity (2.5 for spheres).

The maximum packing fraction depends on the arrangement of particles, determined by particle shape and particle size distribution. So, as it can be seen from equation (3) and (4), viscosity depends on solid volume fraction and maximum packing fraction what implies indirect influence of particle shape and particle size distribution on suspension viscosity. Some authors even suggested models that connect rheological parameters (yield point, consistency index, flow behavior index) and solid volume fraction.

As it was said before, solid volume fraction has a great influence on suspension rheology, but particle size distribution is also very important influencing parameter concerning solid-liquid mixtures rheology. This work contains description of method that was used to obtain models which enable viscosity estimation for different suspensions described with Herschel-Bulkley and Bingham rheological behavior model.

Experiment

Dispersed phase characteristics

Influence of dispersed phase characteristics was monitored with changing particle size distribution, specific pore volume and external specific surface area. Particle and pore size distribution were described using LN distribution function and parameters of that function. Laser scattering using Malvern Mastersizer X was used for particle size distribution measurements. Six kaolin samples had measure of spread (LN function) in range of 0.446–0.512 while measure of particle size range varied from 4.24–4.95 μm (Table 1.). Clay samples had similar measure of spread range as kaolin (0.448–0.513) while measure of particle size range was a little wider (5.07–8.48 μm) (Table 2.). Earlier

Table 1 – Characteristics of kaolin samples

No	External specific surface $S_m, \text{m}^2 \text{kg}^{-1}$	Parameters of LN function for particle size distribution		Specific pore volume $V_p, \text{cm}^3 \text{g}^{-1}$	Specific surface $S_{\text{BET}}, \text{m}^2 \text{g}^{-1}$	Parameters of LN function for pore size distribution	
		σ_{LN}	$x_{50}, \mu\text{m}$			σ_{LN}	$x_{50}, \text{\AA}$
K1	582.6	0.512	4.95	0.0752	17.8	0.656	410
K2	632.5	0.496	4.82	0.0796	18.9	0.632	381
K3	701.3	0.476	4.52	0.0910	20.1	0.616	353
K4	740.2	0.466	4.37	0.0905	20.7	0.578	327
K5	766.2	0.459	4.29	0.0900	21.4	0.541	299
K6	780.9	0.446	4.24	0.0889	21.9	0.537	288

Table 2 – Characteristics of clay samples

No	External specific surface $S_m, \text{m}^2 \text{kg}^{-1}$	Parameters of LN function for particle size distribution		Specific pore volume $V_p, \text{cm}^3 \text{g}^{-1}$	Specific surface $S_{\text{BET}}, \text{m}^2 \text{g}^{-1}$	Parameters of LN function for pore size distribution	
		σ_{LN}	$x_{50}, \mu\text{m}$			σ_{LN}	$x_{50}, \text{\AA}$
C1	504.1	0.513	8.48	0.1050	42.3	0.486	160
C2	549.1	0.516	7.28	0.0961	45.4	0.507	164
C3	610.2	0.503	6.70	0.0905	49.8	0.523	170
C4	658.3	0.501	6.21	0.0826	51.3	0.538	183
C5	727.9	0.497	5.55	0.0802	53.7	0.556	190
C6	782.5	0.488	5.07	0.0753	56.3	0.569	192

research shows that particle external specific surface is of great importance for suspension rheological behavior because of forces acting on particle surface.^{8,9} Therefore, it was necessary to determine external specific surface, although particle size distribution was already defined. Permeability method showed that particle external specific surface for kaolin was in range 582.6–780.9 m² kg⁻¹ and 504.1–782.5 m² kg⁻¹ for clay. Particle size distribution and particle external specific surface are important characteristics of dispersed phase, but complete definition of that phase involves knowledge about pores inside material. Analyzing adsorption isotherms obtained using ASAP 2000 (S_{BET}) it was shown that used kaolin and clay samples had a great quantity of pores inside material, and surface of pores is much higher than external specific surface. In spite of that fact, external specific surface is more significant in study of rheological behavior because attractive and repulsive forces are acting only on the surface and those inside material are negligible. Therefore, it was more interesting to define specific pore volume and it was later shown as justifying. Characteristics of kaolin and clay samples are shown in tables 1 and 2.

