Available online at www.sciencedirect.com
SCIENCE DIRECT®
Journal of Chromatography A, 1059 (2004) 111–119
JOURNAL OF CHROMATOGRAPHY A
www.elsevier.com/locate/chroma

Peralkylated-β-cyclodextrin used as gas chromatographic stationary phase prepared by sol–gel technology for capillary column

Minmin Liang[a,*], Meiling Qi[a], Changbin Zhang[b], Ruonong Fu[a]

[a] *Department of Chemistry, School of Science, Beijing Institute of Technology, Beijing 100081, PR China*
[b] *Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences, Beijing 100085, PR China*

Received 28 August 2004; received in revised form 6 October 2004; accepted 7 October 2004

Abstract

For the first time, three peralkylated-β-cyclodextrins (β-CD), permethylated-β-CD, perethylated-β-CD and perpentylated-β-CD, were coated onto the fused-silica capillary by sol–gel method with simplicity and rapidity. Multiple steps in conventional column preparation technology were avoided. Also, these new columns demonstrated many inherent advantages, the main being the outstanding thermal stability (up to 300 °C), high number of theoretical plates, excellent column-to-column and run-to-run reproducibility, and pronounced selectivity for positional isomers and enantiomers. Using *n*-tridecane as a test reagent ($k = 4.18$), an efficiency value of 3520 theoretical plates/m was obtained on a sol–gel perpentylated-β-CD capillary column (15 m × 0.25 mm I.D.). On the basis of the results, we proposed that the peralkylated-β-CD, which has no terminal hydroxyl group, is encapsulated in the sol–gel network and the whole matrix is chemically bonded to the surface of the fused-silica tubing.

Keywords: Sol–gel technology; Capillary column; Peralkylated-β-cyclodextrin; Positional isomer; Enantiomers

1. Introduction

The proposal of open tubular capillary columns is credited to Dr. Marcel Golay as far back as 1958 [1]. This opened the theoretical possibility of gas chromatography with high resolution. Capillary GC is now a matured separation technique that is widely used in various fields of science and industry [2–4]. However, contemporary technique for the preparation of columns is complex and time-consuming [5–7], which remains one of the most challenging questions in this area. With the introduction of sol–gel capillary column coating technology, it became possible to prepare capillary column with simplicity and rapidity. The sol–gel approach is unique and is applicable to the manufacture of columns for a wide range of separation techniques, including capillary GC [8–11] and capillary electrophoresis [12–19]. The columns prepared by sol–gel process offer good retentive characteristics and hydrolytic stability, compared to those prepared by the conventional methods. Recent developments in sol–gel technology for manufacturing capillary GC columns have stimulated significant interest in both academia and industry. To date, it has been successfully used for the preparation of sol–gel polydimethylsiloxane (PDMS) [8], poly(ethylene glycol) (PEG) [9] and crown ether [10,11] capillary columns in GC. Such columns have shown excellent results. Commercialization of these column technologies has taken place. Although the sol–gel technology can prepare capillary GC columns with simplicity and rapidity, it also has limitations. The stationary phases based on sol–gel technology are all hydroxy-terminated stationary phases, such as PDMS, PEG and crown ether. So, much further work is necessary to extend the range of stationary phase suitable for sol–gel method in GC.

* Corresponding author. Tel.: +86 10 68941647.
E-mail addresses: minml@sina.com, minmlzcb@hotmail.com (M. Liang).

0021-9673/$ – see front matter © 2004 Elsevier B.V. All rights reserved.
doi:10.1016/j.chroma.2004.10.019

Cyclodextrins (CD) are available in different formats: α, β and γ. They can be derivatized with different functional groups at different positions. CD derivatives are widely used and show highly selective separations, especially for positional isomers and enantiomers [20–22]. However, CD derivatives have not any terminal hydroxyl groups and the recent survey [23–28] revealed that few studies have been made concerning the capillary column preparation using CD derivatives as stationary phases by sol–gel technology.

In this paper, the capillary columns coated with three peralkylated-β-CDs by sol–gel method was investigated, and the column performances were determined and evaluated. The sol–gel CDs columns provided excellent chromatographic performances: outstanding stability, excellent reproducibility, and pronounced selectivity for a wide range of test solutes. It showed that the three peralkylated-β-CDs, which are not hydroxy-terminated stationary phases, are also suitable for use as capillary GC stationary phase by sol–gel technology. The mechanism of specific retention based on sol–gel method was discussed and their chiral separation was also investigated.

2. Experimental

2.1. Materials and chemicals

Permethylated-β-CD, perethylated-β-CD and perpentylated-β-CD were provided by China Agricultural University. Fused-silica capillary (0.25 mm I.D.) was obtained from Yong Nian Optical Fibre Factory (Hebei Province, China). Trifluoroacetic acid (TFA, containing 5% water), tetraethoxysilane and methanol were purchased from Beijing Chemical Reagents Company (Beijing, China). The other chemical reagents were analytical grade.

