

AUTOMATIC DEVICE FOR RECORDING AND EXPLORING THE SETTLING CURVES. APPLICATIONS AT THE ASYMPTOTIC CLASSIFYING OF POWDERY SYSTEMS

LIVIU LITERAT

*Department of Chemical Engineering, Faculty of Chemistry and Chemical Engineering,
Babes-Bolyai University, 11, Arany Janos, 3400, Cluj-Napoca*

SUMMARY. An experimental fitting of original conception, with possible applications to the study of the powdery dispersions depot in liquid media, is presented. The outcoming results are compared to those of other techniques specialized in the same purpose.

The tight correspondence between the data and the results offer credibility to the procedure and recommends it as an accessible constructively and economically alternative.

INTRODUCTION

Considering that the principles of settling analysis methods are already known, we aim at presenting an experimental fitting for the recording of the sedimentation curves. We are also interested in a program that calculates the distribution of fractions in a powdery ensemble dispersed in an immobile liquid [1-3].

The experiment data and the interpretation can be considered as a case study meaning to explicitate the working technique and to underline the performances of the procedure.

EXPERIMENTAL FITTING

The installation is a sequential fitting consisting of a sedimentation vessel (fig.1.), an electronic analytic scales and a computer (PC Pentium) interconnected through an interface RS-232C. A program of data acquisition (LabVIEW, National Instruments Co.) records the variation of the mass (m) in time (t) on the sedimentation scales (1, fig.1.) [4].

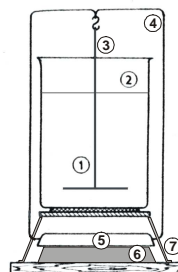


Fig.1. Sedimentation vessel
1. plate to deposit, 2. suspension,
3. hanging wire, 4. frame, 5. scale
support, 6. sensor, 7. support of
the sedimentation vessel

EXPERIMENTAL DATA. INTERPRETATION. STUDY CASE.

In order to test the performances of the installation, several sedimentation curves for watery suspensions of bentonite and alumina powder in different concentrations (below 1%), have been recorded.

The sample preparation and the recording conditions have been strictly controlled in order to avoid any kind of disturbances.

The sedimentation kinetics is recorded as dependence function

$$m = f(t) \quad (1)$$

The sedimentation curves for bentonite and alumina are presented in figure 2.

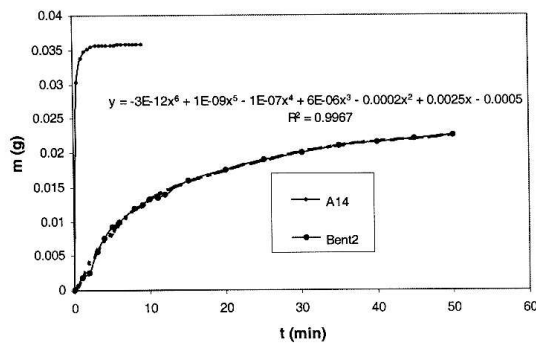


Fig 2. Settling curves: bentonite (•), alumina (•)

Table 1.
The coefficients of the polynomial equations (MATLAB); Alumina A₁₄

Rank	Coefficient	Rank	Coefficient
12	$-3.50 \cdot 10^{-9}$	5	$7.91 \cdot 10^{-2}$
11	$2.04 \cdot 10^{-7}$	4	$-1.76 \cdot 10^{-1}$
10	$-5.29 \cdot 10^{-6}$	3	$2.61 \cdot 10^{-1}$
9	$8.01 \cdot 10^{-5}$	2	$-2.47 \cdot 10^{-1}$
8	$-7.87 \cdot 10^{-4}$	1	$1.36 \cdot 10^{-1}$
7	$5.26 \cdot 10^{-3}$	0	$7.2 \cdot 10^{-7}$
6	$-2.44 \cdot 10^{-2}$		

In the first one, the most probable curve is traced and the adequate definition function is written in the graph. The sixth degree equation (set in Excel) and the determination coefficient ($R^2=0.99967$) are eloquent for the accuracy of the recording [6].

For alumina, the graphic corresponds with a twelfth equation, its coefficients being presented in table 1 (MATLAB) [7].

The fashion of the curves underlines the method's capacity to discriminate the behavior of the two powders during the sedimentation process; the bentonite is softly and highly dispersed compared to the alumina, which is much more roughly.