Rheological behavior of suspensions

Suspensions were obtained dispersing clay or kaolin particles in water at different solid volume fractions (6.4 %, 8.8 %, 11.4 % and 14.2 %). Since six different particle size distributions were used for clay (C1 to C6) as well as for kaolin (K1 to K6), at four solid volume fractions, twenty-four sample suspensions were obtained for clay samples and also for kaolin samples. Both types of suspensions were stable meaning that no settling effect was noticed during measuring since all measurements were carried out at room temperature. Rheological behavior of suspensions was investigated using Brookfield DVIII rheometer. Obtained flow curves showed that existing flow behavior models can be used, and therefore, Bingham model was appropriate for mathematical description of rheological behavior for clay suspensions

$$\tau = \tau_0 + \eta_p \cdot \dot{\gamma} \quad (5)$$

while kaolin suspensions were described using Herschel-Bulkley model

$$\tau = \tau_0 + K \cdot \dot{\gamma}^n \quad (6)$$

Values of rheological parameters are shown in tables 3 and 4.

Table 3 – Rheological parameters for Herschel-Bulkley suspensions

Φ_v , %	Yield point, τ_0 / Pa					
	K1	K2	K3	K4	K5	K6
6.4	0.66	0.59	0.42	0.28	0.14	0.08
8.8	2.23	1.95	1.43	0.95	0.44	0.20
11.4	7.35	6.26	3.52	2.83	2.02	1.50
14.2	20.21	19.11	17.10	15.13	13.34	9.62
Φ_v , %	Flow behavior index, n					
6.4	0.815	0.738	0.558	0.432	0.358	0.338
8.8	0.802	0.721	0.521	0.348	0.309	0.230
11.4	0.765	0.713	0.402	0.285	0.142	0.146
14.2	0.752	0.678	0.254	0.208	0.173	0.134
Φ_v , %	Consistency index, K / Pa s ⁿ					
6.4	0.021	0.023	0.411	0.437	0.507	0.454
8.8	0.045	0.053	0.927	1.223	1.144	1.584
11.4	0.121	0.119	3.347	4.825	5.647	3.814
14.2	0.286	0.565	5.639	8.335	9.734	11.978

Table 4 – Rheological parameters for Bingham suspensions

Φ_v , %	Yield point, τ_0 / Pa					
	C1	C2	C3	C4	C5	C6
6.4	0.45	0.38	0.33	0.20	0.13	0.10
8.8	0.74	0.52	0.45	0.32	0.30	0.22
11.4	7.22	1.35	1.15	1.03	0.78	0.58
14.2	33.14	7.86	6.85	5.26	4.58	2.26
Φ_v , %	Plastic viscosity, η_p / Pa s					
6.4	0.076	0.052	0.055	0.046	0.045	0.035
8.8	0.305	0.124	0.302	0.308	0.263	0.178
11.4	1.078	1.396	1.408	0.807	0.584	0.423
14.2	55.612	33.861	15.854	7.135	6.428	3.822

Results and discussion

Influence of particle size distribution on rheological behavior of suspensions was obvious and can be seen in the change of suspension flow curves (Fig. 1., 2.) and rheological parameters (yield point, consistency index, flow behavior index and plastic viscosity) (Table 3, 4).

