Asian Journal of Chemistry; Vol. 23, No. 2 (2011), 733-739

ASIAN JOURNAL OF CHEMISTRY

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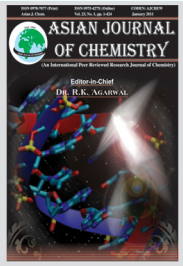

Microwave Irradiation Used to Immobilize the Stationary Phase in Sol-Gel and Organic Polymer Matrix for Micro-HPLC and Capillary Electrochromatography

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(*Received*: 15 March 2010; *Accepted*: 1 October 2010) AJC-9150

A novel design to immobilize the stationary phase for micro-HPLC and capillary electrochromatography has been initially attempted. Porous temporary frit in fused silica capillary was rapidly fabricated by sintering stainless steel powders rapidly, which could withstand a high pressure up to 60 MPa during slurry packing. A mixture of monomer and porogenic solvents such as silica-based and organic-based monomers were pumped into the packed capillary, respectively, followed a quick polymerization by microwave irradiation. The sol-gel network and organic matrix were strong enough to hold back the sorbent particles in place, then rendering the end frits unnecessary. The typical columns prepared demonstrated excellent chromatographic performance using our self-installed micro-HPLC system. Baseline separation of the model analytes was achieved with the largest theoretical plate number (N) up to 40000/m for the last eluted compound (ethylbenzene), whilst a higher N over 74000/m was obtained using the sol-gel immobilized column by CEC. A scanning electron micrograph of a cross-section of the capillary column showed visually the morphological changes of packing material during the microwave irradiation.

Key Words: Microwave irradiation, Fritless packing column, Capillary liquid chromatography, Electrochromatography.

INTRODUCTION

As a hybrid technologies of packed and monolithic capillary columns, the entrapped columns by immobilizing packed microspheres within a matrix attracted considerable attention in the separation fields such as micro-HPLC, capillary electrochromatography (CEC), solid phase microextraction (SPME) *etc.*[1-5]. It behaves the advantages including the available use of numerous packing materials for conventional HPLC columns, minimal alteration of original properties of the sorbent, no need to prepare the inlet and outlet frits. The most two important factors to prepare the entrapped columns possess how to rapidly make a robust and stable temporary inlet frit and how to entrap the stationary phase tightly within the tube. Over the past decades, different strategies have been developed to prepare a frit for retention of the stationary phase in capillaries such as using the taper end columns[6-8], coupled capillaries *via* a polytetrafluoroethylene (PTFE) sleeve or restrictors[9,10], silica-based sintered frits[11,12] and monolithic frits[13-15]. Among the above approaches, various combinations of sol-gel and sintering techniques were the most popular methods for on-column frit preparations[16]. Frits less than 1 mm were made out of the packed bed itself by Lynen *et al.*[9], which were sintered in water by using a perforated heating ribbon. Special attention was paid to the inlet and outlet frit preparation in order to avoid gas bubble formation which renders CEC-ESI/MS problematic. Maiolica *et al.*, also reported self-made frits for nanoscale columns, which were carried out through fixing the glass fibre by sol-gel[17]. The obtained columns appeared to be comparable to "no-frit" columns with near ideal chromatographic characteristics. Wang *et al.*, inserted dry 5 μm Si resins into a fused-silica capillary about 0.5 mm, then dipped it into a sol-gel solution for polycondensation[18]. The on-column frit preparation and its utility for peptide separation in LC-MS/MS were described. These columns with such frits could replace any capillary liquid transfer tubing without any specialized connections up-stream of a spray tip column. Piraino *et al.*,[19] compared the performances of different frits prepared with sol-gel, sintering, photopolymerization and modified sol-gel technologies. It was shown that the modified sol-gel and photopolymerizated frits showed improved reproducibilities. However, the sintered frits are still associated with the least amount of peak dispersion. Recently, a novel single-particle fritting technology was developed to manufacture particulate-packed capillary columns with excellent robustness for CEC use[20]. For the second important factor, it was usually realized by flushing the reactant as a matrix and followed with a polymerization reaction by thermo-initiation, photo-initiation or

