Journal of Chromatography, 238 (1982) 232–236
Elsevier Scientific Publishing Company, Amsterdam — Printed in The Netherlands

CHROM. 14,579

Note

Devices for packing preparative chromatographic columns by "dry-packing" techniques

KRYSTYNA PRUSIEWICZ*, MARIAN KAMIŃSKI and JAN KLAWITER
Institute of Inorganic Chemistry and Technology, Technical University of Gdańsk, 80-952 Gdańsk (Poland)
(Received October 12th, 1981)

Column packings with particle diameters over 25 μm are still widely used in liquid chromatography, especially for preparative purposes, since they are considerably cheaper than the high-performance packings having smaller particles. Packing of columns with packing of such large size is generally carried out by so-called "dry-packing" methods. It follows from previous papers[1–6] that the material used as column packing should be introduced uniformly onto the entire cross-sectional area of the column. Such operations as column vibrating or tapping are frequently automated[3,4,6]. Similarly, feeding the packing to columns during filling can be automated and should considerably improve the reproducibility of filling.

This paper describes devices for filling columns which should be useful in many chromatographic laboratories.

The following column packing techniques were taken into consideration:

(1) tap-fill method, found[6] to be optimal when using a tapping frequency of 120 min^{-1} and column drop from a height of 2 cm

(2) vibration method, optimal at a frequency of *ca.* 1 kHz and acceleration of 5 *g* (ref. 6). Two methods of feeding the packing were investigated.

(1) from the packer shown in Fig. 1a, attached to the column packed by the tap-fill method

(2) from the packer shown in Fig. 1b, undergoing transverse vibrations of frequency 50 Hz, coupled to the column packed vibrationally. Perforated plates (0.1 mm thick) with holes distributed as shown in Fig. 1b or gauzes with various mesh sizes were placed in both cases at the bottom of the packer containers. In order to ensure uniform distribution of silica gel, the perforated plates had seven, three and one hole for the columns of diameter 52, 16.8 and 2–8 mm, respectively.

The following particle fractions of silica gel were used; $\bar{d}$ = 33 μm, 65 μm, 124 μm and 240 μm, where $\bar{d}_p$ is the average particle diameter according to Krumbein[7]. More detailed granulometric characteristics of these packings will be presented elsewhere[6].

The results of investigations of the effect of the hole diameter in perforated plates or the mesh size in gauzes on the packing feeding rate for each method of column filling are shown in Figs. 2–4.

On the basis these results it was found that:

(1) the use of the packing devices shown in Fig. 1 for filling columns by tap-fill

0021-9673/82/0000–0000/$02.75

and vibration methods ensures good reproducibility of the mass feeding rate, particularly for particle fractions with $\bar{d}_p$ = 124 μm and 240 μm.

(2) slightly worse reproducibility was obtained when using packing fractions with $\bar{d}_p$ = 33 μm and 65 μm. This is probably due to the presence in these fractions of particles with diameters less than 15 μm, which are usually introduced by "slurry packing" techniques

