Preparation *in situ* of monoliths based on silica from the sol-gel process for use in capillary liquid chromatography

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Abstract

Capillary monolithic columns based on silica were optimized in order to be used in capillary liquid chromatography.

Introduction

Capillary monolithic columns based on silica have been the subject of many studies in the last few years, due to their high permeability, with efficiencies comparable to packed columns.
However, the conditions of synthesis still are not reproducible and need more research, because of recurrent problems of shrinkage inherent to the sol-gel process.
Therefore, this work aimed at the optimization of the conditions for preparation *in situ* of capillary monolithic columns based on silica with their surfaces modified by thermal immobilization of polydimethylsiloxane (PDMS) and their application in Capillary Liquid Chromatography (CLC)

Results and Discussion

To prepare two different kinds of monoliths (one of pure silica (1) and another of an organic-inorganic hybrid (2)), various conditions were evaluated and the best conditions were: 180 mg of urea was added in 2 mL of AcOH 0,01 mol/L and polyethylene glycol (PEG). After homogenization, tetramethyl orthosilicate (TMOS) was added and, for monolith 2, methyltrimethoxysilane (MTMS) was also added. The mixture was stirred for 30 minutes in an ice bath. The fused silica capillaries (20 cm long by 200 µm i.d. pretreated with 1 mol/L NaOH) were filled using a N_2 flow system. The capillaries were sealed with rubbers and submitted to a thermal treatment with a gelation step using 43 ºC during 24 hours followed by 120 ºC for 4 hours. Table 1 shows the amounts of the components of sol-gel process. Figure 1 shows the complete filling of the capillaries and the morphology of the monoliths. After being washed with water, monolith 1 was activated using 150 ºC during 24 hours, then the columns were filled with a solution of PDMS and hexane (50% v/v).
After 4 days of self-immobilization, the polymer was thermally fixed on the silica surface and the column was evaluated by Capillary Liquid Chromatography, as can be seen on figure 2.

Table 1. Conditions of the synthesis of the monoliths

	Monolith 1	Monolith 2
PEG (mg)	180	100
TMOS (mL)	0,80	0,81
MTMS (µL)	-	90

Figure 1. Capillaries filled with the monoliths 1 and 2, with an augmentation of 450 times.

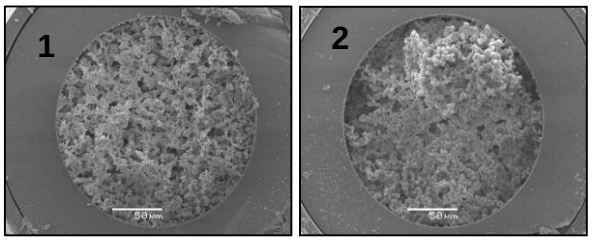
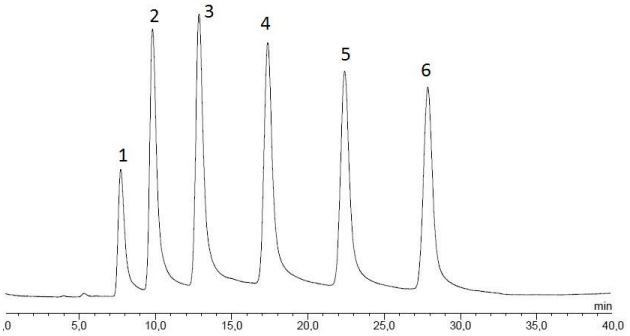


Figure 2. Chromatogram of 6 alkylbenzenes: 1-benzene, 2-toluene, 3-ethylbenzene, 4-propylbenzene, 5-butylbenzene, 6-pentylbenzene on column 1.



The column showed an efficiency of 69400 plates per meter with mobile phase gradient of aqueous ACN (40-65%), flow rate of 1,2 µL/min, injection vol.: 50 nL and detection at 215 nm.

Conclusions

Monolithic columns based on silica were obtained inside capillaries of 200 µm i.d., with complete and homogeneous filling. After thermal immobilization of PDMS on the monolithic surface of pure silica, it was possible to achieve the complete separation of 6 alkylbenzenes with good efficiency by CLC.

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