2.2. Equipment

Column performance was determined on a SP-3700 gas chromatograph (Beijing Analytical Instrument Factory, Beijing, China) equipped with a capillary split injection system and flame-ionization detector. TL-9900 Chromatography Data Station was used for data collecting and processing. Carrier gas was high-purity nitrogen. The thermal stability of peralkylated-β-CD and sol–gel coatings were tested using a HP5890 SeriesII gas chromatograph equipped with a CZ-100 pyrolyzer (Beijing Institute of Technology Instrument Company, Beijing, China).

2.3. Sol–gel column preparation

A fused-silica capillary (15 m × 0.25 mm I.D.) was first rinsed with methylene chloride followed by a brief nitrogen purge for 20 min, then the column was sequentially rinsed with 1.0 mol/L NaOH solution, deionized water, 0.1 mol/L HCl solution for 30 min, respectively, and was washed with deionized water for 30 min again. Afterwards, the column was installed in GC oven and thermally conditioned under a flow of nitrogen for 2 h at 120 °C.

The sol–gel coating solution was prepared as follows: 0.1 g peralkylated-β-cyclodextrin was first dissolved in 800 μL methylene choride, then 120 μL of tetraethoxysilane was added to this solution and mixed for 5 min to obtain homogeneous solution. Then, 80 μL TFA (95%) was added to the solution, and the mixture was thoroughly vortexed. The clear top portion of the resulting solution was introduced into the fused-silica capillary using a nitrogen pressure of 0.4 MPa. After a set of period of in-capillary residence time (about 40 min) at room temperature, the solution was expelled from the capillary under the same pressure and the capillary was subsequently purged with nitrogen for another 30 min. This was followed by temperature-programmed heating from 40 to 240 °C at a rate of 1 °C/min with a hold time of 6 h at 240 °C. While conditioning, the column was purged with nitrogen.

Three columns (columns 1, 2, and 3), which were coated with permethylated-β-CD, perethylated-β-CD and perpentylated-β-CD by sol–gel method, respectively, were prepared.

2.4. Conventional column preparation

Fused-silica capillary tubes (15 m × 0.25 mm I.D.) were treated by depositing sodium chloride onto the inner wall. The columns were then statically coated with a 0.5% (w/v) solution of the stationary phase in dichloromethane. In short, the column was filled with the cyclodextrin dissolved in dichloromethane and the solvent was subsequently removed by applying a controlled vacuum in the range of 50–60 mbar, regulated by a solenoid valve [29–31]. Following filling, the solvent was evaporated at 35 °C under vacuum for about 24 h, finally the columns were conditioned under a slow nitrogen flow at 80, 120, 160 and 180 °C, for 1 h each and finally at 240 °C for 4 h. With this procedure, a uniform coating with a film thickness of 0.31 um was achieved.

Three columns (columns 1-1, 2-2, and 3-3), which were statically coated with permethylated-β-CD, perethylated-β-CD and perpentylated-β-CD, respectively, were prepared

2.5. Pyrolysis-gas chromatography

In order to compare the thermal stability of conventionally wall-coated column and sol–gel coated column, the polyimide coating on a 1 cm segment of sol–gel coated capillary was first removed, and then the bare capillary was pyrolyzed in the probe of pyrolyzer. The *F–T* curve describing the change of pyrolysis fractions (*F*) of the sample with the probe temperature (*T*) was determined in the temperature ranging from 200 to 800 °C. It was assumed that the total

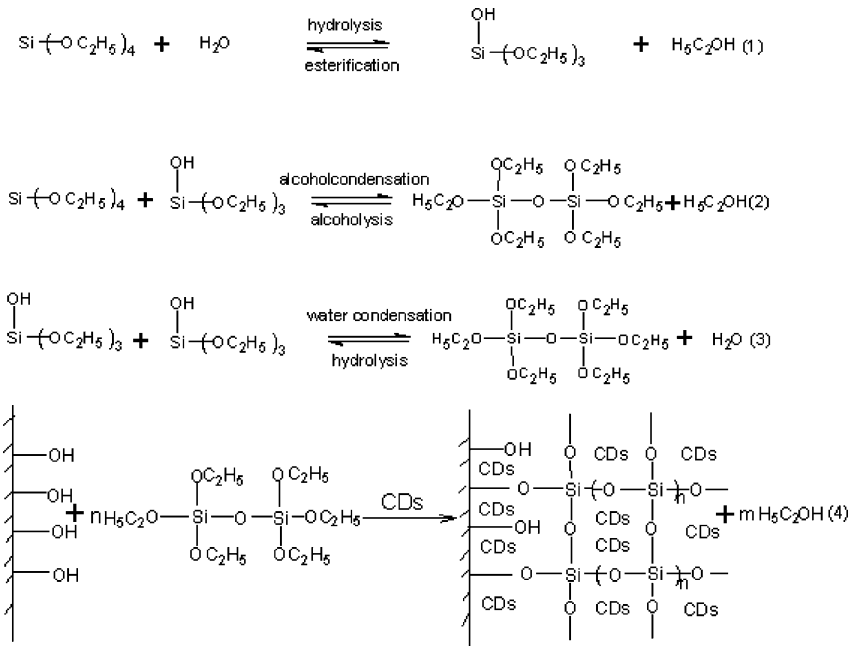


Fig. 1. Reactions involved in the sol–gel columns.