Since the influence of solid volume fraction on rheological behavior was already investigated in some previous works, it was necessary to find out

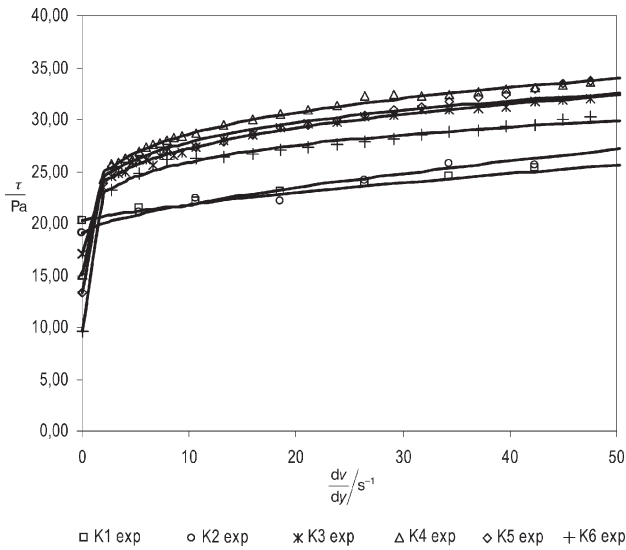


Fig. 1 – Flow curves for Herschel-Bulkley suspensions ($\Phi_V = 14.2\%$); K1 exp – K6 exp are experimental values, while lines represent fitting curves used for modeling approach

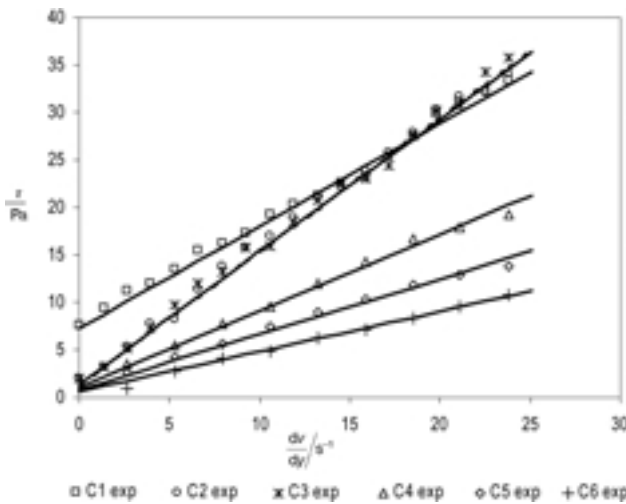


Fig. 2 – Flow curves for Bingham suspensions ($\Phi_V = 11.4\%$); C1 exp – C6 exp are experimental values, while lines represent fitting curves used for modeling approach

whether existing models can be used for description of given system. Previous works showed that some rheological parameters could be directly connected with solid volume fraction using some equations while influence on other rheological parameters was discussed only theoretically.^{2,3,10,11} For example, it was found that relationship of yield point and solid volume fraction can be described using simple power model¹²

$$\tau_0 = a_1 \cdot \Phi_V^{b_1} \quad (7)$$

where a_1 and b_1 are constants corresponding to used system, and dependent on many factors, such as particle size distribution as well as interactions be-

tween dispersed phase and continuous phase. Measurements taken with kaolin and clay suspensions showed that such model can be used for both suspensions since both have yield point. Appearance of yield point can be explained with existing of three-dimensional structures. Particles in suspensions form that kind of structures, so suspension starts to flow only when force threshold is reached.^{13,14} The stronger the structures are, what will happen if there are attractive forces between particles, the higher force is needed to start the flow. Relationship (7) is limited to a narrow particle concentration range. Therefore, lower particle concentration limit was introduced, since suspension does not exhibit plastic behavior under that minimal concentration of the solid phase:

$$\tau_0 = a_1 \cdot (\Phi_V - \Phi_{V0})^{b_1} \quad (8)$$

Φ_{V0} is the minimal solid volume fraction where suspension still exhibit plastic behavior¹⁵. Materials used in this study exhibit plastic behavior above $\Phi = 6.4\%$ solid content.