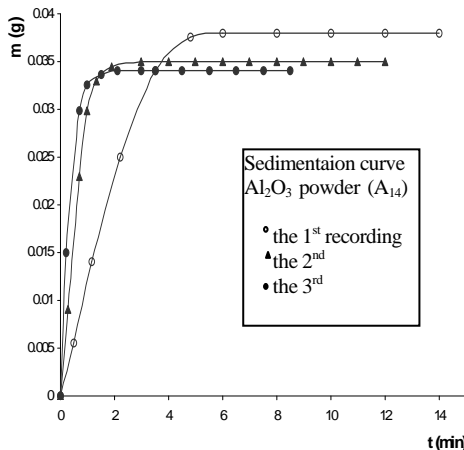


Fig. 3. Sedimentation curves registered after rehomogenisation of the same suspension (alumina)

Another idea that needed checking was to see in what measure, in the same system, after the end of the phase separation process, a remixing of the phases determine the reproduction of the curves at repeat recordings. A problem is faced in situation when rerun of the operation is wanted, and without stopping the recording, after every determination, the dispersion rehomogenizes.

The answer can be found in fig.3. where we can notice in the sedimentation curves a sensitive change of the fashion and laying out, during three successive recordings.

The causes of the changes can be an incomplete mixing of the depot phase, which equals to a modification of the suspension's concentration, a crowding of particles or a solid phase scattering. We attribute the evolution in this case to the first working hypothesis, the final level of the systematically decreasing curves representing proof to this case.

THE EVALUATION OF THE SIZE OF THE PARTICLES.

The sedimentation curves express unmediated correlations between the size (d) and the movement speed (w_0) of the particles during the sedimentation process [2,5].

In the gravitational field (g) the relation $w \sim d$ is [2]

$$w_0 = \left(\frac{4}{3} \frac{d \Delta \rho}{\rho} \frac{g}{\xi} \right)^{\frac{1}{2}} \quad (2)$$

where: $\Delta \rho = \rho_s - \rho$, expresses the difference of the disperse (solid) phase's density (ρ_s) and the dispersing medium (ρ)

$\Delta \rho g$ - represents the driving force of the process

ξ - the strength coefficient (the brake factor of the medium to the movement of the particle) with values dependent on the speed in which the particle moves (the hydrodynamic regime Re) from the shape (Φ) and the roughness of the surface (ε)

$$\xi = f(Re, \Phi, \varepsilon) \quad (3)$$

The movement regime $Re = w_0 d \rho / \eta$ (η - the dynamic viscosity of the medium) discriminate 3 hydrodynamic sedimentation domains [2,5,8]

laminar (Stokes)	$Re < 1$	$\xi = 24 / Re$
intermediate (Allen)	$1 < Re < 10^3$	$\xi = 18.5 / Re^{0.6}$
turbulent (Newton)	$10^3 < Re < 10^5$	$\xi = 0.44$

The speed sedimentation equations corresponding to:

$$w_{0s} = \frac{1}{18} \frac{\Delta \rho}{\eta} g d^2 = k_s d^2 \quad \text{Stokes domain} \quad (4)$$

$$w_{0A} = 0.15 \frac{(g \Delta \rho)^{0.72}}{\eta^{0.42} \rho^{0.29}} d^{1.14} = k_A d^{1.14} \quad \text{Allen domain} \quad (5)$$

$$w_{0N} = 1.74 \left(\frac{\Delta \rho}{\rho} g d \right)^{\frac{1}{2}} = k_N d^{\frac{1}{2}} \quad \text{Newton domain} \quad (6)$$

The equations (4)-(6) rewritten in a more general manner

$$w_0 = kd^x \quad (7)$$

or after logarithmation

$$\ln w_0 = \ln k + x \ln d \quad (8)$$

allow the setting of the sedimentation regime through the values of the x (slope) and k (origin ordinate) parameters from experimental data respectively the selection of the calculus relation $w_0 \sim d$ [8].

THE DISTRIBUTION FUNCTION CALCULATION

A distribution function

$$F(d) = \Delta m / \Delta d = f(d) \quad (9)$$

represents the portion (mass) of the particles from a narrow dimension domain (fraction) corresponding to a certain value of the particle size (diameter) d .

In the fig.4. the sedimentation (1) and distribution (9) curves of bentonite are recorded on the same common graph.

For the same bentonite sample, the distribution function are registered by the Mie method (laser light dispersion) in fig. 5 [1].