sol-gel technologies. Chirica and Remcho[21] made a fritless capillary columns taking methyacrylate-based monomers and a mixture of porogenic solvents to entrap sorbent particles for micro-HPLC and CEC. After the packing column was forced into an organic polymer matrix, it should be placed in an oven at 60 ºC for 48 h. Li and co-workers[22] also applied sol-gel technology to produce sub-micrometer sized organic-inorganic hybrid silica packing particles for CEC. The hybrid silica particles were prepared using TEOS and vinyltriethoxysilane as precursors and were encapsulated with a layer of polymerization product between styrene and divinylbenzene (DVB). Bakry *et al.*[23] demonstrated the immobilization of continuous silica beds including C_{18} reversed phase, anion exchange and chiral stationary phases for micro-HPLC and CEC. It was achieved by *in situ* polymerization of styrene and divinylbenzene in presence of decanol as a porogen and AIBN as thermal initiator. It should be noted that the capillaries should also be placed in a water bath at 70 ºC for 24 h. Furthermore, similar monolithic capillaries by thermal initiation were carried out about 50-70 ºC for 12-72 h[24-26]. Xie and Oleschuk[27] introduced the fastest method for entrapping of octadecylsilane microsphere columns for CEC recently. The polymerization reactant with butyl acrylate monomer was photoinduced and the polymerization under UV illumination could be carried out about several minutes. Initiation by conventional heating presents the disadvantage of long reaction time due to the slow convection of heat, whilst photo-polymerization necessitated the use of capillaries with UV-transparent outer coatings and special UV crosslinker tend to be more expensive than conventional polyimide-coated capillaries. In a general sense, polymerization can be realized by the use of electromagnetic irradiation as the energy source for the polymerization of monomers, oligomers and polymers[28]. Compared with conventional means, microwave heating has the advantages of being volumetric, direct, selective and instantaneously controllable. The interaction between materials and microwaves is direct and occurs as soon as the electromagnetic field is established. All the molecules of material are subject to the electromagnetic field, although the field strength decreases as it gets deeper into the material.

Here, to the best of our knowledge, a novel method to immobilize reversed-phase sorbents within the walls of fused silica capillary tubes was initially developed using microwave irradiation. It was investigated in both typical entrapment matrix of the silica-based and organic-based polymers. Our intention is to develop a common and quick immobilization procedure that would have a minimal effect on the chromatographic performance of the separation media, while at the same time yielding a stable fritless packed column simply and rapidly. The chromatographic and electrophoretic behaviours of the prepared columns have comparatively been evaluated in both modes of micro-HPLC and CEC. Baseline separation of some model compounds could be obtained such as thiourea, benzene, toluene and ethylbenzene on two typical columns. It provided a viable alternative to the traditional methods, with the competitive advantages of simplicity, high efficiency and low expense.

EXPERIMENTAL

All capillary electrochromatography experiments were performed on a Agilent 3DCE system (Agilent Technologies, Inc., Walbronn, Germany) equipped with a diode array detector and the capability to apply up to 1.2 MPa pressure to one or both ends of the capillary. Micro-HPLC experiments were carried out by a CL2001 HPLC system (Beijing Cailu Scientific Instrument Ltd., China). It is equipped with an on-column detector, which a changeable ultraviolet-visible wavelength can be changed in the range of 190-700 nm. A T union was used to split delivering appropriate amounts of the mobile phase. The prepared separation column inlet was installed in one outlet of T union using a PEEK sleeve (0.5 mm.i.d., 1.6 mm o.d.) and a screwed joint. Another capillary (4 m × 100 mm.i.d) for splitting the injection sample and mobile phase was contacted to another outlet of T union. Chromatograms were recorded using the computer software N2000 chromatography data system supplied by Zhida Information Engineering Ltd., Zhejiang University, China. A pneumatic pump (RPL-ZD10, Dalian Replete Scientific Instrument Co., Ltd, Dalian, China) and an ultrasonic bath (KQ-500E, Kunshan Ultrasonic Instrument CO., Ltd, Kunshan, China) were used to drive solvent and slurry into the capillary during column preparation. Irradiation step was carried out in a home microwave oven (Midy Co. Ltd., Guangdong, China) with a microwave output power of 700 W and a frequency of 2450 Hz. An FEI QUANTA 200 Scanning Electron Microscope (Philips-FEI Corporation, Netherlands) was used to study the morphology of monolith. A capillary with the monolith was sectioned into 10 mm segments without sputtering with gold prior to SEM analysis. An analytical balance(Beijing Sartorius Instrumental Limited Company, max = 120 g, d = 0.1 mg) was used to determine the splitting ratio.