Increase of solid volume fraction resulted with increase of the yield point, as expected. Parameters a_1 and b_1 in power model (eq. 8) changed with particle size distribution and therefore those parameters were correlated with the external specific surface. Such approach resulted with, so called, two parameter models that include solid volume fraction and external specific surface:

$$\tau_0 = a \cdot S_m^b \cdot (\Phi_V - \Phi_{V0})^{c-S_m} \quad (9)$$

Constants a , b and c are not dependent of external specific surface and can be used for any solid volume fraction greater than Φ_{V0} . While increase of solid volume fraction resulted with increase of yield point, increase of external specific surface showed yield point decrease. Such phenomena cannot be explained only analyzing external specific surface influence but also some other dispersed phase characteristics. But first of all, other rheological parameters were correlated at same way as yield point. Flow behavior index and consistency index in Herschel-Bulkley model were also correlated with solid volume fraction:

$$n = 1 - a_2 \cdot (\Phi_V - \Phi_{V0})^{b_2} \quad (10)$$

and

$$K = \eta_w + a_3 \cdot (\Phi_V - \Phi_{V0})^{b_3} \quad (11)$$

From equations (10) and (11) can be seen that the power model can be used for estimation of both indexes. In equation (10) flow behavior index is equal to unity when solid phase reaches the mini-

mal value, what means that suspension is showing Newtonian behavior. Consistency index (eq. 11) is changed with solid volume fraction and is dependent of continuous phase viscosity; in given case that was water. When suspension has some minimal solid volume fraction, consistency index is equal to viscosity of continuous phase because there is no plastic behavior under that fraction value. Changes in temperature will change the viscosity of given suspension. Therefore, viscosity of continuous phase is important. Consistency index for Herschel-Bulkley suspensions is also dependent of external specific surface because it was shown that variation in particle size distribution resulted with increase of consistency index. Parameters a and b in equations (10) and (11) were correlated with external specific surface, and two parameter models were obtained:

$$n = 1 - g \cdot S_m^h \cdot (\Phi_V - \Phi_{V0})^k \quad (12)$$

$$K = \eta_w + d \cdot S_m^e \cdot (\Phi_V - \Phi_{V0})^{f-S_m} \quad (13)$$

Mathematical description of plastic viscosity and solid volume fraction was different for Bingham suspensions. In that case, it was found that exponential model gives very good results:

$$\eta_p = \eta_w \cdot e^{a_4 \cdot (\Phi_V - \Phi_{V0})} \quad (14)$$

Equation (14) shows that plastic viscosity is dependent on continuous phase viscosity and solid volume fraction, and parameter a_4 is dependent on external specific surface and can be correlated with it, what gives two parameter equation:

$$\eta_p = \eta_w \cdot e^{p-S_m^i \cdot (\Phi_V - \Phi_{V0})} \quad (15)$$

Experimental data showed decrease in plastic viscosity with increase in external specific surface at the same solid volume fraction value. As it was said before, the greater external specific surface, the stronger are attractive forces between particles, and higher forces are needed to break three-dimensional structures, so yield point, consistency index, and plastic viscosity values are supposed to increase. It was found that yield point for both suspensions and plastic viscosity for Bingham suspensions decrease with external specific surface increase. Such behavior was not expected and therefore viscosity of suspension was analyzed because it includes all rheological parameters and, for example, later on can be used for estimation of hydrodynamic regime (determination of Reynolds number for suspension), for example. Viscosity of Herschel-Bulkley suspensions can be calculated according to equation (16), and for Bingham suspensions according to equation (17):

$$\eta_{HB} = \frac{\tau_0}{\dot{\gamma}} + K \cdot \dot{\gamma}^{n-1} \quad (16)$$

$$\eta_B = \frac{\tau_0}{\dot{\gamma}} + \eta_p \quad (17)$$

Using obtained two parameter models, equation (16) can also be written as:

$$\eta_{HB} = \frac{a}{\dot{\gamma}} \cdot S_m^b \cdot \Phi^{c \cdot S_m} + (\eta_v + d \cdot S_m^e \cdot \Phi^{f \cdot S_m}) \cdot \dot{\gamma}^{1-g \cdot S_m^b \cdot \Phi^k} \quad (18)$$

where $\Phi = \Phi_V - \Phi_{V0}$, and constants given in eq. 18 are the same that were used in two parameter models (eq. 9, 12, 13).

