Chinese Chemical Letters Vol. 15, No. 12, pp 1477-1479, 2004 1477
http://www.imm.ac.cn/journal/ccl.html

Preparation of Polymeric Monolithic Column and Application to the Resolution of Aromatic Compounds in Capillary Electrochromatography

Xia WANG[1], Wen Zhi LI[2], Chun Hui LIU[1], Yong Min LI[1], Li Ren CHEN[1]*

[1]Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 730000
[2]College of Chemistry and Chemical Engeering, Liaocheng University, Liaocheng 252059

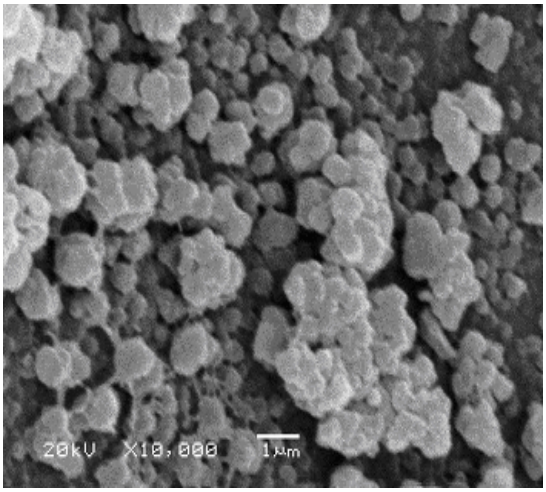
Abstract: Polymeric monolithic capillary column-poly(GMA-co-EDMA-co-AMPS) was prepared and used for resolution of aromatic compounds (benzalcohol, 2-phenylethanol, toluene, ethylbenzene) .

Keywords: Polymeric monolithic column, aromatic compounds, capillary electrochromatography.

Capillary electrochromatography (CEC)[1] is a hybrid technique between capillary zone electrophoresis (CZE) and high performance liquid chromatography (HPLC). Interest for this technique has been motivated by the promising capacity of CEC to combine the selectivity of HPLC with miniaturization and high efficiency power of CZE. In recent years, the preparation and application of monolithic columns has been hot in this field, which is defined as a column consisting of one piece of solid that possesses interconnected skeletons and interconnected flow paths (through-pores) through the skeletons[2]. In general, monolithic columns can be sorted into two classes: silica-based monolith and polymeric monolith.Silica-based monoliths were generally prepared using sol-gel technology to create a continuous sol-gel network throughout the capillary. Polymeric monoliths, including acrylamide-based, acrylate- or methacrylate-based, styrene-based polymers, etc[3], are generally formed inside the capillary by a step-wise chain polymerization reaction initiated by UV light or thermal treatment. Polymerization reaction mixtures usually consist of monomers and cross-linker, initiator and a porogenic mixture of solvents. A variety of monomers can be employed to fabricate the final monolith, being both charged and hydrophilic, to generate an electroosmotic flow (EOF), or uncharged and hydrophobic, to allow reversed-phase interactions. The formation of monolith can be achieved in situ within either untreated[4] or pretreated[5] capillaries. The polymeric monolithic capillaries usually exhibit high selectivity, increased retentively, and chromatographic properties similar to reversed-phase HPLC.

* E-mail: zrswangx@yahoo.com.cn

Figure 1 Scanning electron micrograph of the monolith



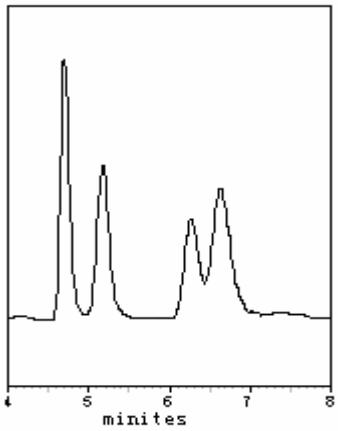
The aim of this study is to present a novel polymeric monolithic column, and apply it to separate aromatic compounds. The polymeric monolith consisted of glycidyl methacrylate (GMA) (monomer), ethylenedimethacrylate (EDMA) (crosslinker), and 2-acrylamido-2-methyl-1-propanesulfonic acid (AMPS) to generate EOF. As expected, the prepared monolithic capillary possessed apparent reversed-phase chromatographic properties. **Figure 1** shows a scanning electron micrograph of the cross section of the poly (GMA-co-EDMA-co-AMPS) bed.