Fused-silica capillaries (100 μm inside diameter, 375 μm outside diameter) were purchased from Yongnian Ruipu Optic Fiber Plant (Yongnian, Hebei Province, China). Stainless steel powders (under 500-mesh) was purchased from Beijing Gelubo Alloy Material Limited Company. Butyl methacrylate (BMA), ethylene dimethacrylate (EDMA) and γ-methacryloxypropyltrimethoxysilane (MPTMS) were obtained from New Jersey, USA. Butyl methacrylate and EDMA were extracted with 5-10 % aqueous sodium hydroxide and water before use. 2-Acrylamido-2-methyl-1-propanesulfonic acid (AMPS), azobisisobutyronitrile (AIBN) and 1,4-butanediol, 1-propanol, acetonitrile, tris(hydroxymethyl) aminomethane, toluene, thiourea, benzene, ethylbenzene were purchased from Beijing Bailingwei Chemical Reagent Company and Tianjing Chemical Reagent Company, China. In CEC experiments, a buffer consisting of a mixture of acetonitrile-tris buffer (10 mM, pH 8.0) 90:10, v/v) was used after filtering and degassing.

Preparation of fritless packing column: Prior to frit fabrication, the capillaries with a length of 25 cm and an inner diameter of 100 μm were rinsed with 1 M NaOH for 0.5 h and then with 0.1 M HCl for 0.5 h. After subsequent flushing with H_2O for about 0.5 h, it was dried by passage of nitrogen gas. The procedure for the temporary frit was developed by our lab, which was carried out as follow: A small number of stainless steel powders (under 500 mesh) were placed into a 1.0

mL vial. One end of a 25 cm polyimide-coated fused-silica capillary with different inner diameter (i.d.) was tapped into the vial to allow some small steel particles to be forced into the capillary head. The frit of sintered powders could be formed at the capillary head by burning butane, which was produced by a commercial welding torch (Fig. 1, step 1). Due to the rapid thermal swelling, stainless powder beads would co-fuse and form a "steel arch" structure and the swelling powders could wedged into the capillary wall[29]. The sintered steel powders and the "stone arch" between the steel mesh and capillary wall could endure a high pressure about 60 MPa enough to hold back the stationary phase in the slurry packing. Then the capillary with a prepared frit was slurry packed with 4 μm ODS silica gel in a 90 mm × 1 mm i. d. stainless steel column as slurry reservoir. The other capillary end was connected to the slurry reservoir by means of a screwed joint and a piece of 1/16" PEEK tubing as sleeve. The slurries (20 % stationary phase in toluene/cyclohexanol, 1/1, v/v) were sonicated for 10 min and filled bubble-free into the slurry reservoir using 1 mL syringe. After rapid closing the reservoir, the capillary was immersed into the ultrasonic bath while the capillary with the end fitting was kept outside in a vertical position. The packing procedures could be carried out by the flush of slurry liquid, followed by methanol within 1 h. After slurry packing, the capillaries were forced with two kinds of polymerization mixture, respectively (Fig. 1, steps 2 and 3). One is a silica-based matrix taking MPTMS as the monomer. It contains both methacrylate and alkoxysilane groups, which was often used to prepare a monolithic matrix by photo-polymerization or thermal-polymerization with the help of sol-gel techniques. Previous reports proved that the MPTMS reactant can create a three-dimensional sol-gel network and it can grow in the vicinity of the capillary walls and become anchored to the inner capillary surface through chemical bonding with the silanol moieties residing along the inner fused-silica capillary surface. In addition, the break up of carbon-carbon double bonds (C=C bonds) of molecules in the monomer due to microwave irradiation could lead to polymerization quickly[30]. By this process of destroying carbon-carbon double bonds, the molecule itself becomes highly reactive and links itself to another highly reactive molecule. The procedures to prepare two typical capillary columns were carried out as follows: For column A, a monomer stock solution, a mixture of 750 μL MPTMS, 22.5 μL 1.0 M hydrochloric acid and 225 μL water, was stirred for 0.5 h at room temperature in the dark. Toluene (170 μL) was added to 30 μL of the monomer stock solution and stirred for 0.5 h at room temperature. About 0.445 mg AIBN was added to the above mixture and stirred for 2 h at room temperature. Another is a methacrylate-based matrix with outstanding chemical stability in a broad pH range, which have widely used for the separation supports of monolithic and packing columns. For column B, the entrapped mixture was made up of 270 μL BMA as a functional monomer, 150 μL EDMA as a cross-linking agent, 0.0024 g AMPS as the EOF modifier, porogenic solvents including 217 μL 1,4-butanediol, 400 μL 1-propanol and 60 μL water and 0.004 g AIBN (1 % monomer, wt. %) as an initiator. The above entrapment solutions were used referred to the recipes of relative monolithic columns[14,30] and should be mixed ultrasonically into a homogenous solution, respectively, followed by purging with nitrogen for 10 min before a small part of the reactant solution was introduced into each capillary, respectively. Here, it should be especially noted that the capillary internal walls for column B was modified by MPTMS solution through a procedure described elsewhere[30]. Thereby, Si-O-Si-C bonds could be formed between the capillary wall and the reactive groups of entrapping mixture which are available for subsequent attachment of reactant to the wall.