peak area was denoted as A at 800 °C where a complete pyrolysis happens and the F value is 100%. If the total areas under the rest temperatures below 800 °C were denoted as a_i, the F_i value can be calculated by $F_i = \sum a_i/A$ ($i = 1, 2, \ldots, n$). The bare capillary was placed in the probe of pyrolyzer and pyrolysis was made under the designated temperatures. Before the next designated temperature, the pyrolyzer was stabilized for 5 min. The F–T relationship was obtained (Fig. 2) according to this procedure. In addition, a 1 cm segment of conventionally wall-coated capillary was treated under the same conditions, and the F–T relationship was also obtained (Fig. 3).

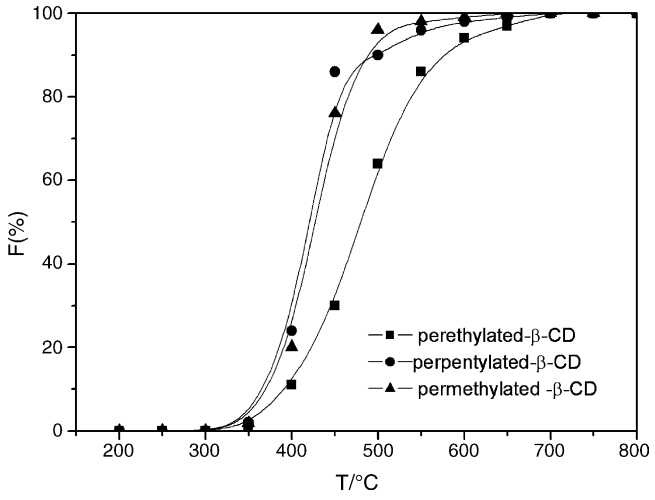


Fig. 2. The F–T relationship of the sol–gel coated peralkylated-β-CD stationary phases. Conditions: carrier gas, N_2; detector, FID, 260 °C; column, 30 m × 0.25mm I.D. fused-silica capillary; stationary phase, polysiloxane SE-54; film thickness, 0.31 um; column temperature, 200 °C.

3. Results and discussion

3.1. Chemical reaction in sol–gel columns

In this sol–gel approach, tetraethoxysilane are polymerized. Three reactions are involved in the process. The first reaction in Fig. 1 is a hydrolysis reaction, while reactions (2) and (3) are condensation reactions. During the first reaction, alkoxy groups are replaced by hydroxyl ones and some alcohol is released. The silanol groups are highly reactive and condense readily with other alkoxysilanes (reaction 2) or with each other (reaction 3). As a result, a siloxane bond (Si–O–Si) is created and one molecule of alcohol is

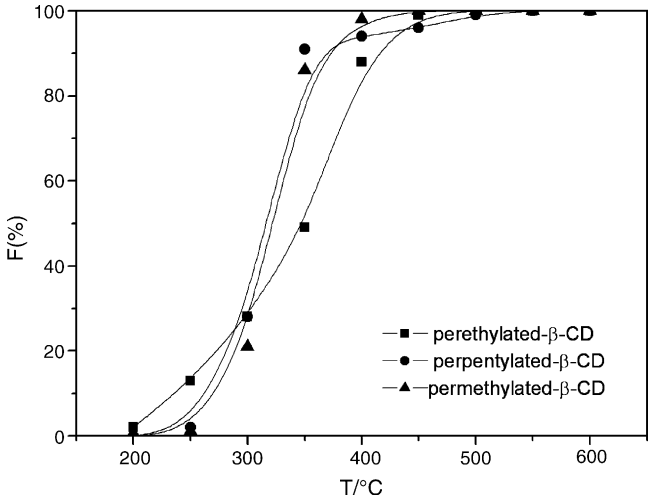


Fig. 3. The F–T relationship of conventionally wall-coated peralkylated-β-CD stationary phases. Conditions: same as those in Fig. 2.

