

## FLOW PROPERTIES OF CHINA CLAYS IN AQUEOUS SUSPENSIONS

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### ABSTRACT

Aqueous suspensions of China clays of different origin were studied to observe the effect of mineralogy, crystallinity on the flow properties. Face-to-face aggregates and their face-to-edge flocs were observed by micrographs and rotational viscometry. The yield stress of suspensions is proportional to the  $n$ th power of the solid content, the value of  $n$  depends on the mineralogy. Positiv and negativ hysteresis of up-curves and down-curves are shown. Suspensions containing kaolinite and illite show positiv hysteresis, in the presence of montmorillonite negativ or so called thixotrop hysteresis are formed.

### INTRODUCTION

China clay is usually prepared for fine ceramic bodies by wet method in which the material is dispersed with water and mixed with feldspar and quartz dispersion. The mixture is then filter-pressed or spray dried for removal of the excess of water. It is desirable that the solid concentration of the clay slip should be high enough to avoid segregation effect arising from the settling out of the coarse particles. This means a minimum limit of the viscosity, the maximum is given by the technological processes. The flow properties are reported here with results on the aqueous suspensions of china clays to show relationship between viscosity and concentration depending on the mineralogy.

### EXPERIMENTAL PROCEDURES

#### *Materials and their characterization*

The quantitative mineralogical analysis of different industrial kaolins was made by X-ray method using a Rigaku Denki D 3F X-ray apparatus. IR-spectra were obtained using a Zeiss UR—10 spectrophotometer. The crystallinity index of the kaolinites was calculated from the IR-spectra by taking the ratio of the absorbances at 3630 and 3705  $\text{cm}^{-1}$ . Grain size was measured by using an Andreasen pipette, the surface was characterized by methylene blue adsorption (Table 1).

#### *Equipment and experimental techniques*

The clay suspensions were prepared overnight using 50 g of clay in distilled water in a laboratory mill. After mixing 30 minutes the  $D-\tau$  flow curves were obtained using rotational viscometer type Rheotest RV. Shearing stress  $\tau$  (Pa) belonging to

TABLE 1

*Mineralogical composition, grain size and crystallinity data of materials*

| China clays          | Mineralogical composition (%) |        |                 |        |                 | grains under 5 micron | methylene blue surface m <sup>2</sup> /g | IR spectra        |                                      |
|----------------------|-------------------------------|--------|-----------------|--------|-----------------|-----------------------|--|-------------------|--------------------------------------|
|                      | kaolinite                     | illite | montmorillonite | quartz | X-ray amorphous |                       |  | E <sub>3700</sub> | Ratio of $\frac{E_{3630}}{E_{3700}}$ |
| Standard             | 67                            | 6      | —               | —      | 27              | 83                    | 16                                       | 1,05              | 0,48                                 |
| SPS                  | 80                            | 5      | —               | —      | 15              | 93                    | 15                                       | 1,13              | 0,46                                 |
| Sedlec (Zettlitz)    | 76                            | 5      | —               | —      | 19              | 86                    | 19                                       | 0,96              | 0,47                                 |
| Grolleg              | 72                            | 7      | —               | —      | 21              | 69                    | 21                                       | 0,90              | 0,46                                 |
| Osmose               | 73                            | 4      | —               | —      | 23              | 67                    | 22                                       | 0,91              | 0,48                                 |
| OKA                  | 63                            | —      | —               | 11     | 26              | 82                    | 20                                       | 0,88              | 0,54                                 |
| RÖKA                 | 72                            | 8      | —               | 7      | 11              | 69                    | 20                                       | 0,79              | 0,55                                 |
| BZ                   | 32                            | 2      | —               | 28     | 38              | 68                    | 14                                       | 0,55              | 0,57                                 |
| Pomeisl              | 85                            | 4      | —               | 11     | —               | 71                    | 37                                       | 0,52              | 0,57                                 |
| Kaolinovo            | 69                            | 4      | —               | 11     | 16              | 68                    | 18                                       | 1,18              | 0,49                                 |
| MEKA                 | 56                            | —      | —               | 23     | 21              | 62                    | 18                                       | 0,72              | 0,52                                 |
| Sárisáp              | 75                            | 6      | —               | 18     | 1               | 61                    | 15                                       | 0,64              | 0,54                                 |
| Caminau              | 66                            | 5      | —               | —      | 29              | 60                    | 17                                       | 0,74              | 0,59                                 |
| Rátka                | 15                            | —      | 25              | 30     | 35              | 69                    | 103                                      | 0,26              | 0,60                                 |
| Illites              | —                             | 39     | —               | —      | 61              | 73                    | 69                                       | —                 | —                                    |
| Füzérvány I-1<br>I-6 | —                             | 20     | —               | 10     | 70              | 54                    | 56                                       | —                 | —                                    |

