Journal of Chromatography, 247 (1982) 167–170
Elsevier Scientific Publishing Company, Amsterdam — Printed in The Netherlands

CHROM. 15,047

Note

Preparation and evaluation of benzyldimethylchlorosilane-treated silica column for high-performance liquid chromatography

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(Received May 18th, 1982)

In previous papers, we considered the effect of different concentrations of 3-aminopropyltriethoxysilane (3APTS)-treated[1] and N-(2-aminoethyl)-γ-aminopropyltrimethoxysilane (N2AAPTS)-treated silica columns[2] for use in high-performance liquid chromatography (HPLC). An additional parameter that must be considered during the bonding process is the degree of coverage of the silica surface. As a significant development of this work, we have now extended the effect of the mean pore diameter and specific surface area of silica to benzyldimethylchlorosilane (BDM)-treated silica columns.

EXPERIMENTAL

Reagents

Naphthalene (NA) and anthracene (AN) were obtained from Wako (Osaka, Japan). Four kinds of highly microporous spherical silica gels differing in mean particle size, mean pore diameter and specific surface area were purchased from Fuji-Davison (Nagoya, Aichi, Japan; Table I). The commercial silica gel Fine-Sil was purchased from Jasco (Tokyo, Japan; Table I). Methanol was used after distillation. All chemicals were of analytical-reagent grade.

TABLE I

SILICA GELS USED

Silica gel	*Mean particle size (μm)*	*Mean pore diameter (Å)*	*Specific surface area (m^2/g)*	*Pore volume (ml/g)*
Fuji-Davison 1*	10.0	70	490	0.87
Fuji-Davison 2*	8.4	153	204	0.78
Fuji-Davison 3*	8.4	180	167	0.75
Fuji-Davison 4*	5.8	85	400	0.85
Fine-Sil	10.0	100	–	–

* These names and serial numbers were assigned by the authors for convenience and are not commercial names.

0021-9673/82/0000–0000/$02.75

Apparatus

The HPLC measurements were carried out using a Kyowa Seimitsu KHU 16 Mini Micro Pump equipped with a Jasco Uvidec 100-II variable-wavelength detector.

Stationary phase

According to the previous methods[1,2], after 4 g of dried Fuji-Davison 1, 2, 3 or 4 or Fine-Sil had been added to 50 ml of a 4% solution of BDM in xylene, the silica gel suspension was refluxed for 5 h, filtered with a glass filter (1 μm), washed several times with xylene, methanol and acetone and then dried *in vacuo* at 70°C for 2 days, finally producing the silica gels for HPLC, which are listed as BDM-treated silica gels in Table II.

TABLE II

SURFACE TREATMENTS, ELEMENTAL ANALYSES AND NUMBER OF ACCESSIBLE BENZYL SURFACE GROUPS FOR GELS PREPARED FROM FUJI-DAVISON 1–4 AND FINE-SIL

Concentration of BDM in xylene: 4%.

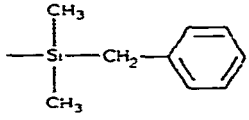
Treated gel	*C found (%)*	*No. of accessible benzyl surface groups per gram* ($\times 10^{21}$)
BDM-1	9.38	0.52
BDM-2	4.69	0.26
BDM-3	3.83	0.21
BDM-4	8.45	0.47
BDM-Sil	8.82	0.49

Column preparation

BDM-treated silica gels were packed into stainless-steel columns (125 × 4.6 mm I.D.) using a balanced density method through a 10-ml stainless-steel packer at a rate of 500 kg/cm^2 with a Kyowa Seimitsu KHW-20 ultra-high-pressure pump.

RESULTS AND DISCUSSION

From the elemental analysis of silica gel treated with BDM, the number of accessible benzyl surface groups per gram of silica gel can be calculated by the following procedure. If BDM is substituted monofunctionally on silica gel, the surface structure of silica gel can be written as



According to the previous methods[1,2], the number of accessible benzyl surface groups per gram is then given by $[(C/100)/12.011 \cdot 9] \cdot 6.022 \cdot 10^{23}$ where C = weight percentage of carbon and $6.022 \cdot 10^{23}$ is Avogadro's number. Substitution of values of C

TABLE III

EFFECT OF ORGANIC SOLVENTS FOR BDM-TREATED SILICA GEL

Concentration of BDM in organic solvent: 4%.

Solvent	*C found (%, w/w)*
Toluene	3.93
Xylene	4.83
Benzene	4.16

TABLE IV

RELATIONSHIP BETWEEN RETENTION VOLUME, NUMBER OF THEORETICAL PLATES AND OPERATING PRESSURE

Operating conditions: column, 125 × 4.6 mm I.D.; mobile phase, methanol–water (70:30); flow-rate, 1.0 ml/min; detection, 254 nm.

Treated gel	*Retention volume (ml)*		*No. of theoretical plates*		*Operating pressure (kg/cm²)*
	NA	*AN*	*NA*	*AN*	
BDM-1	3.18	4.42	2500	1400	25
BDM-2	2.7	3.4	2500	2200	30
BDM-3	2.6	3.1	1900	1500	30
BDM-4	3.75	6.0	1400	1200	50
BDM-Sil	3.65	5.7	1900	1750	30

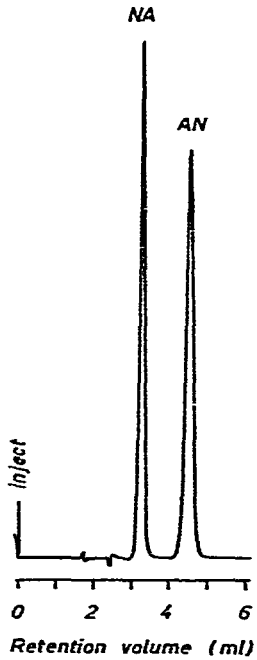


Fig. 1. Chromatogram of NA and AN. Column: BDM-1, 125 × 4.6 mm I.D. Chromatographic conditions as shown in Table IV.

found by elemental analysis into this formula gives the number of accessible benzyl surface groups per gram of silica gel surface, indicated in Table II.

Under reflux conditions (5 h), dry organic solvents for BDM were compared with the elemental analysis data (Table III). From the results, we chose xylene as the solvent for BDM-treated silica.

Table IV shows the relationship between the retention volume (ml), the number of theoretical plates and the operating pressure at which the several kinds of silica gel were treated.

Fig. 1 shows a typical chromatogram obtained on BDM-treated silica gel.

As can be seen from the data in Tables II and IV, the most important parameters of silica gel with respect to the number of accessible BDM groups per gram of silica gel were the mean pore diameter and the specific surface area.

ACKNOWLEDGEMENTS

The authors are indebted to Dr. Hiroshi Kishimoto and Dr. Satoshi Kawai for their valuable advice.

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