Table 1
Chromatographic performance of the six 15 m × 0.25 mm I.D. sol–gel coated columns

Column number	Stationary phase	Retention factor, k[a]	Column efficiency (plate/m)[a]	Retention factor, k[b]	Temperature (°C)[b]	Column efficiency (plate/m)[b]
1	Permethylated-β-CD	3.64	3027	4.57	120	2767
1-1	Permethylated-β-CD	3.87	3041	4.84	120	2870
2	Perethylated-β-CD	3.22	3186	4.21	100	3027
2-2	Perethylated-β-CD	3.14	3117	4.16	100	2812
3	Perpentylated-β-CD	3.64	3370	4.04	120	3154
3-3	Perpentylated-β-CD	3.71	3611	4.18	120	3520

[a] Naphthalene was used as the test reagent and the column temperature was 100 °C.
[b] *n*-Tridecane was used as the test reagent.

released. Through subsequent hydrolysis and condensation reactions, an extended three-dimensional network (gel) is finally formed [32]. At the same time as the network is growing, the loose network of the matrix anchors the cyclodextrins without any chemical binding and produce a integral stationary phase together with cyclodextrins. Then, the sol–gel network growing in the vicinity of the fused-silica capillary inner walls got bonded to the capillary inner surface (reaction 4). Afterwards, the capillary was flushed and a certain part of the gel removed, leaving a coat of several micrometers thickness on the capillary wall. The thickness of this coating can be varied by manipulating the incapillary residence time of the sol solution or by adjusting the concentrations of various ingredients in the sol–gel coating solution. When the analytes were injected into GC, the analytes directly enter into the sol–gel network and interact with CDs in the net.

3.2. Column performance

The column performance was determined and the results are listed in Table 1. The results show that the three sol–gel coated CDs columns possess good column efficiency and are comparable to the efficiency obtained on the relevant conventionally coated column. This indicates that these three CDs are suitable for use as capillary GC stationary phase by sol–gel method.

3.3. Column reproducibility

Six 15 m × 0.25 mm I.D. sol–gel perpentylated-β-CD column were prepared under identical conditions and tested for column-to-column reproducibility. The column efficiency (N), retention factor (k), and separation factor (α) for selected test solutes and R.S.D. values associated with them are presented in Table 2. The R.S.D. values were 0.9% for N, 1.5% for k, and 1.7% for α, respectively. These low values indicate the reproducibility is excellent for this prepared column. The run-to-run repeatability on the sol–gel perpentylated-β-CD column was also excellent. To evaluate it, alcohols mixture was injected in five replicate GC runs. Also, the low R.S.D.s of less than 0.8% in retention times were obtained. These low R.S.D. values bear testimony to the fact that the sol–gel method for the preparation of CD columns is indeed reliable and highly reproducible.

3.4. Thermal stability of the sol–gel columns

The thermal stability and pyrolysis pathway of the sol–gel columns were investigated by pyrolysis-gas chromatography (Py-GC). The F–T relationships [33] for both sol–gel coated peralkylated-β-CDs coating (Fig. 2) and conventionally wall-coated peralkylated-β-CDs coating (Fig. 3) were obtained as indicated in the Section 2.5. The thermal stability comparison between the sol–gel coated peralkylated-β-CDs coating and the conventionally wall-coated peralkylated-β-

Table 2
Column-to-column reproducibility for efficiency (N), retention factor (k), and separation factor (α) on six sol–gel coated perpentylated-β-CD columns[a]

Column	Column efficiency		Retention factor		Separation factor	
	N	R.S.D. (%)	k	R.S.D. (%)	α[b]	R.S.D. (%)
1	3147		4.13		1.30	
2	3154		4.04		1.32	
3	3166	0.9	4.20	1.5	1.28	1.7
4	3109		4.21		1.33	
5	3186		4.17		1.33	
6	3121		4.20		1.34	

[a] Conditions: injection, split (60:1, 240 °C); detector, 260 °C; column temperature, 120 °C constant. Solute for measuring N and k was *n*-tridecane. Conditions for separation factor (α): column temperature, 80 °C constant.
[b] For solute pair *o*-dichlorobenzene and *p*-dichlorobenzene.

Table 3
Retention time repeatability for the prepared test mixture[a], before and after rinsing column 1 with methylene chloride ($n = 3$)[b]

Solute	Before rinsing (average)		After rinsing (average)	
	Retention time (min)	R.S.D. (%)	Retention time (min)	R.S.D. (%)
n-Decane	1.80	0.14	1.76	0.32
n-Undecane	2.47	0.08	2.38	0.20
n-Dodecane	2.81	0.17	2.80	0.22
n-Octanol	4.16	0.11	4.11	0.14
1,4-Butanediol	4.87	0.18	4.74	0.21
Naphthalene	5.24	0.22	5.17	0.31
Tridecane	5.61	0.32	5.47	0.38
2,6-Dimethylaniline	6.47	0.07	6.31	0.11
n-Tetradecane	6.98	0.11	6.69	0.21
2,6-Dimethylphenol	7.13	0.06	7.01	0.14
Methyldecanoate	8.04	0.17	7.87	0.23
2-Ethylhexanoic acid	10.13	0.21	9.74	0.32

[a] Test mixture was prepared as follows: the same volume of 12 kinds of solutes (in the table) were added to a vial and thoroughly mixed to obtained a homogeneous test mixture.
[b] Conditions: column, 15 m × 0.25 mm I.D. fused-silica capillary column; stationary phase, sol–gel permethylated-β-CD; split (60:1, 240 °C); detector, 260 °C; column temperature, programmed from 80 °C at 4 °C min^{-1}.