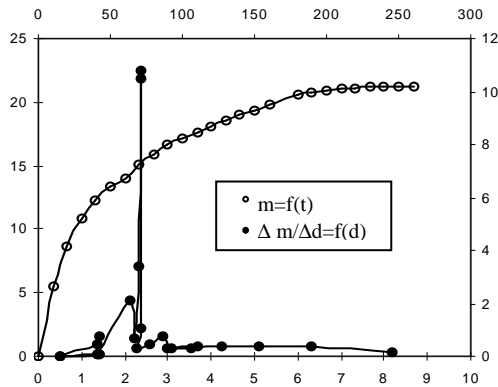


Fig.4. Sedimentation curve and distribution function for bentonite

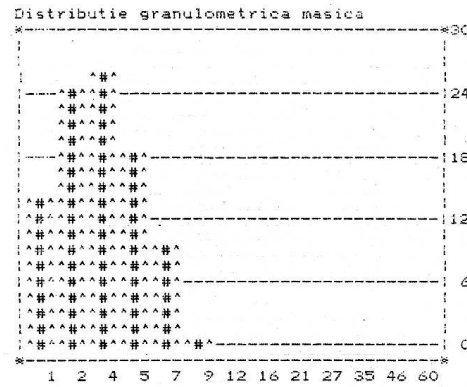


Fig.5. The distribution histogram of fractions in bentonite (laser method)

One can see that the both recording emphasize an unimodal distributin of the powdery fractions with a $2.2 - 2.7 \mu m$ median diameter.

As concern the fractions scattering, the Mie (laser) method [1], more sensitive, shows a spread slightly more extended.

RRS DISTRIBUTION CURVE

With the same experimental data, one also calculated the distribution function in accordance with Rosen-Rammler-Sperling (RRS) method [2,3]

$$R/100 = e^{-(d/d')^n} \quad (10)$$

From the graph (fig.6.) in double logarithmic coordinates (11)

$$\ln \ln 100/R = n \ln d - n \ln d' \quad (11)$$

results a straight extended S shaped line [2]. By the intersection of the linear middle zone with the ordinate $R=36.8\%$ one can read on the abscissa (projection) the characteristic diameter d' of the granular ensemble and from its inclination (slope), the uniformity factor n or the powder scattering. The obtained values are $d = 2.7 \mu m$, and $n=1.4$ in very good accordance with the other methods applied.

CONCLUSIONS

The data synthesis underline the following conclusions.

- The suggested proceeding shows performances comparable with other sophisticated and expensive techniques destined in this domain.
- The recording and calculating fittings are characterized by versatility and allow kinetic studies for different other processes with mass variation.
- The fitting permits, by changing the components (electronic scales) and programming data acquisition, the adapting of sensitivity for different working precisions.
- The constructive and financial accessibility are also important characteristics.

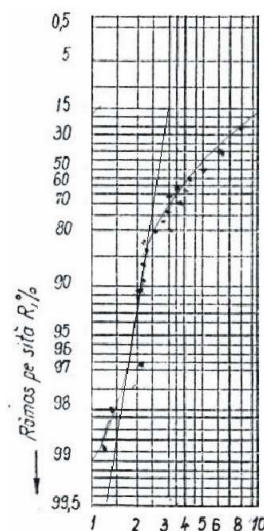


Fig. 6. Rosen-Rammler-Sperling diagram

REFERENCES

- [1]. I.N. Stanford, Particle size analysis in Pharmaceutics. The Science of dosage form design. Editor M.E. Aulton. Editor Churchill Livingstone, Edinburgh-London-Melbourne-New York, chap. 33, p. 564-578, 1988
- [2]. L. Literat, Operații și utilaje în industria materialelor oxidice. Aplicații. Ed. Univ. Babeș-Bolyai, Cluj-Napoca, 1994

LIVIU LITERAT

- [3]. K. Leschonski, Particle characterization. Present State and Possible Future Trends; Preprint, First World Congress Particle technology, Part I, Nurnberg, p.1-16, 1986
- [4]. L. Literat, I. Muresan, P. Muresan, Automatic determination of vapours adsorption isotherms on porous solids. Acta Technica Napocensis, technical University of Cluj-Napoca, Section CMM, 42 p. 139-146, 1999
- [5]. B. Koglin, Untersuchung zur Sedimentationsgeschwindigkeit von Einzelteichen, Freiberg, Forschung sh. A, A 484, p. 35-44, 1971
- [6]. G.E. Jones, Excel 5, Editura ALL Educational S.A., București, 1995
- [7]. Table Curve Program – Jandel Scientific USA
- [8]. L. Literat, Rev. chim. 45, nr. 10 p 921, 1994