

# ANALYSIS IN A LIQUID SEDIMENTOGRAPH OF THE DISPERSION OF SUSPENSIONS OF VITREOUS SILICA

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Since the Podolsk Refractory Products Plant has now mastered the production of immersible quartz containers by slip casting it has become necessary to organize the large-scale monitoring of the dispersion of the suspensions since this factor determines the constancy of the rheological and sedimentation properties of the slip and also the physicochemical properties and the proportion of rejected articles after casting and firing [1, 2]. The recommended method of determining the composition of the powders using a sedimentation pipette has several disadvantages; the most significant of these is the long time required for the analysis.

In the present article we give some results in the development of a method of analyzing the dispersion of suspensions made from vitreous silica using a liquid sedimentograph designed by the Semibratovsk Branch of the All-Union Scientific-Research Institute of the Gas Industry. The grain composition is determined in the liquid sedimentograph using a method in which the deposit is accumulated and its amount continually recorded by a sensitive magnetoelectric balance. The theoretical basis and the principle involved in accumulating the deposit have been described in detail in [4].

A diagram of the liquid sedimentograph is given in Fig. 1.

The particles of powder from the suspension poured into the container 1 are deposited in the pan 2 which is suspended at one end of the beam of an ADV-200 analytical balance. The particles deposited from the suspension unbalance the scales and the magnetic rod 4 suspended at the opposite end of the beam is drawn out of a solenoid 5. At the same time, the pointer of the balance with a shutter 6 made of black paper attached to its end is displaced. The shutter is displaced and intersects the light beam from the lamp 7 which illuminates the mirror 8 and then falls onto the photoresistor 9.

Until the photoresistor is illuminated there is no current in the solenoid circuit and the balance is in the zero position. When the particles are deposited on the pan, the balance moves out of the zero position, the shutter is opened, transmitting the light, and thus the resistance of the light cell (photoresistor) is lowered. As a result, current flows through the circuit and there is a voltage across the resistor 10 which is recorded by the electronic potentiometer 11. When the current flows through the solenoid circuit, the core is drawn in and the scales return to the near-zero position. The force drawing in the core is equal to the mass of the deposited particles.

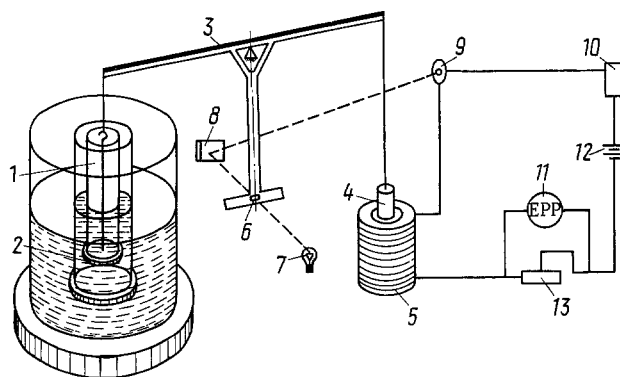


Fig. 1. Diagram of the liquid sedimentograph: 12) stabilized power supply; 13) resistance; remainder of symbols are explained in the text.

Podolsk Refractory Products Plant. Translated from *Ogneupory*, No. 12, pp. 45-48, December, 1980.

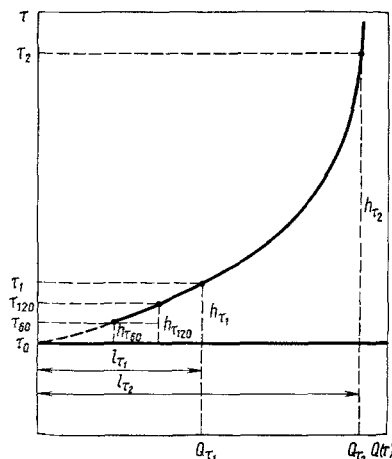


Fig. 2. Accumulation curve of deposit obtained in the liquid sedimentograph:  $\tau$ , time taken for accumulation of deposit, min;  $Q(\tau)$ , relative mass fraction of the deposit in a time  $\tau$ , %;  $\tau_0$ , starting time of accumulation of deposit;  $\tau_{60}$ ,  $\tau_{120}$ , the fixed times on the deposit accumulation curve 60 and 120 sec, respectively, after the stirring of the suspension in the cylinder had stopped, sec;  $h_{60}$ ,  $h_{120}$ , the distance on the deposit-accumulation curve, respectively, after the fixed times of 60 and 120 sec, cm;  $\tau_1$ ,  $\tau_2$ , the deposit-accumulation times for  $Q_{\tau_1} = 20-30\%$  and  $Q_{\tau_2} = 70-90\%$ , min;  $h_{\tau_1}$ ,  $h_{\tau_2}$ , the distance on the deposit-accumulation curve corresponding to  $\tau_1$  and  $\tau_2$ , allowing for the initial time  $\tau_0$ , cm;  $l_{\tau_1}$  and  $l_{\tau_2}$  are the distances on the deposit-accumulation curve corresponding to  $Q_{\tau_1}$  and  $Q_{\tau_2}$ , cm.