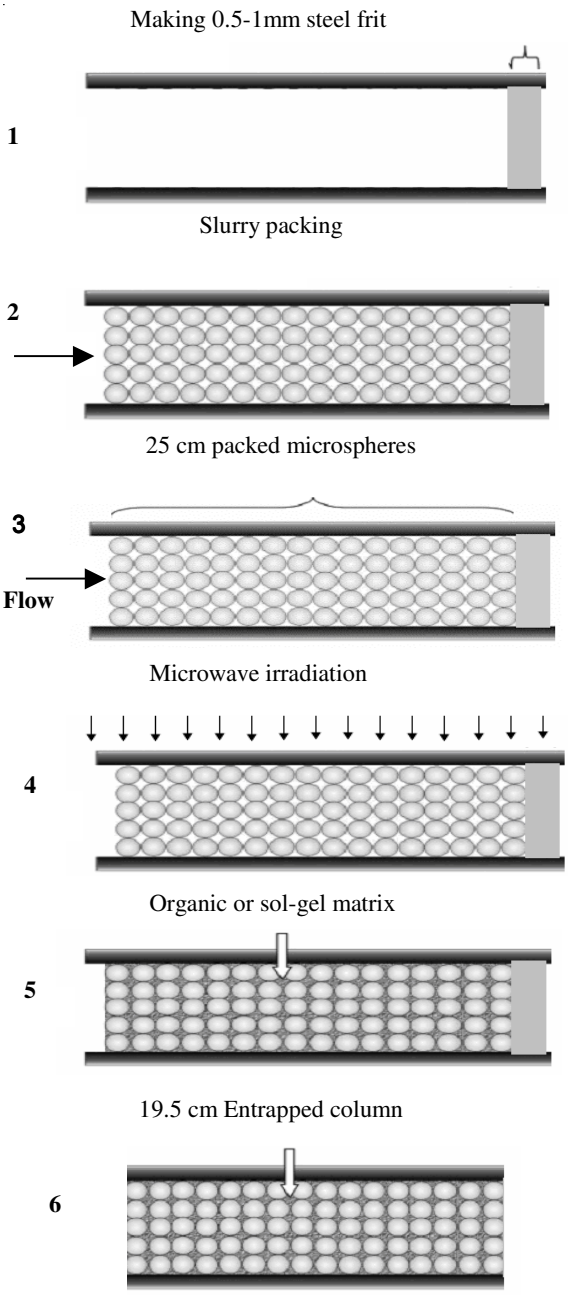

Fig. 1. Schematic column preparation procedures

After several column volumes of the polymerization mixture had passed through the capillary, the HPLC pump was turned off and the outlet of the capillary was sealed with a septum. Then, each 25-cm-long microsphere-packed capillary was exposed to a home microwave oven for microwave irradiation about several minutes, which can facilitate microsphere

