A RHEOLOGICAL MODEL FOR CEMENT AND CLAY SUSPENSIONS

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A method is given for selecting an adequate rheological model for a viscoplastic medium.

Bingham Shvedov and Ostwald de Waal rheological models [1-3] are widely used to describe the rheological behavior of the cement and clay suspensions used in drilling deep oil and gas wells.

However, sometimes these models do not correspond to the actual flow curves for such suspensions, because the Bingham-Shvedov model does not incorporate the nonlinearity of the flow curves, while the Ostwald de Waal model neglects the limiting shear stress. Current chemical treatments used with cement and clay suspensions, particularly polymers (metas, gipan, okzil, KMTs,\* etc.), increase the nonlinear ranges. Major errors may arise in hydraulic calculation if the nonlinearity is neglected. The Casson-Shulman nonlinear viscoplastic model is therefore of interest, since it is free from these shortcomings [4]. One therefore has to examine the adequacy of the Casson-Shulman model with regard to the rheological behavior of various cement and clay suspensions at a variety of concentrations and for various solid phases at different temperatures and in the presence of other reagents.

Measurements on the rheology of the dispersed system require allowance for factors such as nonstationary behavior in the rheological characteristics (particularly for cement suspensions), sedimentation, wall and end defects, etc. This study was performed with a capillary rheometer of novel design type UITs-1 and with Reotest-1 and Reotest-2 rotation rheometers [5]. The working capillaries in the UITs-1 were stainless-steel tubes of length 0.9-1.5 m and of internal diameter  $(5.963-10.007)\cdot 10^{-3}$  m; the range of shear rates was from 10 to  $600 \text{ sec}^{-1}$ . The UITs-1 differs from standard capillary rheometers in containing a multistage telescopic plunger system, in which one working stroke gives six different flow rates. The flow rates are changed without halt or modification in the rheometer. At each flow rate measurements are made of the pressure difference across the working parts of several capillaries of different diameters and lengths.

The measurements with the rotation viscometers were made with six combinations of cylinders with relative gaps from 0.81 to 0.98. These gave shear rates in the range from 0.2 to  $1312 \text{ sec}^{-1}$ . Various grades of Portland cement were used in the cement suspensions (Komsomolets, Bezmeinskii, Bol'shevik, Zdolbunovskii, Sterlitamakskii, etc.) which are widely used in cementing boreholes. The grain sizes of the initial materials were such that the specific surfaces were 2000-10,000 cm<sup>2</sup>/g, while the water-cement ratio W/C ranged from 0.3 to 1.0. The chemical reagents were hydrolyzed polyacrilonitrile (gipan) and lignosulfonate (okzil) at concentrations of 0.5-2.0 and 0.2-1.0% in terms of the mass of the cement, respectively. The clay suspensions were prepared from Druzhkovo and Sarigyukh bentonites with concentrations of the solid phase from 3 to 22%. The temperatures of the suspensions varied from 20 to 50°C. The suspensions were treated with a copolymer of methacrylic acid and methacrylamide (metas) at concentrations of from 0.1 to 0.8% by mass.

Techniques from stationary rheology were used in determining the flow curves for the cement suspensions; the time periods within which there were no visible changes in the measurable characteristics were established by trial for each suspension. This allowed us to evaluate the kinetics of the structuring by means of quasistationary yield curves. Stationary flow curves were provided for the clay suspensions by leaving the clay paste to stand

\*These names are explained below.

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for prolonged periods before preparing the suspensions and carefully mixing the suspensions before measurement.

A study of the sedimentation effects in the capillary and rotation rheometers showed that the mean error from sedimentation was not more than 2-3% in most instances, although the maximum error was 10% for coarse-grained systems. The solution was prevented from separating in the working chamber of the UITs-1 rheometer by a four-blade stirrer. The optimum speed for the stirrer was selected by a special method.

Additional tests were done in order to estimate the effects of the dimensions of the inlet parts of the capillaries on the readings of the UITs-1 rheometer with the suspensions. It was found that the dimensions of the inlet section did not exceed those for viscous liquids or cement suspensions immediately after preparation, when such a suspension can be considered as a two-phase medium. As time passed, i.e., as thixotropic and coagulation effects developed, the relative size of the inlet section began to increase substantially  $(l/d \ge 200)$ , which is due not only to the development of the boundary layer but also to equilibration in the system, which requires a certain time. It was also found that the relative length of the inlet section increased as the diameter of the capillary was reduced. These phenomena were also detectable for the clay suspensions. The data were processed by Mooney's method and showed that the flow curves were substantially dependent on the gap size and capillary diameter; the separation of the curves into families was considered as indicating wall effects. This occurred in about 70% of the experiments. The data showed that the contribution from wall effects increased as the diameter of the capillary fell or as the gap in the rotation system was reduced, and the over-all effect may be as much as 200%. There was a relative fall in the significance of the wall effects as the shear rates were increased. The flow curves after correction for the wall effect at  $\dot{\gamma} \ge 30~{\rm sec^{-1}}$  became linear for the highly fluid cement suspensions, whereas the nonlinearity persisted for the suspensions of lower fluidity. Mooney's method was used to estimate the wall effects and to correct for the inlet and sedimentation effects; this gave flow curves invariant under the dimensions and types of the rheometers with an error of  $\delta \leq \pm 10$  %.