CDs coating was made. The obtained *F–T* curve indicated that all of the three kinds of CD stationary phases in the sol–gel network are stable up to 300 °C (shown in Fig. 2) while these conventionally wall-coated CD stationary phases can only be stable up to 200 °C (shown in Fig. 3), much lower than corresponding sol–gel stationary phase. Pyrolysis occurs at 310 °C in sol–gel perethylated-β-CD stationary phase, 306 °C in sol–gel permethylated-β-CD stationary phase, and 300 °C in sol–gel perpentylated-β-CD stationary phase, which are in good agreement with the baseline evaluation results. These findings show that the maximum operating temperatures are 310 °C for sol–gel perethylated-β-CD stationary phase, 306 °C for sol–gel permethylated-β-CD stationary phase and 300 °C for sol–gel perpentylated-β-CD stationary phase. The comparison results indicated that the sol–gel columns offered excellent thermal stability compared with the conventional columns. Also, in the pyrolysis process, we can see that sol–gel perethylated-β-CD stationary phase decomposed much more slowly than the sol–gel perpentylated-β-CD and sol–gel permethylated-β-CD stationary phase. Hence, the sol–gel CDs columns are more stable than conventionally wall-coated CDs columns. The sol–gel coating, to some extent, can prevent the CDs stationary phase, encapsulated in the sol–gel network, from bleeding.

3.5. Solvent stability of the sol–gel columns

The solvent stability was tested by comparing the solute retention times in triplicate before and after rinsing the sol–gel permethylated-β-CD column with methylene chloride (Table 3). Data presented in Table 3 suggest that the solute retention time do not change obviously, which showed that this column has excellent solvent stability. Strong immobilization and structural integrity are important attributes of the prepared sol–gel permethylated-β-CD stationary phase that contributes to this excellent retention time reproducibility.

3.6. Deactivation ability

The deactivation quality was evaluated by using alcohols and amine derivatives as test solutes. Alcohols and amine derivatives are two classes of compounds prone to tailing because of their polar interactions with the residual silanol groups. The results were shown in Figs. 4 and 5, indicating the excellent peak symmetries obtained for both alcohols and amine derivatives, highlighting the high degree of inertness of the column itself. The sol–gel coated peralkylated-β-CD columns offered satisfactory deactivation possibly probably because less surface silanol groups are exposed for the reason

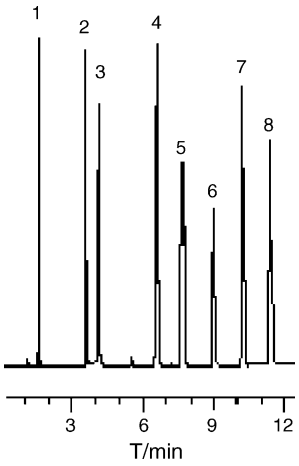


Fig. 4. GC separation of alcohols on column 1. Conditions: injection, split (60:1, 240 °C); detector, FID, 260 °C; column temperature, programmed from 60 °C at 4 °C min^{-1}; peaks: (1) methanol; (2) ethanol; (3) *n*-butanol; (4) *iso*-butanol; (5) *n*-heptanol; (6) *n*-octanol; (7) 1,4-butanediol; (8) *n*-decanol.

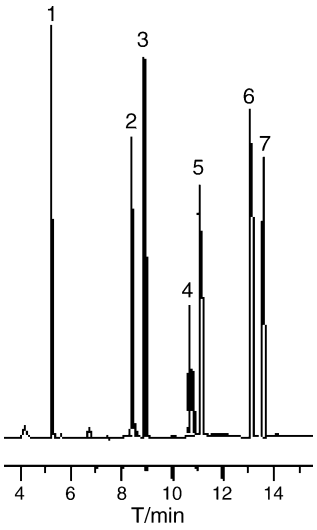


Fig. 5. GC separation of amine derivatives on column 2. Conditions: column temperature, programmed from 50 °C at 5 °C min^{-1}, others are the same as in Fig. 4. Peaks: (1) *N*,*N*-dimethylaniline; (2) *N*-methylaniline; (3) *N*-ethylaniline; (4) 2,6-dimethylaniline; (5) 2-ethylaniline; (6) 3-ethylaniline; (7) 4-ethylaniline.

that, during the sol–gel process, some of the surface silanol groups are derivatized and others are buried under the sol–gel network it.