the increasing and decreasing shearing rates  $D(s^{-1})$  were determined. For characterizing flow properties the viscosity  $\eta = \frac{\tau}{D}$  and the slope of the curves  $m$ , calculated by the following formula:  $m = \log D_1 - \log D_2 / \log \tau_1 - \log \tau_2$ .

## RESULTS

The micrographs show that no individual particles but aggregates are present in the aqueous suspension of china clays (Figs 1 and 2). Dispersion to a certain extent is taking place, but the primary structure being built closely together is always maintained along the faces (Fig. 2). In these aqueous suspensions the edges and

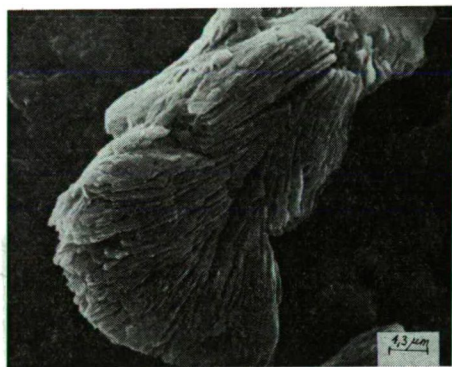


Fig. 1. Aggregate in the Sedlec (Zettlitz) kaolin-water suspension



Fig. 2. Particles in the aqueous suspension of Sedlec (Zettlitz) and OKA kaolins

faces will mutually attract giving rise to a so-called "edge-to-face" flocculation. The typical flow curves of these natural flocculated clays show that different minimum stress has to be applied before these systems begin to flow (Figs 2 and 3). This yield stress is proportional to the  $n$ th power of the solid concentration were the value of  $n$  is depending on the mineralogical composition of materials (Table 2, Fig. 4). Between the up-curves and down-curves a hysteresis loop may be observed. To avoid the misunderstanding we call positiv hysteresis in this case when the shearing stress — and viscosity — in the descending branch are the higher. In general china clay sus-

TABLE 2

Flow properties of Sedlec (Zettlitz) kaolin suspension as a function of solid concentration

| Solid conc. vol. % | Yield point Pa | Viscosity mPas $D = 243 s^{-1}$ |      | m    |
|--------------------|----------------|---------------------------------|------|------|
|                    |                | up                              | down |      |
| 14                 | 10             | 125                             | 130  | 5,38 |
| 17                 | 21             | 221                             | 226  | 5,74 |
| 20                 | 50             | 408                             | 433  | 7,58 |
| 24                 | 75             | 744                             | 744  | 6,17 |
| 28                 | 140            | 1562                            | 1586 | 5,26 |
| 31                 | 150            | 3100                            | 3200 | 3,50 |

pensions have positiv hysteresis (Table 3.). The width of the hysteresis loop obtained has been considered to be a measure of the degree of dispersity under given circumstances. These irreversible changes associated with measuring viscosity by rotational viscometer are caused by a progressive and irreversible breakdown of aggregated clay