Subsequent processing amounted to fitting the invariant cruves to the various rheological model; each curve was fitted to Bingham Shvedov, Casson Shulman (n = 2, 3, 4, 5), and Ostwald de Waal models, with subsequent evaluation of the adequacy of the model. The rheological characteristics were determined by two independent methods. In the integral method, the data are represented in consistent variables and are fitted to the models in those variables. The differential method involves preliminary smoothing in the consistent variables by means of polynomials, followed by numerical differentiation to obtain sets of  $\tau_i vs \dot{\gamma}_i$  curves, which are fitted to the rheological models in differential form.

Figures 1 and 2 show the typical relative errors in fitting the flow curves for cement suspensions to the various models; Fig. 1a shows the results for Peschanistyi cement with W/C of 0.55 and 0.52 for a grain size s of 0.35 m<sup>2</sup>/g. The Bingham Shvedov model led to relative errors up to 40% for W/C = 0.55 for small values of  $\gamma$ , as against  $\delta$  of 3-5% for  $\dot{\gamma} \ge 260 \text{ sec}^{-1}$ . A similar picture was found with the Ostwald de Waal model. The Casson-Shulman model with n = 2 can reduce the relative error to  $\delta = \pm 4\%$  throughout the range of shear rates. However,  $\delta$  increased with the concentration of the solid phase. For example,  $\delta_{max} \approx 80\%$  for the Bingham-Shvedov model with W/C = 0.52, while  $\delta_{max} \approx 60\%$  for the Ostwald de Waal model. The more nonlinear flow curve required the use of the Casson-Shulman model with n = 3, which gave  $\delta = \pm 5\%$ .

The relative error with the Bingham-Shvedov and Ostwald de Waal models also increased as the grain size was reduced. For example, Bol'shevik cement with W/C = 0.55 gave maximum errors increasing from 20 to 80% and from 15 to 50%, respectively, for s ranging from 0.33 to 0.45 m<sup>2</sup>/g (Fig. 1b). At the finer grain sizes, it was necessary to complicate the Casson-Shulman model, particularlyin order to reduce the relative error to the acceptable range of  $\delta = \pm 5-7\%$  for s = 0.33 m<sup>2</sup>/g, which required n = 3, whereas for s = 0.45 m<sup>2</sup>/g this required n = 4.

The proportion of tricalcium aluminate largely determines the structure and kinetics of cement, and the spatial coagulation strength of the suspension increases with the proportion of that compound [6]. It is therefore of interest to compare the errors of approximation for cements of different mineral compositions. Parts b and c of Fig. 1 show that identical W/C and s cause an increase in the relative error when the proportion of tricalcium aluminate increases from 4.6 to 5.3% (for the Bol'shevik and Komsomolets cements), and reduction of this to  $\delta = \pm 5\%$  requires the Casson-Shulman model with n = 5 (Fig. 1c).



Fig. 1. Curves relating the relative error to the mean shear rate for various flow laws. a) Peschanistyi cement solution: 1) Bingham Shvedov model; 2) Casson-Shulman model, n = 2; 3) Casson-Shulman model, n = 3; 4) Ostwald de Waal model. b) Bol'shevik: 1) Bingham Shvedov model; 2) Casson-Shulman model, n = 4; 3) Ostwald de Waal model; 4) Casson-Shulman model, n = 3. c) Komsomolets: 1) Ostwald de Waal model; 2) Bingham Shvedov model; 3) Casson-Shulman model, n = 5.

It is also of interest to examine the effects of chemical treatment of the suspensions, in particular the clay ones, on the rheological behavior; metas was used as the polymer as being a typical representative of the acryl reagents. Figure 2 shows characteristic flow curves for Druzhkovo clay and Sarigyukh bentonite before and after metas treatment. Metas results in nonlinearity in the curves at low velocity gradients, and this nonlinearity increases with the concentration. The error in fitting to the Bingham-Shvedov model also increases. Table 1 gives fitting results for Sarigyukh bentonite treated with metas. The fitting error is least if the Casson-Shulman model is used with n of 3-5, as Fig. 2 shows. Table 1 also indicates that the power-law model does not result in a substantial reduction in the error relative to the Bingham-Shvedov model; both models are equally unsatisfactory. The largest reduction in the mean relative error was obtained from the Casson-Shulman model for clay concentration c of 3-6%. At these concentrations, the Bingham-Shvedov model resulted in average relative errors of 23-24%. There was no marked change in the character of the flow curves on changing the temperature from 20 to 50°C, and the errors from the Bingham-Shvedov and Ostwald de Waal models may be as high as those at t = 20°C (Table 1).