Equation (17) in form of two parameter models can be written as:

$$\eta = \frac{a}{\dot{\gamma}} \cdot S_m^b \cdot \Phi^{c \cdot S_m} + \eta_w \cdot e^{p \cdot S_m^i \cdot \Phi} \quad (19)$$

where parameters a , b and c are different from those in equation (18), but are used because the yield point for Bingham suspension can be expressed using equation (9) as well as for Herschel-Bulkley suspension.

Viscosities were obtained at shear rate of 50 s^{-1} , and equations (18) and (19) give good results at that shear rate. Results showed that viscosity of suspension is changed with solid volume fraction and external specific surface, but also dependent of some other dispersed phase characteristics. Figures 3 and 4 represent change of relative viscosity for Herschel-Bulkley and Bingham suspension at 50 s^{-1} , according to experimental results, while figures 5 and 6 represent comparison of experimental relative

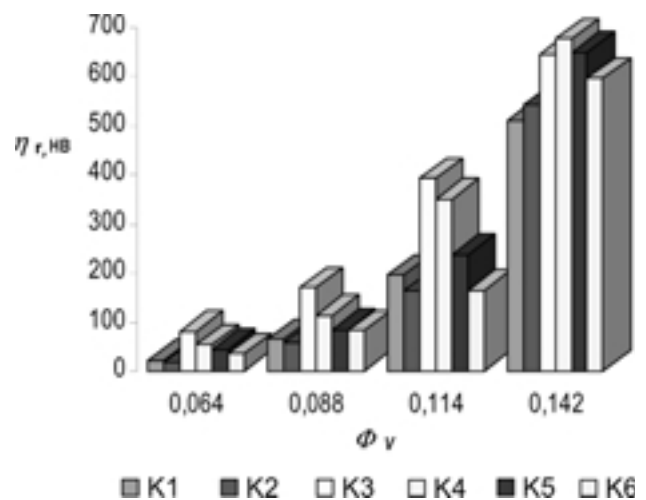


Fig. 3 – Relative viscosities of Herschel-Bulkley suspensions at different solid volume fractions and external specific surfaces (shear rate 50 s^{-1})

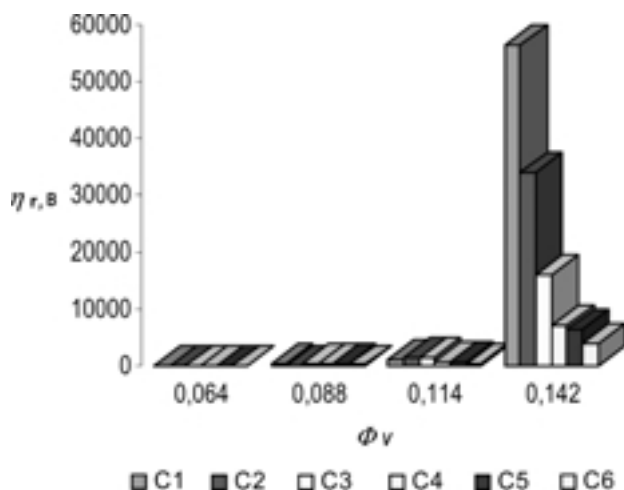


Fig. 4 – Relative viscosities of Bingham suspensions at different solid volume fractions and external specific surfaces (shear rate 50 s^{-1})

