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CRITICAL OPERATING PARAMETERS OF THE FLUID GRADING METHOD

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SUMMARY

Optimum conditions were found for the grading of chromatographic supports and packings. The standard deviation of the graded fraction with a diameter of 25 μm was $\pm$ 2.5 μm at the pressure regulation attained. An optimum period of time was suggested for the grading of one fraction as well as for the distribution of a sample into a greater number of narrower fractions. A critical volume of sample was determined so as to enable the device to provide the maximum output.

INTRODUCTION

Homogeneous distribution of the sizes of the grains in chromatographic columns enables the maximum separation efficiency to be obtained[1–5]. The fluid method for the grading of packings[6] gives fractions that are sharply distributed and from which such particles that differ from the selected fraction in their size and density are removed. In this work, we examined the uses of this method for the grading of adsorbents, supports and packings in air, water and some other liquid media. The advantages and disadvantages of this technique and the effects of particle shape on the sharpness of the fraction gradings were studied. The possibility of grading individual fractions that differ from one another by 1–2 μm follows from the analysis of the operating conditions.

In the present work, attention was paid to the optimization of the grading process, mainly to the maximum attainable sharpness of the grading; to the output of the apparatus used (expressed by the amount of graded fraction in grams per unit time); and to the filling capacity of the apparatus (output of the apparatus for various fillings of the material graded).

EXPERIMENTAL

The following materials were used for grading: Corasil I with a diameter of 28 μm; glass beads with a diameter of 50–100 μm (Jablonec Glass Works, N.E.,

Czechoslovakia); and Porasil B, 100–150 mesh (Waters Associates, Framingham, Mass., U.S.A.).

The regulation of the pressure in the apparatus was modified. For damping pressure impacts of the membrane pump (a), a system of vessels (Fig. 1) was used with a volume of 500 ml connected by means of tubes with inserted porous plates serving as resistances (g), and a vessel with a large volume (i) as a capacity link. The final pressure regulation was carried out by adjusting the height of the level of the water column in the pressure regulator (h).

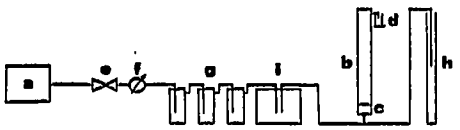

Fig. 1. Scheme of the apparatus. a = Membrane pump; b = grading tube; c = porous plate; d = fraction receiver; e, f = flow regulation elements.

A microscope with a scale served to measure the particle diameters. Twenty particles were always measured, and their average size and standard deviation were calculated.

The maximum obtainable sharpness of the grading was checked by taking fractions at a pressure that was always higher by 10 mm of water. The output of the grading apparatus used was expressed in terms of the weight of the fraction graded per unit time. The grading was carried out at a constant pressure of the flowing liquid and the weight increase of the graded fraction in the collector was determined. The same procedure was carried out with three fillings of various sizes. The height of the layer was determined directly in the tube for five weights of the sample.

RESULTS AND DISCUSSION

The maximum obtainable sharpness of the grading under the described conditions can be seen from Fig. 2. Two fractions were taken, the first of which was graded at the inlet pressure of the grading air (60 mm of water) (Fig. 2b) and the second one was taken at a pressure of 70 mm of water (Fig. 2c). The values in Fig. 2 show that the first fraction consists of particles of various sizes, irregular shapes and various densities. This is shown also by a large variation in the individual values, with a standard deviation of 8.4 μm at a diameter of 21.9 μm. After removing this portion, the second fraction was obtained by increasing the flow-rate of air, which was homogeneous as far as the size, shape and density of the particles are concerned. The diameter of the particles from this fraction was 25 μm, with a standard deviation of 2.5 μm.

In order to evaluate the accuracy of the results from the grading apparatus, a relationship that was derived from the Hagen–Poiseuille equation and from the Stokes relationship was used:

$$d_p^2 = \frac{9\,R^2}{8\,(s-\varrho)\,g\,l} \cdot \frac{P_1^2 - P_2^2}{P^0}$$

where d_p is the diameter of the spherical particle, R is the radius of the grading tube,

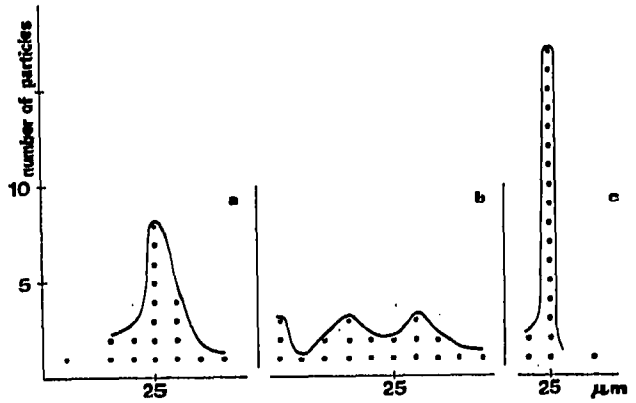

Fig. 2. Distribution of the particles of (a) a non-graded sample, (b) fraction I and (c) fraction II.

s and ϱ are the densities of the particle and medium, respectively, l is the length of the grading tube, g is the acceleration due to gravity and P_1, P_2 and P^0 are the inlet pressure, the outlet pressure and the pressure at which the gas flow-rate is measured, respectively.