Fisher's F test [7] was used to examine the adequacy of these models in the description of the experimental curves; in the case of Sarigyukh bentonite containing metas, it was found that the Bingham-Shvedov model was adequate in only 30% of the experiments (out of 20), while the Casson-Shulman model with n of 2, 3, 4, and 5 was adequate in 55, 77, 90, and 77% of the cases, respectively. The Ostwald de Waal model was adequate only in 22%. This shows that the Casson-Shulman model gives the best fit with n = 3 and n = 4. This model had no appreci-

Errors of Approximation and Adequacy Evaluations for Rheological Models for Sarigyukh Containing Metas TABLE 1. Rentonite

				D							-									
. %		 	Binch	am-Sl	hvedov					Casso	n-Shulman	pom u	el					Ostwa	ld de	Vaal
		10	່	nodel			n=2			1=3			n=4			n=5			nodel	
Clay	Metas.	ບວນດວ	σ, %	Fe.	eval	σ, %	ц,	eval.	G, %	ц С	eval.	ď, %	E.	eval.	α, %	a z	eval.	a, %	e L	eval.
9,0	20	0,1	11,5	0,18	Adequate	10,4	0, 13	Adequate	10,4	0,14	Adequate	10,0	0,15	Adequate	10,4	0,15	Adequate	11,2	0,21	Adequate
3,0	20	0,5	19,6	5,48	Not ade-	7,8	2,21	Not ade-	5,6	0,88	Adequate	5,7	0,63	Adequate	6,7	1,04	Adequate	11,6	16,07	Not ade- quate
9,0	20	0,5	11,4	2,28	Not ade-	8,0	0, 75	Adequate	7.4	0,66	Adequate	7,4	0,81	Adequate	7,5	0,98	Adequate	9,3	2,39	Not ade-
9,0	50 (	0,1	12,4	0,58	Adequate	0'11	0,40	Adequate	10.6	0,34	Adequate	10,4	0,32	Adequate	10,3	0,31	Adequate	10,0	0,28	Adequate
3,0	20	0,5	21,0	17,05	Not ade-	8,9	5,33	Not ade-	7,5	2,16	Not ade-	7,6	1,79	Adequate	8,1	1,86	Adequate	12,1	42,76	Not ade- quate
3,0	20	0,1	22,8	1,11	Adequate	8,2	0,36	Adequate	9,1	0,30	Adequate	10,4	0,49	Adequate	11,3	0,74	Adequate	17,0	3,80	Not ade- quate
3,0	50	0,1	24,0	10,30	Not ade-	9,2	3,30	Not ade-	7,1	1,61	Adequate	6,8	1,72	Adequate	7,2	2,52	Not ade-	11,7	19,75	Not ade-
9,0	20	0,5	12,8	58,41	Not ade-	6,5	14,06	Not ade-	6,0	10,91	Not ade- quate	6,6	15,35	Not ade- quate	7,1	21,14	Not ade-	10,4	69,43	Not ade-
6,0	35	0,3	20,0	10,41	Not ade-	8,1	2,55	Not ade- quate	8,5	1,40	Adequate	10,0	1,64	Adequate	10,7	2,24	Not ade-	17,3	10,16	Not ade- quate

Note: The tabulated value of F is 2.10.





able advantage over the Bingham-Shvedov model in fitting the curves for Sarigykh bentonite without the addition of metas, whereas the Ostwald de Waal model resulted in 15-18% increase in the mean relative error. Qualitatively similar results were obtained for the metas treatment of Druzhkovo-clay suspensions.

These rheological studies on clay and cement suspensions thus show that the Bingham-Shvedov and Ostwald de Waal models commonly used to describe the rheological behavior of such suspensions in most instances describe the flow curves unsatisfactorily and are often inadequate. The Casson-Shulman model substantially reduces the error of approximation and allows one to give an adequate description of the flow curves. The Casson-Shulman model is therefore to be recommended for describing the rheological behavior of clay and cement suspensions.

## NOTATION

τ, shear stress; γ, shear rate; l, capillary length; d, capillary diameter;  $\delta$ , rms deviation; s, particle size; c, solid phase concentration; n, nonlinearity index in the Casson-Shulman model.

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