3.7. Application

The sol–gel coated peralkylated-β-CD columns provide high selectivity and enhanced resolution of position isomers, and the three sol–gel coated peralkylated-β-CD columns were used to separate several positional isomers, as shown in Figs. 6 and 7 and Table 4. Nitrotoluene and dimethoxybenzene were baseline separated on column 1 and column 2, respectively. These three sol–gel coated CDs columns all

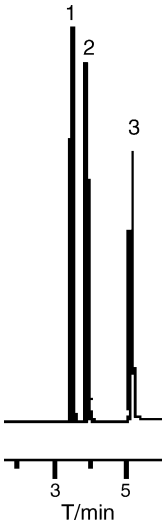


Fig. 6. GC separation of nitrotoluene on column 1 at 120 °C. Peaks: (1) *o*-nitrotoluene; (2) *m*-nitrotoluene; (3) *p*-nitrotoluene.

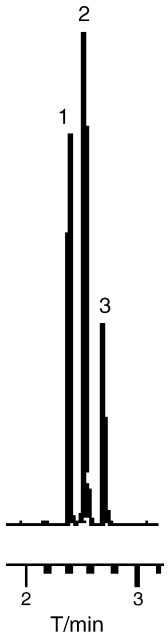


Fig. 7. GC separation of dimethoxybenzene on column 2 at 120 °C. Peaks: (1) *m*-dimethoxybenzene; (2) *p*-dimethoxybenzene; (3) *o*-dimethoxybenzene.

showed much strong ability for the separation of the positional isomers of xylene, cresols and nitrobromobenezene. This indicates that the separation ability of these CDs to these positional isomers do not decrease when they are coated onto the inner wall of capillary columns by sol–gel technology.

Conventionally coated CDs capillary columns have been used successfully for chiral separation [34–36]. Here, we demonstrate the gas chromatographic separations of carvone, menthol, camphor and limonene on the prepared sol–gel CDs columns, as shown in Fig. 8 and Table 5. The chromatographic data obtained for limonene states the ability of the sol–gel coated permethylated-β-CD column to fully separate the two enantiomers and a resolution *R* of 1.52 was obtained (Fig. 8a). The low separation factor α of 1.07 is typical for enantioselective separations on cyclodextrin column and is

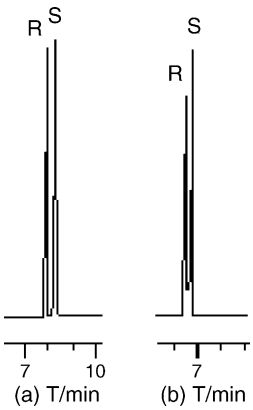


Fig. 8. GC separation of limonene on (a) column 1 at 80 °C and (b) column 1-1 at 80 °C.

Table 4
Separation of disubstituted benzene isomers on the four prepared columns

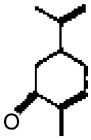
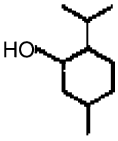
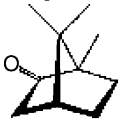
Solutes	Column number	Peak order	Temperature (°C)	Retention factor			Relative retention	
				k_1	k_2	k_3	$\alpha_{2/1}$	$\alpha_{3/2}$
Xylene	1	*o*, *m*, *p*	60	3.74	4.52	5.96	1.21	1.32
	2		60	3.51	4.11	5.10	1.17	1.24
	3		60	2.74	3.01	3.34	1.10	1.11
	1-1		60	3.81	4.57	5.76	1.20	1.26
Dichlorobenzene	1	*o*, *m*, *p*	80	4.74	4.79	6.08	1.01	1.27
	2		80	5.01	5.71	6.34	1.14	1.11
	3		80	4.36	4.80	5.76	1.10	1.20
	1-1		80	4.72	4.72	5.90	1.00	1.25
Cresols	1	*o*, *m*, *p*	140	5.10	6.34	6.74	1.24	1.06
	2		140	4.87	6.07	6.17	1.25	1.02
	3		140	4.21	5.84	5.91	1.39	1.01
	1-1		140	5.01	6.27	6.44	1.25	1.03
Chlorotoluene	1	*o*, *m*, *p*	90	4.21	4.38	4.95	1.04	1.13
	2		90	3.15	3.46	3.74	1.10	1.08
	3		90	3.26	3.26	3.29	1.00	1.01
	1-1		90	4.07	4.31	4.74	1.06	1.10
Nitrochlorobeneze	1	*m*, *o*, *p*	140	3.41	3.89	4.20	1.14	1.08
	2		140	3.21	3.79	4.05	1.18	1.07
	3		140	4.11	4.60	4.78	1.12	1.04
	1-1		140	3.37	3.91	4.22	1.16	1.08
Nitrotoluene	1	*o*, *m*, *p*	120	3.75	4.12	5.50	1.10	1.33
	2		120	3.14	3.67	4.96	1.17	1.35
	3		120	3.21	3.84	5.14	1.20	1.34
	1-1		120	3.87	4.17	5.47	1.08	1.31
Dimethoxybenzene	1	*m*, *p*, *o*	120	2.13	2.41	2.71	1.13	1.12
	2		120	2.00	2.25	2.50	1.12	1.11
	3		120	2.11	2.27	2.57	1.08	1.13
	1-1		120	2.17	2.44	2.80	1.12	1.15
Nitrobromobenezene	1	*m*, *o*, *p*	140	6.21	10.80	14.48	1.74	1.34
	2		140	4.37	6.99	8.53	1.60	1.22
	3		140	5.04	8.67	9.80	1.72	1.13
	1-1		140	6.17	10.24	11.88	1.66	1.16