A fused-silica capillary (100 μm I.D.× 375 μm O.D.) of about 36 cm was washed with 1 mol/L NaOH and water for 1 h, respectively, followed by methanol for 30 min. Then the capillary was dried by the passage of N2 for 5 h. γ-MAPS solution of methanol (1:1, v/v) was filled in the capillary. After that, the capillary was kept in a column heater at 35˚C overnight. Finally, the residual reagents were flashed out with methanol and water, respectively, followed by drying with N2 flow for 5 h. Thus the layer of γ-MAPS was introduced onto the inner wall of the capillary. AMPS of appropriate weight was dissolved in a solution consisting of EDMA and GMA. The porogenic solvent contained 75% v/v *n*–propanol and 25% v/v 1,4-butanediol. The monomer and porogenic solvents were mixed at a ratio of 25:75 vol. %. AIBN with weight/total monomer volume of 1% was utilized as the initiator for the in situ polymerization. After ultrasonication for 3 min, a small part of the polymerization mixture was filled in the pretreated capillary to a total length of 20 cm. The capillary were plugged with silicon rubber blanket at the both ends and kept in the column heater at 60˚C for 14 h. A detection window was created at the end of continuous polymer bed by removing the polyimide coating using a razor blade. Then the capillary was placed to the CE instrument and rinsed with methanol for 1 h with a pressure of 100 psi, followed by NaOH for 1 h, water 1 h, and the running buffer 3 h, respectively. Finally, the capillary was

equilibrated by applying a voltage of 10 kV until the electric current and flow rate were stabilized.

The prepared monolithic column was applied to the separation of aromatic compounds. Toluene, ethylbenzene, benzalcohol, and 2-phenylethanol were used as test compounds. The experimental results showed that buffer consisting of acetonitrile and phosphate was suitable for the separation of aromatic compounds. The retention factors (k´) for the four compounds were 0.186, 0.306, 0.563, and 0.654, respectively. The resolutions (Rs) were 0.96 for benzalcohol and 2-phenylethanol, and 0.99 for toluene and ethylbenzene. The theoretical plate number reached 39000/m.

Figure 2 The electrochromatogram of four aromatic compounds



Mobile phase:50% ACN in 5 mmol/L of phosphate buffer (pH 7.7); applied voltage: 15 kV with 20 psi at the inlet end; electrokinetic injection: 5 kV for 2s. UV detection: 254 nm. Capillary: 21/31.2 cm, 100 μm I.D× 375 μm O.D. temperature: 25˚C.
Peaks: benzalcohol, 2-phenylethanol, toluene, ethylbenzene. Obtained on P/ACE MDQ.

Acknowledgment

The authors thank the Chinese Academy of Sciences for financial support to “Hundreds of Talents” program.

References

1. T. Tsuda, K. Nomura, G. Nakagawa, *J. Chromatogr*., **1982**, *248*, 241.
2. T. Nobuo, K. Hiroshi, I. Norio, *et al.*, *J.Chromatogr. A*, **2002**, *965*, 35.
3. L. Q. Crisina, D.M. Nicola, M. Virginie, *et al.*, *Electrophoresis*, **2003**, *24*, 917.
4. E. C. Peters, M. Petro, F. Svec, *et al.*, *Anal.Chem.* ,**1998**, *70*, 2288.
5. R. A. Wu, H. F. Zou, H. J. Fu, *et al*., *Electrophoresis*, **2002**, *23*, 1239.

Received 6 November, 2003