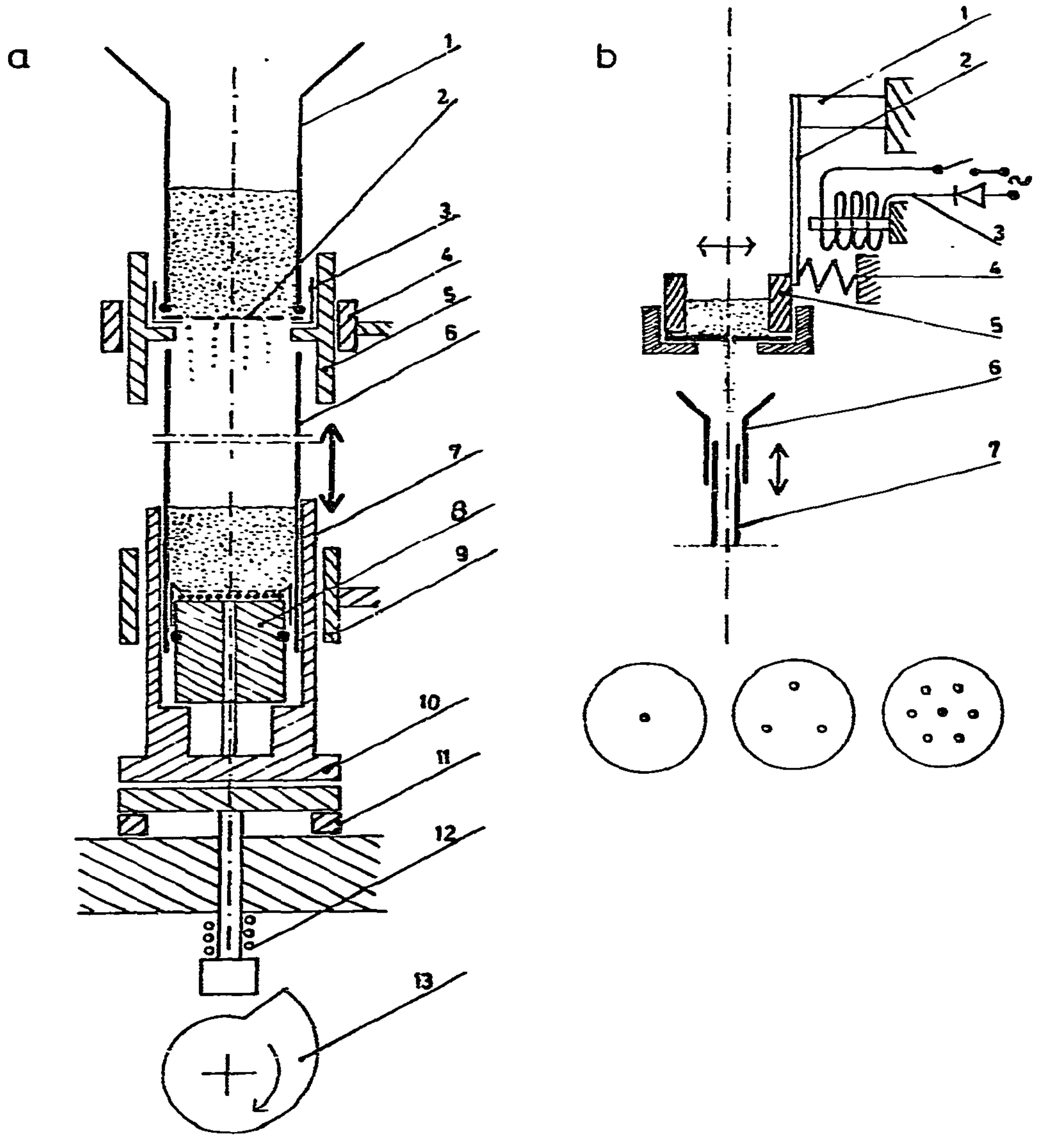

Fig. 1. Schematic diagram of devices for uniform packing of columns by the tap-fill method (a) and the vibration method (b). a, 1 = Container; 2 = perforated plate, 0.1 mm thick; 3 = nut; 4,9 = guiding rings; 5 = coupling; 6 = columns; 7 = nut; 8 = outlet head; 10 = sliding table; 11 = hard rubber washer; 12 = spring; 13 = motor-driven cam. b, 1 = Holder; 2 = elastic plate; 3 = electric circuit 6.3 V a.c. with electromagnet; 4 = spring; 5 = container; 6 = funnel; 7 = column.

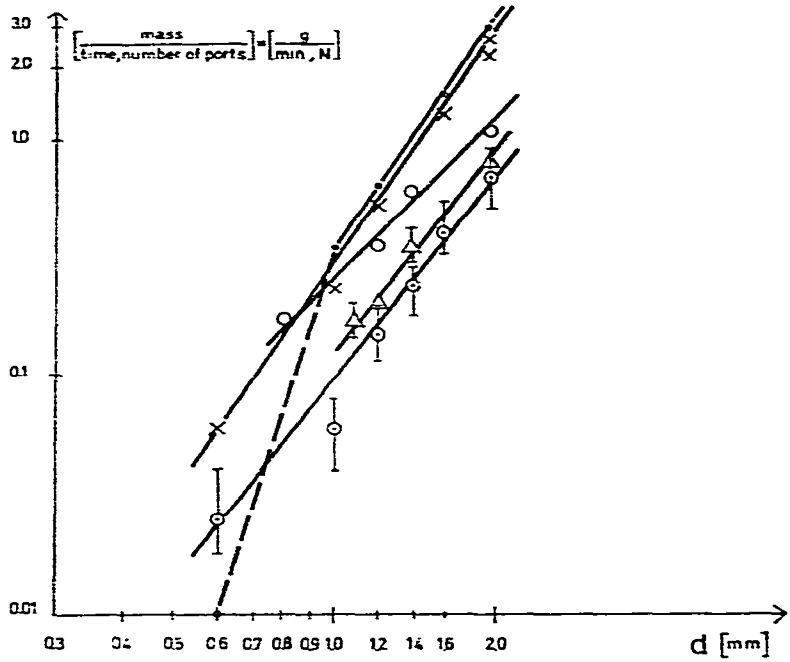

Fig. 2. The effect of hole diameter, *d*, of the perforated plate on the mass feeding rate of silica gel to chromatographic columns filled by the tap-fill method (Fig. 1a). $\bar{d}_p$ = 240 μm (●), 124 μm (×) or 33 μm (⊙) for columns of diameter 2.8 and 17 mm; 33 μm (○) for columns of diameter 52 mm; 65 μm (△).