much better than the results obtained on the conventionally coated permethylated-β-CD column (Fig. 8b), the α, R values are 1.04 and 1.21, respectively. The results in Table 5 also show that for different derivatives of these racemic compounds, the enantioseparation results obtained on the three sol–gel coated CDs columns are comparable to the results obtained on the conventionally coated CDs column.

Host–guest interaction via inclusion complex formation is the explanation for the isomer resolution obtained with CD derivatives [37–40]. Cyclodextrins are cyclic molecules with a tapered 3D-form. The separation process is based on the degree of accessibility of the components in the cyclodextrin cavity. If a molecule fits better in the cavity, it will show a higher retention factor. On the basis of the obtained results, we propose that since peralkylated-β-CDs have not any terminal hydroxyl groups, they cannot get incorporated in the sol–gel network structure. Differently, they are encapsulated in the sol–gel network, and then the whole matrix is bonded to the surface of the fused-silica tubing. When the analytes were injected into GC, they go into the sol–gel network and further enter the CD's cavity and form an inclusion compound. The stabilities of the inclusion compounds are different, and the analytes are eluted at different times. So, the separation process depends on shape of cyclodextrin and the molecule to be separated. However, it is impossible to track the exact amount of the chiral selector taken in the sol–gel network, and it is very difficult to predict the elution order. The lack of a well understood recognition mechanism impedes the controlled development of sol–gel stationary phases with predictable enantioselectivity.

Some of the limitations encountered thus far with capillary columns made in the sol–gel process include a higher-than-usual bleed profile and lower chromatographic efficiency, when compared with commercially produced CDs columns. It may be for the reason that the CDs stationary phase was encapsulated in the sol–gel matrix instead of being part of the matrix. In addition, the stationary phases made in the sol–gel process comprise recurring organic and inorganic domains, which is different from the conventional stationary phases that are either totally organic (as most commonly used

Table 5
The capacity factor k and separation factor α of some racemic compounds on the four prepared columns

Solute	Column no.	Temperature (°C)	Retention factor, k		Separation factor, α
Carvone 	1	110	8.54	8.88	1.04
	2	110	8.47	8.64	1.02
	3	110	8.50	8.76	1.03
	1-1	110	8.62	8.96	1.04
Menthol 	1	100	18.71	19.08	1.02
	2	100	16.34	16.50	1.01
	3	100	17.08	17.25	1.01
	1-1	100	16.23	16.39	1.01
Camphor 	1	110	8.92	9.15	1.02
	2	110	8.87	9.05	1.02
	3	110	8.41	8.58	1.02
	1-1	110	8.67	8.76	1.01
Limonene 	1	80	5.09	5.45	1.07
	2	80	4.97	5.17	1.04
	3	80	4.76	4.99	1.05
	1-1	80	4.14	4.30	1.04

in gas–liquid chromatography) or totally inorganic (as most commonly used in gas–solid chromatography). This structure may make the sol–gel CDs columns to have lower efficiency. Also, this attribute may open new possibilities for unique chromatographic selectivities.

4. Conclusion

A new type of capillary GC column, which encapsulated peralkylated-β-CDs in the gel matrix, was developed using sol–gel method. The chromatographic performances of CDs-encapsulated columns for the separation of the positional isomers and enantiomers were investigated. The results showed that the sol–gel coated peralkylated-β-CDs columns offered baseline resolution for most of the isomers and these three peralkylated-β-cyclodextrins are suitable for use as capillary column GC stationary phase by sol–gel technology.