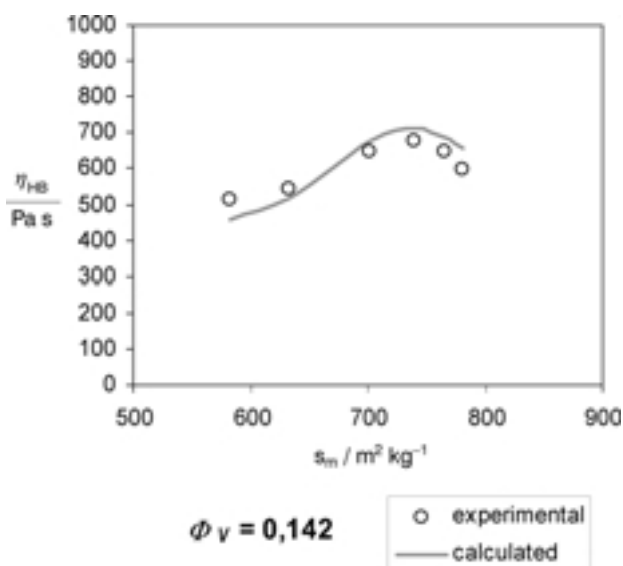


Fig. 5 – Comparison of experimental and calculated (eq. 18) viscosities for Herschel-Bulkley suspensions at different external specific surfaces ($\Phi_V = 14.2\%$ solid content)

viscosities and those obtained using models (18) and (19).

When analyzing Herschel-Bulkley suspensions (Fig. 3 and 5) and their relative viscosities it can be seen that at the same solid volume fraction, viscosity increase with increase of external specific surface to some maximum value and then starts to decrease while external specific surface still increases. Explanation for such behavior was found in specific pore volume. Specific pore volume for kaolin samples increases at the same time with increase of external specific surface. When maximum specific pore volume is reached, it starts to decrease with increase of external specific surface. Maximum value of specific pore volume and maximum suspension

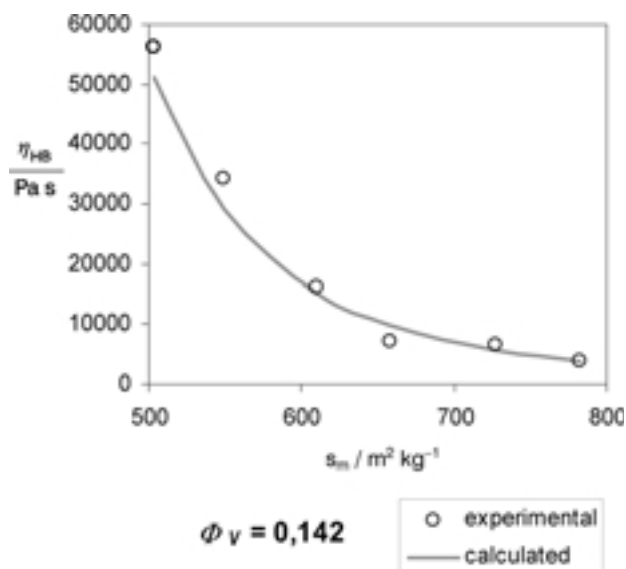


Fig. 6 – Comparison of experimental and calculated (eq. 19) viscosities for Bingham suspensions at different external specific surfaces ($\Phi_V = 14.2\%$ solid content)

viscosity occurred at same value of external specific surface. That means that specific pore volume has a greater influence on rheological behavior than change of external specific surface. When value of specific pore volume starts to increase, decrease of apparent solid volume fraction happens because of continuous phase that is filling the pores. Therefore, there is a smaller quantity of continuous phase in suspension what resulted with greater contact surface area per unit volume, and greater value of contact surface area means increase of suspension viscosity. When specific pore volume starts to decrease, reverse process is happening. Smaller quantity of continuous phase is filling the pores, apparent solid volume fraction decreases, and although external specific surface still increases, viscosity of suspension decreases. Differentiation of equation (18) gives the equation that enables determination of external specific surface “critical” value:

$$\begin{aligned} \frac{d\eta_{HB}}{ds_m} = & \frac{a \cdot b}{\dot{\gamma}} \cdot S_m^{b-1} \cdot \Phi^{c \cdot S_m} + \\ & + \frac{a \cdot c}{\dot{\gamma}} \cdot S_m^b \cdot \Phi^{c \cdot S_m} \cdot \ln(\Phi) + \\ & + [d \cdot e \cdot S_m^{e-1} \cdot \Phi^{f \cdot S_m} + d \cdot f \cdot S_m^e \cdot \Phi^{f \cdot S_m} \cdot \ln(\Phi)] \cdot \\ & \cdot \dot{\gamma}^{(-g \cdot S_m^b \cdot \Phi^k)} - \\ & - (\eta_w + d \cdot S_m^e \cdot \Phi^{f \cdot S_m}) \cdot \dot{\gamma}^{(-g \cdot S_m^b \cdot \Phi^k)} \cdot \\ & \cdot g \cdot h \cdot S_m^{b-1} \cdot \Phi^k \cdot \ln(\Phi) \end{aligned} \quad (20)$$

