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Note

Vapour-phase method for preparing column packings for gas–liquid chromatography using a vacuum technique

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An important operation in the preparation of gas chromatographic columns is the coating of the stationary phase on the solid support surface. The problem of coating is connected with the distribution of the liquid layer and hence with the height equivalent to a theoretical plate (HETP). The method of coating is of importance both in analytical gas–liquid chromatography (GLC) and in investigations on the nature of the interaction between the solute, stationary phase and support surface.

In previous papers[1,2] the possibility of coating the stationary phase in the vapour phase on the solid support was demonstrated. Packings with different stationary phases were prepared, including squalane, dinonyl phthalate, 1,2,3-tris-(2-cyanoethoxy)propane and polymethylsiloxane PMS-100, and the applicability of the method for liquids with different vapour pressures was demonstrated.

According to some data[3], the application of a vacuum when preparing the packing results in improved spreading of the liquid film, acceleration of mass transfer and increased efficiency.

The present work was an attempt to combine the best points of the method of vapour-phase coating with a low-pressure technique. For this purpose, the fluidized bed technique used in previous work[2] had to be modified, so that the movement of the particles was attained by means of vibration instead of a gas flow.

EXPERIMENTAL

The apparatus for coating the stationary phases in the vapour phase (Fig. 1) allows a fluidized bed technique, under vacuum or at atmospheric pressure, to be used.

A sieving machine (MLW Labortechnik, Ilmenau, G.D.R.) was used as the source of vibration.

Table I lists the phases used and the percentage loading, which is time dependent, at a pressure of $4 \cdot 10^{-2}$ Torr. Chromosorb P (60–80 mesh) (Carlo Erba, Milan, Italy) was used as the solid support. For some experiments Silochrom C-80 (Reachim, Stavropol, U.S.S.R.), a wide-pore silica gel with a specific surface area of 101 m^2/g, was used[4]. The amount of stationary phase in each packing was checked by the evaporation method[5].
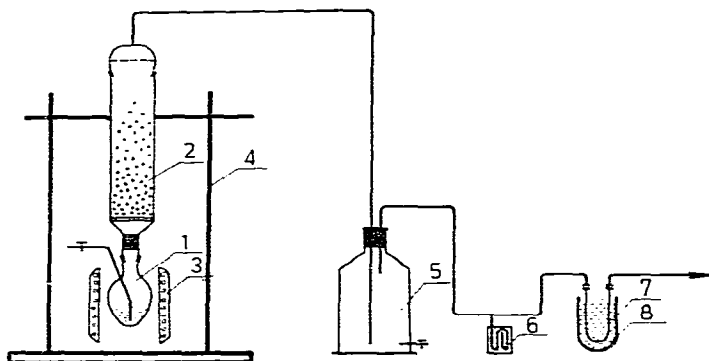
Fig. 1. Apparatus for coating. 1 = Saturator with stationary phase; 2 = tube containing the support; 3 = electrical furnace; 4 = vibrator; 5 = buffer flask; 6 = McLeod gauge; 7 = vapour condenser; 8 = Dewar flask.

TABLE I

CONDITIONS AND RESULTS OF PREPARATION OF PACKINGS

Support	Stationary phase	Temperature (°C)	Pressure (Torr)	Time (h)	Degree of coating (%)
Chromosorb P	Squalane	170	$4 \cdot 10^{-2}$	2	4.47
				4	8.45
				6	14.01
	1,2,3-Tris-(2--cyanoethoxy)-propane	190	$2 \cdot 10^{-2}$	3	12.35
Silochrom C-80	Squalane	130	$1 \cdot 10^{-1}$	0.25	1.34
				0.5	2.69
				1	6.77
				1.5	11.64

As solutes n-hexane, benzene and n-propanol were chosen, as they are representative of different groups of solutes according to the Kiselev classification[6]. The measurements of their retention volumes and the efficiency of the packings were studied with a Tswett 6A gas chromatograph equipped with a flame-ionization detector. The column dimensions were 2 m × 3 mm I.D. and the temperature was 80° in all instances.

Packings with the same percentage loading were also prepared by the usual wet method and the efficiencies of the two types of packings were compared.

RESULTS AND DISCUSSION

Packing preparation

The data in Table I show that liquid phases such as squalane and 1,2,3-tris-(2-cyanoethoxy)propane could be coated on the support surface with percentage loadings commonly used in laboratory practice.

It is evident that an important factor influencing the amount of the stationary phase coated is the nature of the support surface. For instance, the large surface area

of Silochrom C-80 was coated faster than the surface of Chromosorb P, even though the temperature was lower and the pressure was higher.

