US007955574B2

(12) **United States Patent**
Fryxell et al.

(10) **Patent No.: US 7,955,574 B2**
(45) **Date of Patent: Jun. 7, 2011**

(54) **POROUS THIN FILM AND PROCESS FOR ANALYTE PRECONCENTRATION AND DETERMINATION**

(75) Inventors: **Glen E Fryxell**, Kennewick, WA (US); **Xiaohong Li**, Richland, WA (US); **Raymond S Addleman**, Benton City, WA (US); **Richard Skaggs**, Richland, WA (US)

(73) Assignee: **Battelle Memorial Institute**, Richland, WA (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 258 days.

(21) Appl. No.: **12/242,924**

(22) Filed: **Oct. 1, 2008**

(65) **Prior Publication Data**

US 2010/0077837 A1 Apr. 1, 2010

(51) **Int. Cl.**
G01N 33/00 (2006.01)

(52) **U.S. Cl.** **422/501**; 422/401; 422/500

(58) **Field of Classification Search** 422/101, 422/99, 56, 401, 501, 500
See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

4,942,135	A	7/1990	Zaromb
5,001,453	A	3/1991	Ikejiri et al.
5,142,143	A	8/1992	Fite et al.
5,481,110	A	1/1996	Krishnaswamy et al.
6,171,378	B1	1/2001	Manginell et al.
6,455,003	B1	9/2002	Anvia et al.
6,808,742	B2	10/2004	Rouse et al.
6,852,299	B2	2/2005	Kohmura et al.
6,946,161	B2	9/2005	Yamada et al.
7,016,462	B1	3/2006	Keville et al.
7,147,695	B2	12/2006	Mitra

FOREIGN PATENT DOCUMENTS

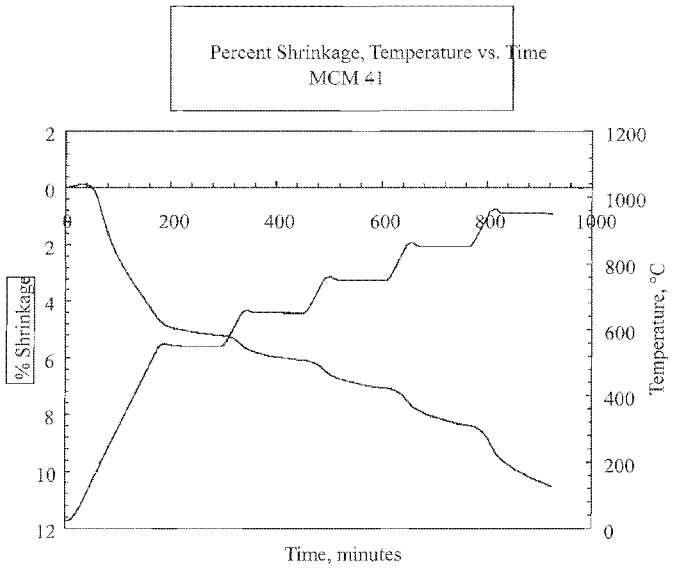
JP	2002137913	A	5/2002
WO	2006026067	A1	3/2006
WO	2007127921	A2	11/2007

OTHER PUBLICATIONS

Katsoulidis, et al, Ordered mesoporous CoOx/MCM-41 materials exhibiting long-range self-organized nanostructured morphology, Elsevier, Jan. 1, 2006, pp. 71-80.
Lu, et al, Microporous Silica Prepared by Organic Templating: Relationship between the Molecular Template and Pore Structure, American Chemical Society, Apr. 29, 1999, pp. 1223-1229.
International Search Report/Written Opinion.
Zhao, Dongyuan, et al., Continuous Mesoporous Silica Films with Highly Ordered Large Pore Structures, Advanced Materials, Wiley-VCH Verlag, Weinheim, DE, vol. 10, No. 16, Nov. 10, 1998.
Bruinsma, P. J., et al., Low K Mesoporous Silica Films Through Template-Based Processing, Mat. Res. Symp., Materials Research Society, USA, vol. 443, Jan. 1, 1997.
Zhang, Xiaoming, et al., Synthesis of Transition-metal Containing MSU Mesoporous Silica Molecular Sieves, Chemistry Letters, Chemical Society of Japan, Tokyo, JP, No. 8, Aug. 1, 1998.

Primary Examiner — Sam P Siefke
(74) *Attorney, Agent, or Firm* — James D. Matheson

(57) **ABSTRACT**

A porous thin film and a method are disclosed. The film has a generally uniform thickness and includes fused silica particles that provide multiple types of pores in the film. Particles are generally uniformly distributed on a surface and have a generally open structure that provides the film with an open interface readily accessible to fluids. The film provides for preconcentration of, e.g., heavy metals in fluid and gas samples that provides for direct determination of the analytes.

40 Claims, 6 Drawing Sheets