entrapment (Fig. 1, step 4). The temporary retaining frit was then cut off, then methanol or acetonitrile was flushed through the capillary column back and forth with an HPLC pump to remove unreacted monomer, porogenic solvent and non-entrapped microspheres and the column was cut to the desired length (19.5 cm) and coupled to an empty capillary segment on which a detection window was made for next use (Fig. 1, steps 5 and 6). To prevent the prepared columns from drying, they were stored by carefully immersing both ends of the capillary into vials containing acetonitrile solvent. Prior to micro-HPLC and CEC experiments, the prepared capillary should hyphenate with another empty capillary by a commercial connector (Upchurch Company, USA). A window about 1 cm length was created by scraping the polyimide coating of the empty capillary with 150 mm × 100.i.d. (from the window to the capillary outlet = 8.5 cm). The hyphenated capillaries were flushed with mobile phase for 0.5 h before experiments. A preconditioning step was performed by applying a stepwise increase in voltage up to 30 kV over the column, until a stable current was observed in the mode of CEC.

RESULTS AND DISCUSSION

Chromatographic evaluation using the self-installed capillary-HPLC system: A capillary-HPLC system was successfully constructed for micro-column separation by using a simple eluate splitting system, a self-preparation of fritless column and an on-capillary column detector[29]. Mobile phase micro flow rates obtained by splitting the main solvent stream generated by a conventional liquid chromatography are particularly convenient as a result of the inherent simplicity of the system and the great reliability and reproducibility of standard pumping devices. In general, sample injection was carried out by a nano-injector in a commercial capillary-HPLC instrument. In present experiment, eluent flow through the capillary column was controlled by a custom built adjustable flow splitter based upon a T-piece connector with a capillary. The backpressure enforced on the separation column could be adjusted by the change of another capillary inner diameter and length. The connection between the T-piece and two capillaries was carried out using a PEEK sleeve (405 μm i.d., 1.6 mm o.d., Upchurch Scientific Co. Ltd., USA) and a screwed joint, respectively. Splitting ratio was calculated by weighing the eluate from the outlets of two capillaries, which were collected in a sealed vial, respectively. Usually a capillary column needs tubing at the end of the column for connecting with the detector. However, the connection and analytical cell can inevitably induce a dead volume in a conventional HPLC system. Considering the detrimental influence of the dead volume, an on capillary column UV detector was used in the developed capillary HPLC system. Furthermore, the actual injection volume can be calculated by determining the splitting ratio (the weight of eluent from the splitting capillary to that from the separation column).

The effect of flow rate on the theoretical plate number of each analyte (column A) was plotted by the developed capillary LC system. All analytes eluted in the order of thiourea, benzene, toluene, ethylbenzene. With the flow rate displayed in the collected software increased from 0.6000-1.3000 mL/min, the actual flow rate in the capillary tube was change in the range of 0.243-0.572 μL/min and back pressure of the prepared column increased from 2.6-6.2 MPa. The relevant splitting ratios were changed in the range of 2037-2875. Baseline separation of all model compounds could be achieved in the optimal experimental conditions. The curves were plotted using 6 points, with each point repeated at least three times, respectively. The largest theoretical plate number (N) was obtained up to 50000/m for the retained component (Fig. 2), with the constant mobile phases (ACN/H_2O = 90/10, v/v). Fig. 3 showed that the N values were changed with the percentage of mobile phases with a constant flow rate of 0.900 mL/min. The curves were plotted using 6 points, with each point repeated at least three times, respectively. With the acetonitrile percentage was increased from 70-95 % with a stepwise change of 5 % ACN, the eluent strength of mobile phases increased and retention time for the last analyte was reduced from about 9.1-4.6 min (Fig. 4), whilst no obvious change of column efficiency was observed. Back pressure of the prepared column decreased from 6.0-3.8 MPa because of the lower viscosity of acetonitrile and the relevant splitting ratio were changed in the range of 2101-3070.

