11th International Conference
on Advanced Materials

Rio de Janeiro Brazil
September 20 - 25

ICAM2009

Preparation and characterization of capillary monolithic columns for nano-chromatography

Yacine Badjah Hadj Ahmed, Metaab Al Badrani, Zeid Al Othman*,
Abdul Rahman Al Warthan
King Saud University, College of Science, Chemistry Department 2455, Riyadh 11451, Saudi Arabia
* Corresponding author
E-mail: zaothman@ksu.edu.sa

ABSTRACT - In the few last years, many research groups have paid great attention to the study and development of new miniaturized separation methods offering the possibility to achieve good resolutions and high efficiency in a short analysis time making use of very low volumes of both samples and reagents. Among these separation methods, nano-liquid chromatography (nano-LC) seems to be very promising even for practical applications in different fields, especially in the analysis of chiral compounds of pharmaceutical, clinical, environmental and/or agro-chemical interest. However, the successful development of these techniques is closely related to the technical challenges associated with the column manufacturing. The obtained columns offer several advantages over classical packed columns, e.g., rapid separations achieved in a short analysis time with high efficiency, use of minute volumes of mobile phases as well as small amounts of packing materials.

The present work aimed to the preparation and characterization of capillary columns suitable for use in nano-scale high performance liquid chromatography. Column tubing used for these preparations consisted of a polyimide coated fused-silica capillary of inner diameter 100µm. The tube capillary inner surface was first activated by sequential rinsing with several solvents then treated by a suitable difunctional reagent in order to enable covalent attachment of the monolith. After rinsing and drying steps, the capillary was filled with a mixture containing butyl or hexyl acrylate as reacting monomers, 1,3-butanediol diacrylate as crosslinker, azobisisobutyronitrile as radical polymerization initiator and a suitable porogen mixture. After sealing the ends of the capillary, the mixture was allowed to react *in-situ* by heating. After removing the seals, the monolithic column was washed with several solvents in order to flush all the excess reagents then dried under a nitrogen stream.[1-3]

Four capillary columns having a 25cm length were prepared by varying the monomer and the monomer to porogen ratio. The obtained columns were characterized by several methods. The Fourier transform infrared spectra of the four reticulated polymers showed the absence of the vibration bands of the monomers. Morphology characterisation of the monolithic stationary phases was performed by means of both optical microscopy and scanning electron microscopy (SEM). Microphotographs of the prepared capillary columns illustrated a uniform structure of the monolith bed with no gap between the inner wall of the capillary and the monolithic phase while a well-developed macroporous structure, typical for this kind of polymeric supports is also observed.[1-3]

References

1. Norman W. Smith, Zhengjin Jiang, Review: Developments in the use and fabrication of organic monolithic phases for use with high-performance liquid chromatography and capillary electrochromatography, Journal of Chromatography A, xxx (2007) xxx–xxx
2. Renan Wu, Lianghai Hu, Fangjun Wang, Mingliang Ye, Hanfa Zou, Review: Recent development of monolithic stationary phases with emphasis on microscale chromatographic separation, Journal of Chromatography A, xxx (2007) xxx–xxx
3. Isabelle Gusev, Csaba Horvath, Streaming potential in open and packed fused-silica capillaries, Journal of Chromatography A, 948 (2002) 203–223