The circuit is so arranged that the mass of the continually deposited particles is equal to the magneto-electric force of the solenoid whose value is recorded on the potentiometer strip in the form of a deposit-accumulation curve. The choice of the analysis conditions and preparation of the sample have been discussed in [4], taking into account possible errors.

We used distilled water as the dispersion medium for the analysis. In the region of pH 6-7, no coagulation of the suspension was found. The mass of the sample of the experimental suspension was calculated, allowing for moisture content, from the following formula:

$$P_{ad} = \frac{\rho_s P_d}{\rho_s - \rho_l},$$

where  $P_{ad}$  is the mass fraction of air-dried material in the suspension, mg;  $\rho_s$ , density of the solid phase of the suspension,  $g/cm^3$ ;  $P_d$ , maximum mass fraction of deposit in the bowl (calibration of device), mg; and  $\rho_l$  is the density of the dispersion medium,  $g/cm^3$ .

A portion of the suspension (1.5-2.0 g) was dispersed in 1 liter of distilled water. For the analysis we took 40 ml of the prepared suspension of concentration 1.0-1.5 g/liter.

The analysis included the following operations. Before the beginning of the analysis, distilled water (Fig. 1) was poured into the cylinder to a height of 15-16 cm, the same as that used for the particle deposition. The pan was suspended on the beam. After stabilizing the apparatus, the scales were balanced with small weights and the scale of the potentiometer was calibrated by loading the arm of the beam with 10 mg weights. The maximum deflection on the potentiometer scale was 30 mg. The pan, with the balance switched off, was tightly attached by means of pin to the lower end of the cylinder, from which the water was partially drawn off by a hose.

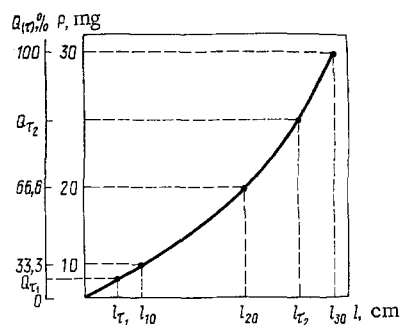


Fig. 3

Fig. 3. Graph of the nonlinearity of the instrument:  $l$  is the distance on the recorder strip corresponding to a load of 10, 20, 30 mg ( $l_{10}$ ,  $l_{20}$ ,  $l_{30}$ ).

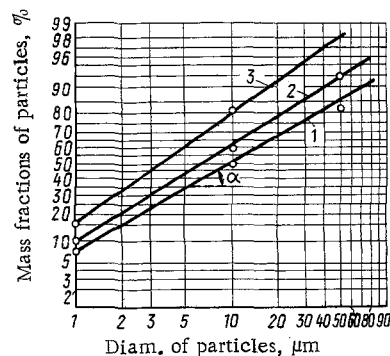


Fig. 4

Fig. 4. Size distribution of particles in a vitreous silica suspension: 1) transparent; 2) opaque; 3) from waste suspension after casting.

The prepared suspension was poured into the cylinder, water added to the 15 cm mark, and then the suspension was stirred with a stirrer. The pan was detached from the end of the cylinder and the balance switched on. The deposition of the particles was recorded 60 and 120 sec after the stirring of the suspension in the cylinder had been stopped. From the moment the balance was switched on, the recording device registered the mass of deposit in the form of an accumulation curve (Fig. 2).

The accumulation curve of the mass of precipitate recorded on the potentiometer strip can be treated using the normal methods for calculating the sedimentation curves, namely, graphical differentiation or analysis. The analytical method gives more accurate results and also makes it possible to construct the distribution curve over a wide range of particle sizes, assuming that the size distribution of the particles is described by some dependence. In this particular case we used the formula proposed by Avdeev [4, pp. 150-152]. Below we give the sequence for the calculation of the sedimentation curve.

A graph of the nonlinearity of the instrument is constructed (Fig. 3). On the deposit accumulation curve we chose values of  $\tau_1$  and  $\tau_2$  on either side of the greatest curvature (normally  $\tau_1$  is taken at  $Q_{\tau_1} = 20-30\%$  and  $\tau_2$  at  $Q_{\tau_2} = 70-90\%$ ).