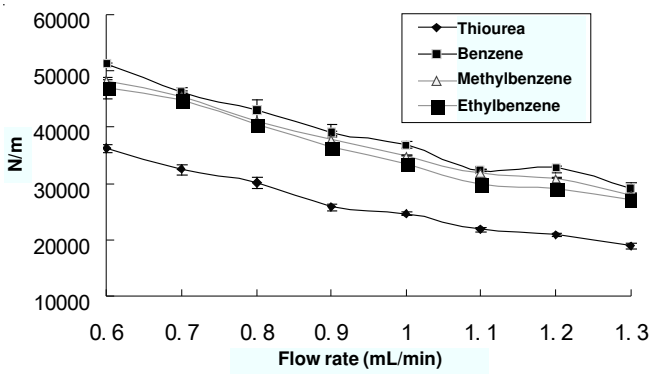

Fig. 2. N-u curves for the model analytes

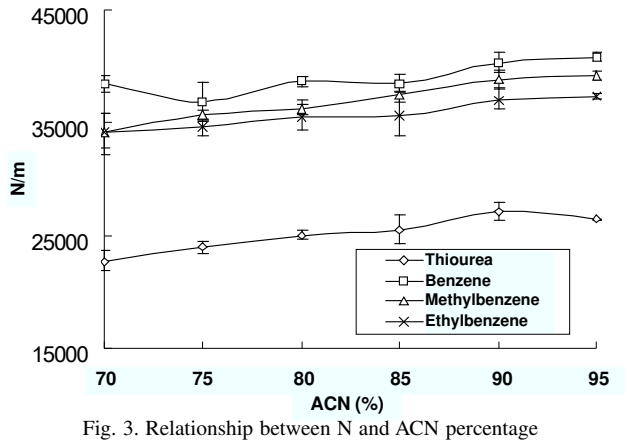

Fig. 3. Relationship between N and ACN percentage

Identical investigation of chromatographic performance was carried out using column B and baseline separation of four model analytes was obtained with the same peak orders as column A. The effect of flow rate on the N values of each analyte was plotted with the constant mobile phases (ACN/H_2O = 90/10, v/v). With the flow rate displayed in the collected software increased from 0.6000-1.3000 mL/min, the actual

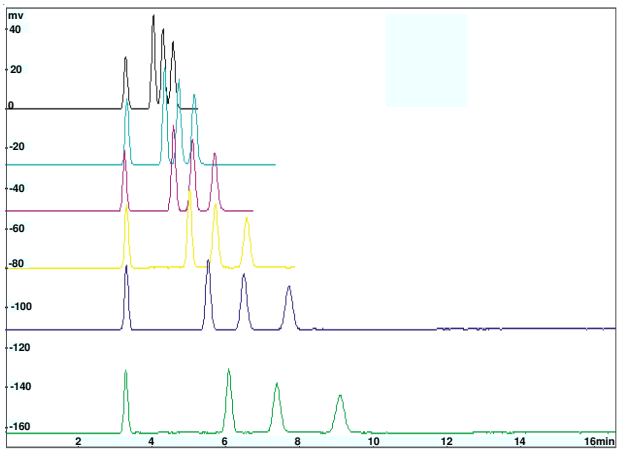

Fig. 4. Typical chromatograms with the change of mobile phases

flow rate in the capillary tube was change in the range of 0.239-0.436 μL/min and back pressure of the prepared column increased from 2.7-6.4 MPa. The relevant splitting ratios were changed in the range of 2504-3274. The curves were plotted using 8 points, with each point repeated at least three times, respectively. N-u curves for the model analytes were shown in Fig. 5. The highest theoretical plate number was obtained up to 40000/m for the retained component, respectively. Fig. 6 showed that the N values were changed with the percentage of mobile phases with a constant flow rate of 0.900 mL/min. The curves were plotted using 6 points, with each point repeated at least three times, respectively. With the acetonitrile percentage was increased from 70-95 % with a stepwise change