TABLE 3

Flow properties of kaolin-water suspensions

| Kaolin            | Solid conc.<br>Vol. % | Yield point<br>Pa | Viscosity mPas<br>$D=243\text{ s}^{-1}$ |      | $m_I$ | $m_{II}$ |
|-------------------|-----------------------|-------------------|---|------|-------|----------|
|                   |                       |                   | up                                      | down |       |          |
| Standard          | 24                    | 150               | 1033                                    | 985  | 6,9   | 3,2      |
| China clay        | 24                    | 95                | 841                                     | 841  | 7,1   | 3,1      |
| Sedlec (Zettlitz) | 24                    | 75                | 744                                     | 744  | 6,2   | —        |
| Grolleg           | 24                    | 62                | 504                                     | 504  | 8,3   | 4,1      |
| Osmose            | 27                    | 80                | 350                                     | 560  | 6,2   | 4,2      |
| OKA               | 27                    | 100               | 1466                                    | 1560 | 4,9   | 1,8      |
| RÖKA              | 27                    | 70                | 913                                     | 937  | 5,1   | —        |
| BZ                | 27                    | 70                | 1490                                    | 1514 | 4,9   | —        |
| Pomeisl           | 27                    | 40                | 528                                     | 600  | 6,9   | 3,3      |
| Kaolinovo         | 27                    | 72                | 648                                     | 648  | 5,1   | 1,6      |
| MEKA              | 27                    | 50                | 860                                     | 860  | 4,4   | 1,6      |
| Sárisáp           | 30                    | 23                | 327                                     | 432  | 3,1   | 2,3      |
| Caminau           | 34                    | 58                | 793                                     | 744  | 3,9   | 2,5      |
| Rátka             | 27                    | 25                | 327                                     | 308  | 2,5   | 3,2      |
| Illites           |                       |                   |   |      |       |          |
| Fűzéradvány       |                       |                   |   |      |       |          |
| I-1               | 20                    | 90                | 841                                     | 937  | 5,4   | —        |
| I-6               | 20                    | 240               | 2211                                    | 2320 | 7,3   | 13,9     |

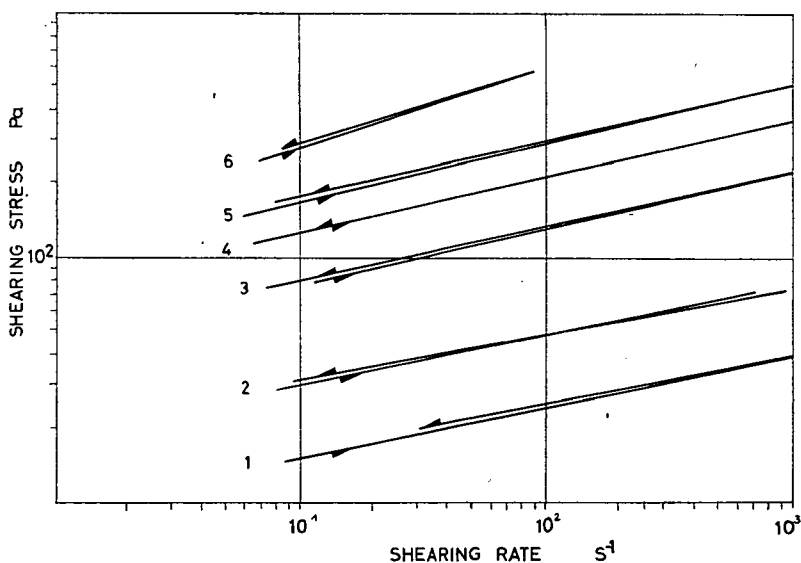


Fig. 3. Effect of dilution on the flow curves of Sedlec (Zettlitz) kaolin. Solid concentrations by vol. %: 1—14; 2—17; 3—20; 4—24; 5—28; 6—31

particles assisted by shearing. The slope of the curves is depending on the concentration (Table 2) and the composition of clays (Table 3). Two slopes of the flow curves ( $m_I, m_{II}$ ) may be distinguished in general. The shear rate belonging to the inflexion is different in case of material investigated, and increases with increasing concentra-

