Separation of compounds on a titania monolithic capillary column

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Introduction

The aim of this study was to develop a titania monolithic capillary column. Traditionally, silica packed columns are used but many problems are encountered with extreme pH and high temperature.
In order to minimize solvent consumption and reduce the analysis time, column dimensions were reduced. However, due to many practical drawbacks associated with the column packing, a new generation of stationary phases was recently developed: monolithic beds synthesized in situ in capillary columns that eliminated these packing problems.
In previous studies, silica and zirconium monolithic stationary phases were extensively used, whereas few articles describe the use of titanium alkoxide as a monolithic stationary phase precursor.

Experimental conditions

Material synthesis:
Titania monoliths were prepared from a solution containing titanium n-propoxide ($Ti(O^nPr)_4$, hydrochloric acid (HCl), Formamide (FA) and water (H_2O).
The molar ratio of the synthesis was:
$Ti(O^nPr)_4/HCl/FA/H_2O$ = 1 /0.5 / 0.5 / 20.5.
This mixture was introduced to an open capillary tube (length = 15cm and internal diameter = 75µm) and stored at a temperature of 30°C for one night.

Measurements:
Separations were carried out with an Agilent HP ^{3D}CE capillary electrophoresis system in the nano liquid chromatography mode.
Solutes: a mixture of Caffeine, Naphtalene, β-hydroxy-ethyl theophylline and Theophylline was prepared in the mobile phase. Injection: 2 bars during 0.2 min. Elution: ΔP = 6 bars.
Mobile phases were made of ACN and TRIZMA buffer with proportions of 95/5 (v/v).

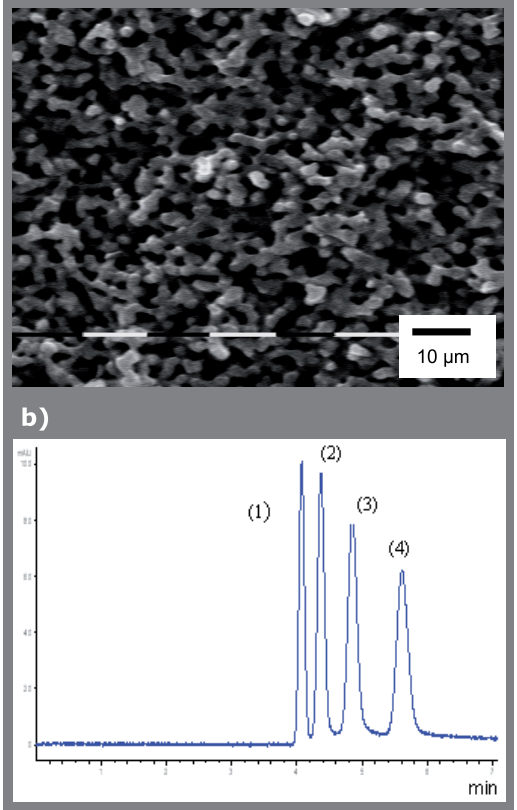

Figure 1:
(a) Titania monolith SEM picture at x1200
(b) Separation with 95/5 ACN/Trizma buffer (v/v)

Results and discussion

The obtained monoliths morphological characteristics can be observed in figure 1a (d_{pores} = 5µm, $d_{skeleton}$ = 2µm).
The capillary column was able to separate the 4 compounds. Changing the ACN/TRIZMA buffer mobile phase ratio, shifted the retention time factors (k) according to the HILIC mode.
In figure 1b, Naphtalene (1) which was used as a dead time marker, was first eluted. Then, caffeine (2), β-hydroxy-ethyl Theophylline (3) and Theophylline (4) were eluted respectively.
The results illustrated the suitability of titania monoliths as a stationary phase. This will pave the way for new types of stationary phases that are good alternatives for silica monoliths.

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