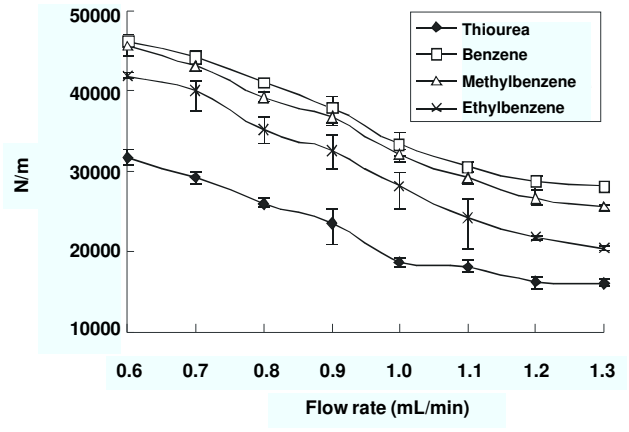

Fig. 5. N-u curves for the model analytes

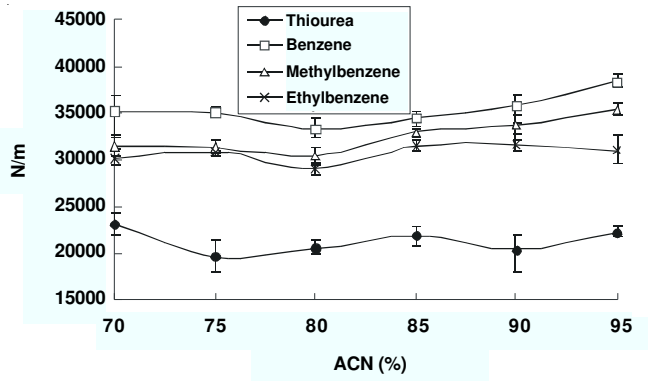

Fig. 6. Relationship between N and ACN percentage

of 5 % acetonitrile, the eluent strength of mobile phases increased and retention time for the last analyte was reduced from about 12-5.2 min, whilst no obvious change of column efficiency was observed. Back pressure of the prepared column decreased from 7.0-4.0 MPa and the relevant splitting ratios were changed in the range of 2585-3414.

The prepared columns A and B were selected for the investigation of stability of the immobilized stationary phases. Identical experimental conditions were operated using the same displayed flow rate of 0.9000 mL/min and mobile phase percentage (ACN/H_2O = 90/10, v/v). The results of the inter-day and intra-day precisions both indicated the good run to run reproducibility could be easily obtained. For the inter-day precisions using column A, the relative standard deviations (RSD) of the migration time for each analyte and resolution for each peak pair (n = 6) were calculated between 0.88-0.96 % and 0.84-1.31 %, respectively. For the intra-day precisions using column A, the RSD of the migration time and resolution (n = 18, three days) were calculated between 0.95-1.06 % and 1.21-1.56 %, respectively. Furthermore, for the inter-day precisions of column B, the RSD of the migration time and resolution (n = 6) were calculated between 0.71-0.76 % and 0.50-0.77 %, respectively. For the intra-day precisions of column B, the RSD of the migration time and resolution for each analyte (n = 18, three days) were calculated between 3.00-3.57 % and 1.89-2.82 %, respectively. It should be noted that the immobilized stationary phases within the columns could endure the quick flush, column regeneration or equilibration, operation under the high pressure, which showed its enough mechanical strength for the long usage in micro-HPLC.

Evaluation of electrochromatographic performance of the fritless columns: For the promising columns, the simplified van Deemter curves in CEC were comparatively plotted. A reversed-phase mechanism was observed for the analyte separation. Solution partitioning between the mobile and stationary phases is the main mechanism responsible for their retention of the model compounds. The migration order of the columns is similar to that of reversed-phase chromatography, the analytes with larger molecular weight or more hydrophobic analytes were eluted later than the analytes with smaller molecular weight or more hydrophilic. Although columns A and B possessed similar chromatographic performances in the range of 40000/m ~50000/m, apparent differences of column efficiency were observed in the application of CEC separation (Fig. 7). In CEC mode, the applied voltage between 16 and 30 kV and the applied gas pressures between the inlet and outlet of 1.2 MPa were operated throughout all experiments, the linear velocities in the range of 0.43-1.04 mm/s were obtained using column A and a higher column efficiency with a theoretical plate height in the range of 13.5-15.4 μm was achieved, corresponding to the largest theoretical plate number of 74000/m for the last eluted (ethylbenzene). Whilst the slower linear velocities in the range of 0.38-0.74 mm/s were obtained for column B and a lower column efficiency with a theoretical plate height in the range of 80-138 μm was achieved. Typical CEC separation of the model analytes using columns A and B was shown in Fig. 8. Apparent difference of column efficiency for columns A and B was probably attributed to the different structures of immobilized matrix. Both inorganic sol-gel and