Until this value is reached, external specific surface area has a greater influence on suspension viscosity. When the external specific surface value is greater than “critical”, material structure is very

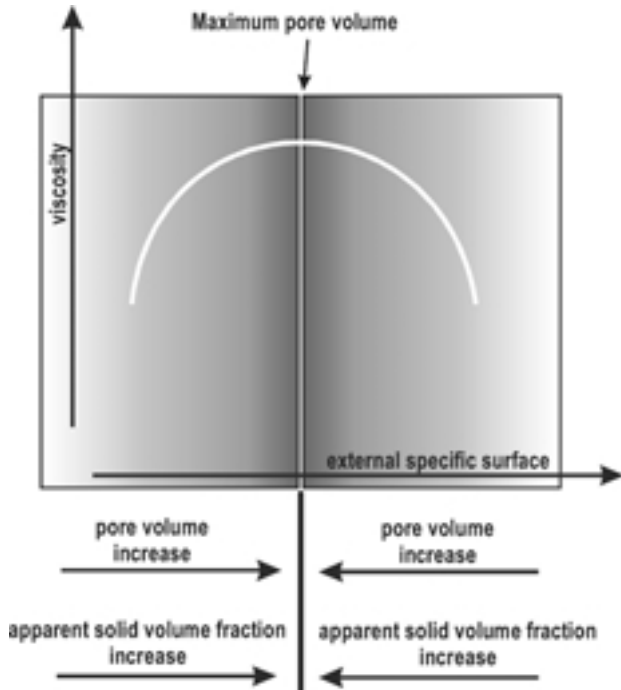


Fig. 7 – Influence of pore volume and external specific surface on Herschel-Bulkley suspension viscosities

important for suspension viscosity. Influence of external specific surface and specific pore volume on viscosity of Herschel-Bulkley suspensions is schematically represented in figure 7.

Same thing happened with Bingham suspensions. Results showed that viscosity of Bingham suspensions decrease with increase of external specific surface for all solid volume fractions (Fig. 4

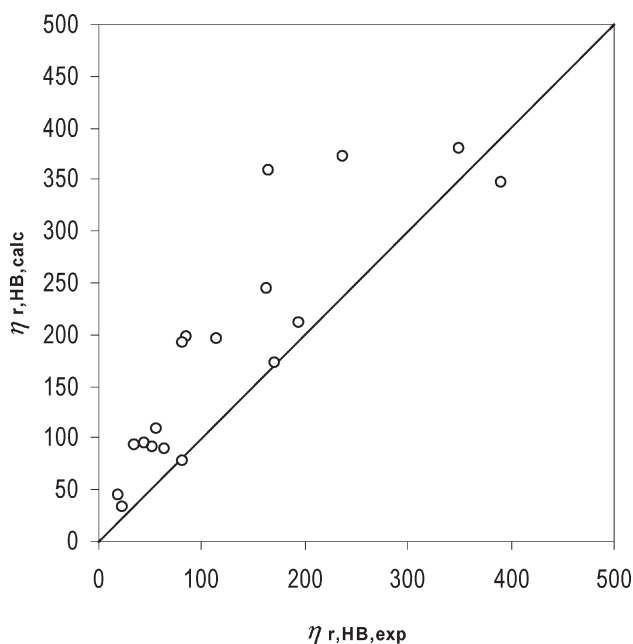


Fig. 8 – Experimental and calculated viscosities; Herschel-Bulkley suspensions

and 6). Analysis of material structure showed that specific pore volume decreases while external specific surface increases and that was just confirmation of theory based on Herschel-Bulkley suspensions. The only difference is that “critical” value of external specific surface is smaller than minimal used in experiment.

