2006 TAIWAN INTERNATIONAL SCIENCE FAIR

CATEGORY : Physics

PROJECT : High speed size-exclusion chromatography using spherical meso-structured cellular foam

AWARDS : Physics First Award

SCHOOL : Raffles Junior College

FINALISTS : Ong Shu Ren Sarah

COUNTRY : Singapore

Abstract of Exhibit
Taiwan International Science Fair

CATEGORY: **Engineering**

TITLE: **High Speed Size-exclusion Chromatography (SEC) Using Spherical Meso-structured Cellular Foam (MCF)**

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Introduction

Size-exclusion chromatography (SEC) is often used to determine the molecular weights of and separate polymers and proteins. The porous packing of the SEC column effects the separation of molecules, with larger molecules eluting earlier.

Interest in high-speed SEC for larger molecules has been building, especially for combinatorial polymerization reactions and online SEC-MS applications. Mechanical stability of the packing, which siliceous materials have more of than polymeric ones, therefore needs to be improved. Several silicas have been explored but limited pore sizes and pore volumes have restricted their usage to separating small molecules.

Siliceous MCF templated using oil-in-water microemulsions has good potential for SEC packing because it has ultralarge pore size (20-50 nm), high porosity and sturdy skeleton. However conventional MCF consists of highly irregular particles and hence cannot be used as packing.

Purpose of research

We sought to create uniform, spherical MCF particles with ultrahigh porosity and narrow pore and window size distribution and explore their usability for high-speed, low-solvent usage SEC.

Procedure/data analysis

With uncomplicated modifications to the traditional MCF synthesis technique significant

changes in particle morphology were achieved. With low-temperature aging in a quiescent state, desired MCF particles (3-5 μm) were obtained. This is verified from SEM and N_2 sorption analysis. With calcination carried out much higher than the usual 550°C, micropores were removed, increasing the packing's selectivity. Pore and window sizes were controlled by the amount of organic swelling agent trimethylbenzene and the mineralizing agent NH_4F.

Three MCF samples were prepared from this pathway. Due to chokepoint effect, window size rather than the pore size would be critical for SEC. Hence sample names MCF-16, MCF-17 and MCF-26 where the numbers denotes the window size (nm).

The samples were individually slurry-packed into columns through which 12 polystyrene standards were individually run. With corresponding elution volumes, calibration curves were plotted. The good linearity in the size-exclusion regions is desirable for accurate molecular weight determination. This helps confirm narrow window size distribution. Despite using only one small-dimensioned column, the slopes at the size-exclusion regions are gradual (desirable for high SEC resolution). This could be credited to the ultrahigh porosity of MCF materials (~10-14% higher than commercial packing). Increasing the window size improves the effectiveness of separation in higher molecular weight ranges

- MCF-10: $5.1\times10^2 - 1.9\times10^4$ Da
- MCF-17: $2.2\times10^3 - 6.9\times10^4$ Da
- MCF-26: $5.8\times10^3 - 1.8\times10^5$ Da

A mixture of four polystyrene standards was used to assess separation performance. Studying the elution profiles, with increasing porosity and pore size, separation resolution increased and selectivity among large molecules improved. Single MCF columns achieved high-resolution separation comparable to that of two commercial polymeric columns in series (Waters HR5E, particle size 5 μm and similar effective molecular weight range), with 80% less solvent usage and 60% shorter duration.

Conclusion

Separations usually need long columns and high flow rate for high resolution and rapid analysis, producing high pressure. MCF columns, which can withstand pressures up to 5000 psi, will thus be more suitable than other commercial columns. The experiments have shown that spherical MCFs are great for high-speed SEC of low to middle range M_w molecules (polymers, large biomolecules).

評語

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