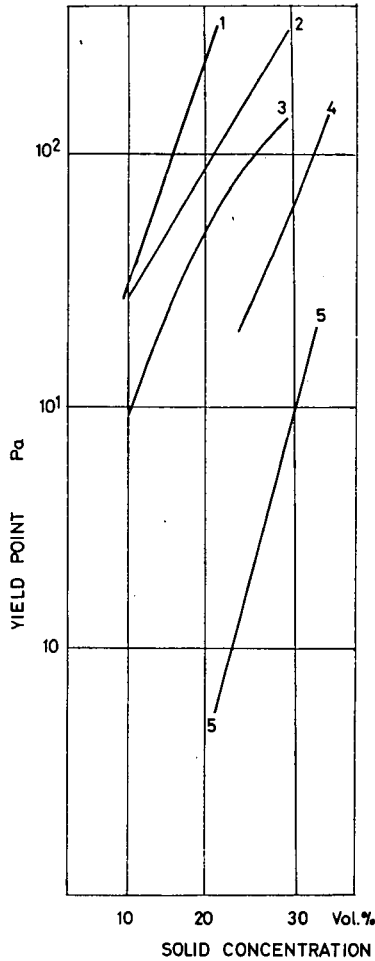


Fig. 4. The dependence of yield value from solid concentration (1- I-6, illite; 2- I-1 illite; 3- Zettlitz kaolin; 4- Meka kaolin; 5- Rátka kaolin)

tion. This reversible phenomenon shows the presence of attracting, non spherical particles in the suspensions. The yield points, viscosity and the slope values  $m_I$  show a loose comparison with crystallinity (Table 1) and the quantity of clay minerals in the clays (based on X-ray and  $3700\text{ cm}^{-1}$  stretching vibration band of the IR-spectra). The relationship between these values is disturbed by the properties of the surface.

In the presence of montmorillonite (Rátka kaolin) thixotrop hysteresis loop is formed in the aqueous suspensions. The relatively low viscosity and yield stress show peptization to a limited extent.

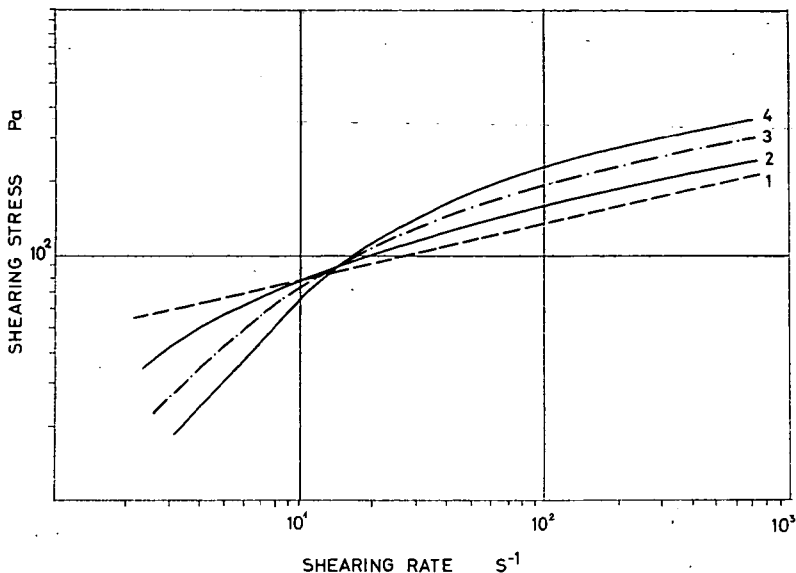


Fig. 5. Flow curve of Sedlec (Zettlitz) kaolin suspension (vol. % 20) as a function of temperature: 1—25 °C; 2—40 °C; 3—60 °C; 4—90 °C

The great part of china clays is contaminated by illite. Investigating the effect of illite on the rheological properties illite suspensions were measured. In case of the two types of the illite of Füzérradvány deposit high degree of dispersity can be observed. The hysteresis is positiv, the water content needed for a given viscosity is very high.

Under the influence of increasing temperature the flow properties of the suspensions are changing in a characteristic manner. From the flow curve of the Sedec-(Zettlitz) kaolin (Fig. 5) it may be seen that at a shearing rate of  $27 \text{ s}^{-1}$  sections with two different slopes have been developed. Within the range of the low shearing rate the viscosity is decreasing with increasing temperatures. At higher shearing rates, however, due to the increased grade of dispersity, increasing viscosity values could be measured. Viscosity of the illite suspension — to 60 °C — was hardly changing in dependence of the increased temperature.

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