Because the initial time  $\tau_0$  of the accumulation of precipitate was not recorded, the accurate values of  $\tau_1$  and  $\tau_2$  are calculated on the basis of the rate of movement of the recorder strip and of the points on the deposit-accumulation curve which correspond to times of 60 and 120 sec from the moment when stirring of the suspension in the cylinder stopped, in accordance with the formulas

$$\tau_1 = \frac{h_{\tau_1}}{h_{\tau_0}}, \quad \tau_2 = \frac{2h_{\tau_2}}{h_{\tau_{120}}}$$

From the values found for  $l_{\tau_1}$  and  $l_{\tau_2}$ , we find  $Q_{\tau_1}$  and  $Q_{\tau_2}$  from the nonlinearity graph.

According to the tabular values for the time functions of sedimentation [5, 6] calculated from the formula  $Q(\tau) = 100(1 - e^{-1/X})X$ , we find the corresponding values of  $Q_{\tau_1}$  and  $Q_{\tau_2}$  and the values of  $X_1$  and  $X_2$ , where  $X$  is a function of the sedimentation time, equal to  $\mu(\tau - \Delta\tau)$ .

From the equations  $\beta = [\log(X_2/X_1)/\log(\tau_2/\tau_1)]$  and  $\mu_0 = X_1\tau_1^{-\beta} = X_2\tau_2^{-\beta}$ , we calculate the parameters  $\beta$  and  $\mu_0$ .

Using the equation  $d = \sqrt{18H\eta/(\rho_s - \rho_l)60\tau}$  (where  $d$  is the diameter of the particles,  $\mu\text{m}$ ;  $H$ , height of the precipitate,  $\text{cm}$ ;  $\eta$ , viscosity of water,  $\text{Pa}\cdot\text{sec}$ ) we determine the precipitation time set by the different values of the particle diameter (1, 2, 3, 5, 10, 20, 30, 40, 50, and 60  $\mu\text{m}$ ). From the values found for  $\tau_1$  we determine the values of  $X_1$  using the formula  $X_1 = \mu_0\tau_1^{\beta}$ . Starting with  $\beta$  and  $X_1$  and using the tables for the values of accumulative distribution curve  $g(\tau) = [1 - \beta - (\beta/X)]Q(\tau) + 100\beta$ , we find the relative concentration of particles of diameter greater (or less) than the specified and construct the distribution curve for the particles according to their size (Fig. 4).

The results of the analysis of the disperseness of the suspensions are plotted on a logarithmic probability graph (Fig. 4). We determine the tangent of the angle  $\alpha$  characterizing the slope that the straight line makes with the abscissa (width of grain component) and the average-surface diameter of the particles  $d_{AVS}$  using the formula  $d_{AVS} = 100 / (\sum \Delta Q / d_{AV})$ , where  $\Delta Q$  is the mass fraction of the particles of a certain cut with an average particle size  $d_{AV}$ , %.

The statistical treatment of the results of the analysis shows that the relative error in the determination of the average-surface diameter of the particles is  $\leq 12\%$ .

The main error is caused by the degree of uniformity both in relation to the grain composition and also in relation to the concentration of solid phase per unit volume in the case of the analysis of the suspension of concentration 1.0-1.5 g/liter.

## CONCLUSIONS

A method of determining the grain composition of suspensions of vitreous silica using a liquid sedimentograph has been developed and introduced at the Central Plant Laboratory of the Podolsk Refractory Products Plant. The results of the analysis can be evaluated from the tangent of the angle the straight line makes with the abscissa on the log-probability graph and the average surface diameter of the particles. The relative error in the determination of the average-surface diameter is  $\leq 12\%$ .

Using this method it is possible to determine the disperseness of the suspensions made from  $Al_2O_3$  and other materials characterized by a grain composition between 0.5 and 100  $\mu m$ .

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## KINETICS OF FORMATION OF SPINELS IN THE $MgO-FeO(Fe_2O_3)-Al_2O_3-Cr_2O_3$ SYSTEM

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The calculation of the kinetics of formation of spinels in the  $MgO-FeO(Fe_2O_3)-Al_2O_3-Cr_2O_3$  system is complicated by several difficulties one of which is the determination of the amount of newly formed product. The task can be simplified when one of the initial materials is in the form of smooth grains on whose surface the complex spinel is formed. The general composition of a spinel of this particular system can be expressed by the formula  $(Mg, Fe)O \cdot (Cr, Al, Fe)_2O_3$ . The reflection coefficients of polished slices of corundum and spinel differ significantly (Fig. 1) and this makes it possible to give a quantitative estimate of the rate of formation of a spinel from the thickness of its layer on the surface of a corundum grain.

As the starting materials for the production of the spinel we used electrocorundum, grade EB-50 (State Standard 3647-71); ferrous oxide (analytically pure grade); and a mixture of  $MgO + MgCr_2O_4$  containing, %:  $Cr_2O_3$ , 65.72;  $MgO$ , 33.71;  $Al_2O_3$ , 0.01;  $Fe_2O_3$ , 0.02;  $CaO$ , 0.42;  $SiO_2$ , 0.04; and  $SO_4$ , 0.06. The  $MgO + MgCr_2O_4$

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