References

[1] M.J.E. Golay, Gas Chromatogr. (1958) 67.
[2] R.E. Clement, Gas Chromatography: Biochemical, Biomedical, and Clinical Applications, Wiley, New York, 1990.
[3] A. Mark Adams, Z.L. Chen, P. Landman, D. Timothy, Colmer Anal. Biochem. 266 (1999) 77.
[4] K. Mastovska, J. Hajslova, M. Godula, J. Krivankova, V. Kocourek, J. Chromatogr. A 907 (2001) 235.
[5] C.L. Woolley, R.C. Kong, B.E. Richter, M.L. Leo, J. High Resolut. Chromatogr. 7 (1984) 329.
[6] J. Bouche, M.J.M.J. Verzele, J. Gas Chromatogr. 6 (1968) 501.
[7] L.G. Blomberg, J. Microcolumn Sep. 2 (1990) 62.
[8] D.X. Wang, L. Sau, A. Malik, Anal. Chem. 69 (1997) 4566.
[9] C. Shende, A. Kabir, A. Malik, Anal. Chem. 75 (2003) 3186.
[10] Z.R. Zeng, W.L. Qiu, J. Chromatogr. 4 (2000) 304.
[11] D.H. Sun, L.C. Li, Chem. Rep. 3 (2002) 182.
[12] J.D. Hayes, Ph.D. Dissertation, University of South Florida, Tampa, FL, 2000.
[13] J.D. Hayes, A. Malik, J. Chromatogr. B 695 (1997) 3.
[14] J.D. Hayes, A. Malik, Anal. Chem. 72 (2000) 4090.
[15] J.D. Hayes, A. Malik, Anal. Chem. 73 (2001) 987.
[16] K. Sakai-Kato, M. Kato, T. Toyo'oka, Anal. Chem. 74 (2002) 2943.
[17] K. Sakai-Kato, M. Kato, T. Toyo'oka, Anal. Biochem. 308 (2002) 278.
[18] M.T. Dulay, J.P. Quirino, B.D. Bennett, R.N. Zare, J. Sep. Sci. 25 (2002) 3.
[19] M.T. Dulay, J.P. Quirino, B.D. Bennett, M. Kato, R.N. Zare, Anal. Chem. 73 (2001) 3921.
[20] D.W. Armstrong, W.Y. Li, A.M. Stalcup, H.V. Secor, R.R. Izuc, J.I. Seeman, Anal. Chim. Acta 234 (1990) 365.
[21] C. Bicchi, A.D. Amato, P. Rubiolo, J. Chromatogr. A 843 (1999) 99.
[22] C. Bicchi, V. Manzin, A.D. Amato, P. Rubiolo, Flavour Fragr. J. 10 (1995) 127.
[23] R. Cervini, G. Day, A. Hibberd, G. Sharp, Chem. Aust. 68 (2001) 16.
[24] Z. Zeng, W. Qiu, H. Xing, Z. Huang, Anal. Sci. 16 (2000) 851.
[25] C. Shende, A. Kabir, E. Townsend, A. Malik, Anal. Chem. 75 (2003) 3518.
[26] H. Minakuchi, K. Nakanishi, N. Soga, N. Ishizuka, N. Tanaka, Anal. Chem. 68 (1996) 3498.

[27] N. Ishizuka, H. Kobayashi, H. Minakuchi, K. Nakanishi, K. Hirao, K. Hosoya, T. Ikegami, N. Tanaka, J. Chromatogr. A 960 (2002) 85.
[28] B. Channer, P.U. Uhl, M.R. Euerby, A.P. McKeown, H. Lomax, G.G. Skellern, D.G. Watson, Chromatographia 58 (2003) 135.
[29] R.F. Arrendale, L.B. Smith, L.B. Rogers, J. High Resolut. Graphia 45 (1997) 255.
[30] R.F. Arrendale, R.F. Severson, O.T. Chortyk, J. Chromatogr. Resolut. Chromatogr. 20 (1997) 85.
[31] R.F. Arrendale, R.F. Severson, T. Chortyk, J. Chromatogr. High Resolut. Chromatogr. 21 (1998) 113.
[32] M.M. Collison, Anal. Chem. 29 (1999) 289.
[33] R. Dai, Y. Ye, A. Luo, R. Fu, S. Zhang, G. Xie, S. Jin, J. Anal. Appl. Pyrolysis 42 (1997) 9.
[34] Z. Juvancz, J. Szejtli, Trends Anal. Chem. 21 (2002) 379.
[35] S. Schurch, A. Saxer, S. Claude, R. Tabacchi, B. Trusch, J. Hulliger, J. Chromatogr. A 905 (2001) 175.
[36] V. Schurig, Trends Anal. Chem. 21 (2002) 647.
[37] W.A. Koening, D. Ichrln, T. Runge, J. High Resolut. Chromatogr. 13 (1990) 702.
[38] S. Li, W.C. Purdy, Anal. Chem. 64 (1992) 1405.
[39] K. Kano, H. Kamo, S. Negi, T. Kitae, R. Takaoka, M. Yamaguchi, H. Okubo, M. Hirama, J. Chem. Soc., Perkin Trans. 2 (1999) 15.
[40] R. Reinhardt, M. Richter, P.P. Mager, P. Hennig, W. Engewald, Chromatographia 43 (1996) 187.