The deviation of particle size can be found from the total differential of this function for inlet and outlet pressures:

$$\mathrm{d}\,(d_p) = \sqrt{\frac{9\,R^2}{8\,(s-\varrho)\,g\,l\,P^0}}\,\mathrm{d}P_1 + \sqrt{\frac{9\,R^2}{8\,(s-\varrho)\,g\,l\,P^0}}\,\mathrm{d}P_2$$

depending on the fluctuations of the inlet and outlet pressures, assuming that the remaining parameters remain constant. Changes in the barometric pressure that occur during the time when one fraction is taken can also be neglected with respect to the changes in the inlet pressure, and the dependence can therefore be expressed by the relationship

$$\mathrm{d}\,(d_p) = 1.5 \cdot 10^{-4}\,\mathrm{d}P_1$$

where the values are given for glass beads graded with air in a tube with a diameter of 1.6 cm and a length of 64 cm.

As our results show a standard deviation of 2.5 μm for the grading of Corasil I at a pressure of 70 kp/m^2, our regulation device operates with a 2% fluctuation of the pressure. If the regulated flow-rate fluctuates by only 1%, the grading of regular spherical particles is possible up to fractions with a standard deviation of 1 μm. For particles with irregular shapes and varying densities, the grading is poorer.

The dependence of the grading speed on the sample size and on time can be seen from the values in Table I. It is obvious from Fig. 3 that the weight of the graded fraction increases logarithmically with time and that the increase in the graded fraction in a given interval is higher the larger is the sample dosed into the measuring apparatus. The course of all three curves shows that in order to obtain the maximum amount of a single fraction in the minimum time possible, neglecting some losses, the highest possible dose must be inserted in the apparatus. By this means, a 50% increase in the amount of the graded fraction is obtained in the same time.

TABLE I

TIME DEPENDENCE OF THE GRADED FRACTION ON THE SIZE OF THE SAMPLE

Sample, glass beads; pressure: 260 kp/m².

Height of the layer (cm)	*Weight of the layer (g)*	*Time (min)*	*Weight of the graded fraction (g)*
2	5.46	10	0.0043
		30	0.0104
		90	0.0134
		210	0.0156
		900	0.0161
3	8.19	10	0.0055
		30	0.0124
		90	0.0186
		210	0.0208
		900	0.0238
4	10.92	10	0.0067
		30	0.0128
		90	0.0205
		210	0.0244
		900	0.0327

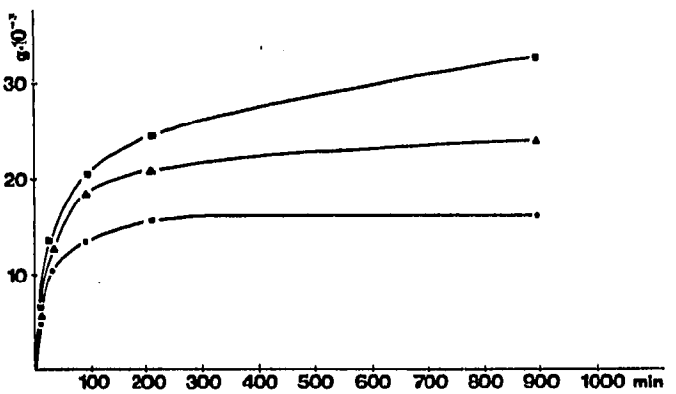

Fig. 3. Time dependence of fraction grading on the size of the sample. Dose: ●, 5 g; ▲, 8 g; ■, 12 g.

If the sample is graded into more fractions or the maximum yield of one fraction is required, it is necessary to put into the apparatus the so-called critical volume of the graded material, v_k. This is the maximum volume of the sample that does not increase the height of the grading layer (see Fig. 4). The critical volume of the graded material was, in the case of the apparatus used, the volume giving a filling height equal to the diameter of the grading tube. A period of 2 h is then sufficient for obtaining 95% of the fraction required, while with twice the volume it is necessary to continue for 13 h in order to obtain an equivalent grading.

The dependence of the height of the grading layer on the volume of the material graded can be seen from Fig. 4. The point of slope change in the graph indicates the value of the critical volume of the grading tube used.

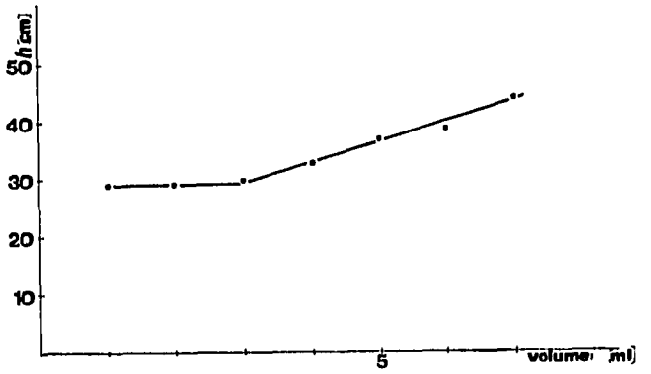

Fig. 4. Dependence of the height of the grading layer on the volume of the sample.

CONCLUSION

It has therefore been proved experimentally that the regulation of the pressure and flow-rate of a fluid medium are the decisive factors in obtaining the best grading by means of the fluid technique. The grading of chromatographic supports and packings into fractions with a standard deviation of 1 μm can be obtained by means of the optimal regulation of the pressure and flow-rate. The optimal time period required for the grading of a given fraction of a particular material can be found and a so-called critical volume of the material exists that permits the maximum output from the grading apparatus.

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