The amount of stationary phase in the packing could be controlled by varying the time. The relationship between the percentage coating and time is almost linear (Fig. 2).
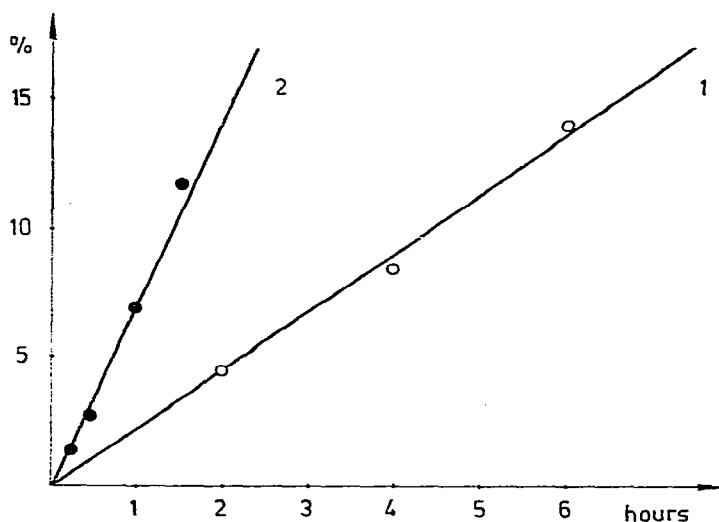

Fig. 2. Plot of percentage coating *versus* time. 1 = Squalane on Chromosorb P; 2 = squalane on Silochrom C-80.

Column efficiency

According to Van Deemster's equation, the HETP gives information about the diffusion and mass transfer process that occurs on the packing particles. As it depends on the distribution of the stationary liquid, the HETP appears to be an indirect but very convenient measure of it.

The data for the columns investigated show in general (Table II) that the HETP values are lower when the coating is carried in the vapour phase and under vacuum than when the classical method of coating in solution is used.

Fig. 3 shows, for example, the Van Deemter curves for benzene on packings prepared by different methods with 1,2,3-tris-(2-cyanoethoxy)propane as the stationary phase on Chromosorb P. Similar curves were obtained for n-hexane and n-propanol.

TABLE II

HETP VALUES AT A GAS FLOW-RATE OF 25 cm/sec

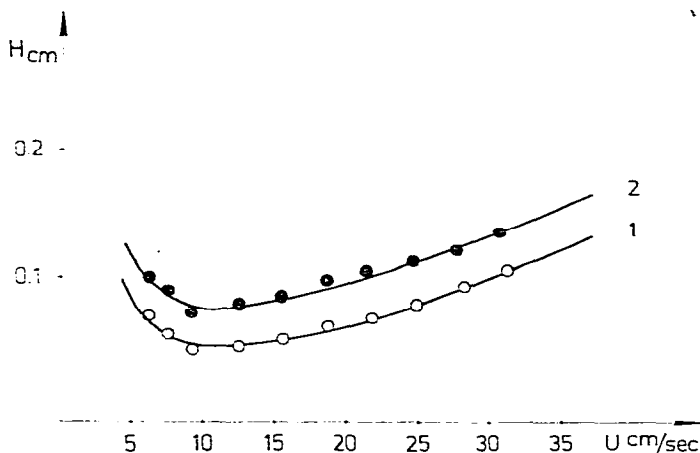| Packing	Method of coating	Degree of coating (%)	HETP (cm)		
			n-Hexane	Benzene	n-Propanol
Chromosorb P–squalane	Vacuum	14.01	0.120	0.118	0.102
	Solution	13.26	0.132	0.146	0.122
Chromosorb P–1,2,3-tris-	Vacuum	12.35	0.550	0.080	0.096
(2-cyanoethoxy)propane	Solution	11.50	0.720	0.112	0.122

H_{cm}

Fig. 3. HETP as a function of linear gas flow-rate. Stationary phase, 1,2,3-tris-(2-cyanoethoxy)-propane on Chromosorb P; temperature, 80°; solute, benzene. 1 = 12.35% of stationary phase coated in vapour phase under vacuum; 2 = 11.5% of stationary phase coated in solution.

Retention volumes

    Table III gives the retention volumes of *n*-hexane, benzene and *n*-propanol at 80°. It can be seen that the retention volumes depend on the method of coating. In the case under investigation, the packings coated with the liquid phase in the vapour phase under vacuum give higher retention volumes, than packings obtained by using the usual wet method for coating. A similar influence of a vacuum was reported previously[7].

    It can be concluded that the method of vapour-phase coating under vacuum offers the possibility of preparing column packings with greater efficiency than those prepared in solution. The retention volumes depend on the method of coating.

TABLE III

SPECIFIC RETENTION VOLUMES

Packing	Method of coating	Degree of coating (%)	Retention volume (cm³)		
			n-Hexane	Benzene	*n*-Propanol
Chromosorb P–squalane	Vacuum	14.01	1.31	1.94	22.29
	Solution	13.26	0.85	1.28	14.67
Chromosorb P–1,2,3-tris-	Vacuum	12.35	0.05	1.04	4.07
(2-cyanoethoxy)propane	Solution	11.50	0.02	0.99	4.06

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