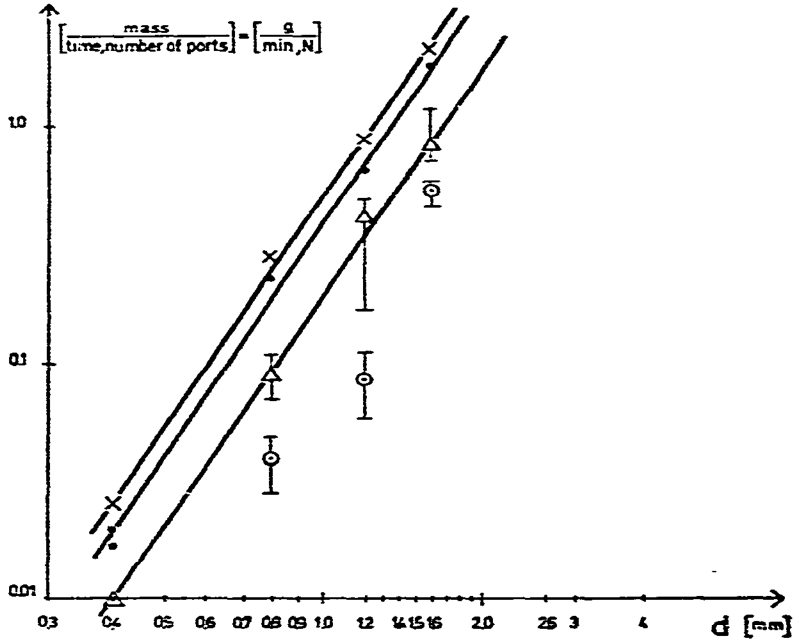

Fig. 3. The effect of hole diameter, *d*, of the perforated plate on the mass feeding rate of silica gel to chromatographic columns filled by the vibration method (Fig. 1b) $\bar{d}_p$ = 240 μm (●), 124 μm (×), 65 μm (△) and 33 μm (⊙).

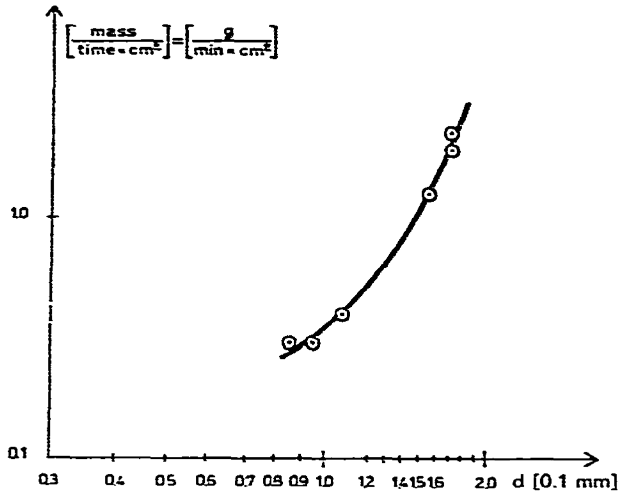

Fig. 4. The effect of gauze mesh size, *d*, on the mass feeding rate of silica gel with $\bar{d}_p$ = 33 μm to chromatographic columns filled by the tap-fill method (Fig. 1a).

(3) the average density of the packing for columns filled by the tap-fill method was 0.56 g/cm^3

(4) in order to achieve favourable filling conditions the height of the bed of packing in the container should be constant, the actual value being immaterial for fractions with $\bar{d}_p$ = 124 and 240 μm, whereas for fractions with $\bar{d}_p$ = 65 and 33 μm the optimal height was 20 μm

(5) the use of perforated plates with hole diameters lower than 1.2 mm had a deleterious effect on the reproducibility of the results, even for the smallest particles with $\bar{d}_p$ = 33 mm

(6) no effect of column diameter on the feeding rate of packings of the silica gel type was observed with the exception of the particle fraction with $\bar{d}_p$ = 33 μm fed to a column of diameter 52 mm packed by tap-fill method. An increase in tapping intensity through increase of column mass resulted in an increase in feeding rate.

(7) no difference in the efficiency of columns of diameter 52 mm packed by the tap-fill method was observed when packings of a given particle size were introduced at the same feeding rate, using at the bottom of the packer a gauze or a perforated plate with seven holes

(8) employing in the packer the plate with not less than one hole per 3 cm^2 of cross-sectional area of the column ensures homogeneity of the packing, as evidenced by the symmetrical peaks of test substances. Analogous results were obtained with a gauze.

In conclusion, the experimental results permit one to predict and adjust the feeding rate of packings of the silica gel type through the choice of diameter and number of holes in a perforated plate. Presumably the packing methods described will also be useful for other types of packings, *e.g.*, alumina and magnesium oxide. In addition, it seems probable that these packers can also be employed for filling columns by other "dry-packing" methods.

REFERENCES

1 J. C. Giddings and E. N. Fuller, *J. Chromatogr.*, 7 (1962) 255.
2 J. Halász and M. Naefe, *Anal. Chem.*, 44 (1972) 76.
3 S. T. Sie and N. van den Hoed, *J. Chromatogr. Sci.*, 7 (1969) 257.
4 J. N. Done and J. H. Knox, *J. Chromatogr. Sci.*, 10 (1972) 606.
5 J. Halász, *Z. Anal. Chem.*, 277 (1975) 257.
6 J. Klawiter, M. Kamiński and J. S. Kowalczyk, *J. Chromatogr.*, 241 (1982) in press.
7 M. Beyer (Editor), *Handbuch der Mikroskopie*, Verlag Technik VEB, Berlin, 1973.