Comparison of experimental and calculated viscosities based on obtained two parameter models showed good agreement what could be seen in figure 8 and 9. Mean relative deviation for viscosities of Herschel-Bulkley suspensions is up to 35 %, and for Bingham suspensions up to 20 %. Obtained empirical models give good results when higher suspension viscosities are used, what is very important in industries where used suspensions are mostly high concentrated and very viscous.

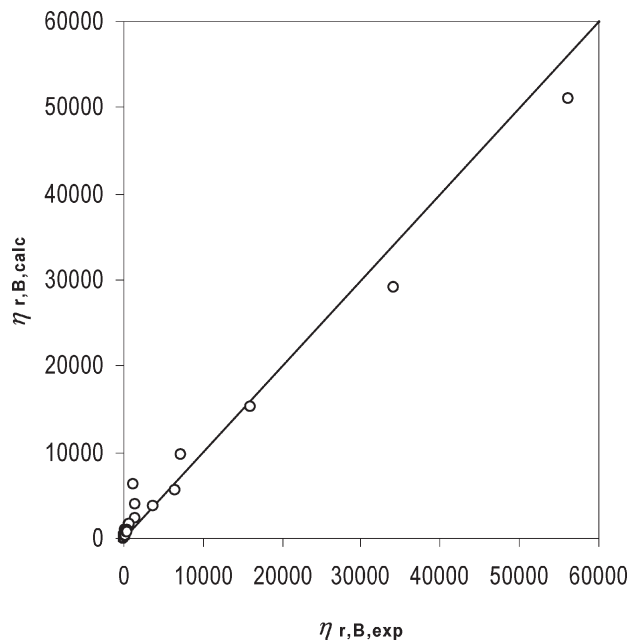


Fig. 9 – Experimental and calculated viscosities; Bingham suspensions

Conclusions

Rheological behavior of suspensions depends on solid volume fraction and dispersed phase characteristics.

External specific surface is most important dispersed phase characteristic relevant for defining of rheological behavior until “critical” value of external specific surface is reached. It is important to notice that at certain values of external specific surface, specific pore volume also has influence on rheological behavior of suspension.

Obtained two parameter models for both suspensions showed that they can be used for estima-

tion of rheological parameters especially for viscous suspensions mostly used in the process industry. Equation (20) could be very useful for determination of “critical” value of external specific surface, but also includes a large number of parameters and therefore needs further research. The other limit of given models is that they can be used only for Newtonian disperse medium since rheological behavior of continuous phase has an influence on final behavior of suspension. Therefore, given models can be used with mentioned limitations, but possibilities of using such models for different types of suspensions still need further work for confirmation.

Symbols

$a_1, a_2, a_3, a_4, b_1, b_2, b_3$ – constants in one-parameter models

$a, b, c, d, e, f, g, h, k, p, r$ – constants in two-parameter models

K – consistency index (Herschel-Bulkley model), Pa sⁿ

n – flow behavior index (Herschel-Bulkley model)

S_{BET} – specific surface (BET method), m² kg⁻¹

S_m – external specific surface, m² kg⁻¹

V_p – specific pore volume, m³ kg⁻¹

x_{50} – measure of particle size range (LN function), m

Φ_V – solid volume fraction, %

Φ_{V0} – minimal solid volume fraction, %

$\Phi_{V,\text{max}}$ – maximum packing factor, %

$\dot{\gamma}$ – shear rate, s⁻¹

η – viscosity, Pa s

η_B – Bingham suspension viscosity, Pa s

η_{HB} – Herschel-Bulkley suspension viscosity, Pa s

η_p – plastic viscosity, Pa s

η_r – relative viscosity

η_w – water viscosity, Pa s

σ_{LN} – measure of spread (LN function)

τ – shear stress, Pa

τ_0 – yield point, Pa

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