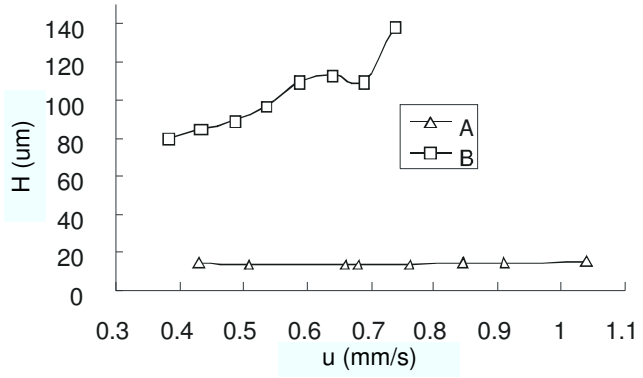

Fig. 7. Van deemter plots of ethylbenzene using columns A and B

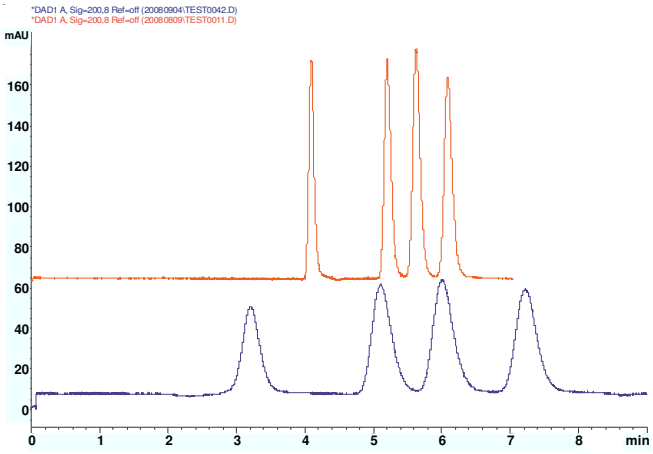

Fig. 8. Comparative CEC separation using columns A and B. Experimental condition: injection 10 kV* 5 s, 20 kV, both ends 1.2 MPa, 14 ºC, buffer: AcN/tris (10 mmol/L, pH = 8.0), 90:10 v/v

organic solutions formed the inorganic and organic macromolecules and surrounded on the surface of particle sorbents under the microwave irradiation. Some rudimental silanol group for column A surrounded on the surface of inorganic polymer, which led to stronger EOF in the eletro-driven separation. Column B modified by MPTMS solution created less zeta potential on the surface of inner wall, which prolonged the separation time and caused apparent peak broadening in electro-driven separation. The run to run reproducibilities of columns A and B were investigated using the identical separation voltage of 20 kV. For column A, the RSD values of the migration time for each analyte and resolution for each peak pair (n = 8) were obtained between 0.74-0.77 % and 0.50-1.69 %, respectively. Whilst the RSD values of the migration time and resolution (n = 8) were obtained for column B between 2.91-6.75 % and 7.13-9.66 %, respectively.

Characterization of the fritless columns: Strict control of the morphology of the separation beddings is important that provides a good separation efficiency and a low resistance to flow in both the electro-driven and pressure-driven separation modes. Entrapped-microsphere columns have the advantage of immobilizing the chromatographic material, preventing the formation voids within the column and eliminating the need for either an inlet or outlet frit. Generally, the original non-entrapped ODS microspheres usually behaved with a clean smooth surface with an average size of 4 μm. Fig. 9 showed the changes of the appearance of the ODS microspheres in the capillary after polymerization. After immobilization using the

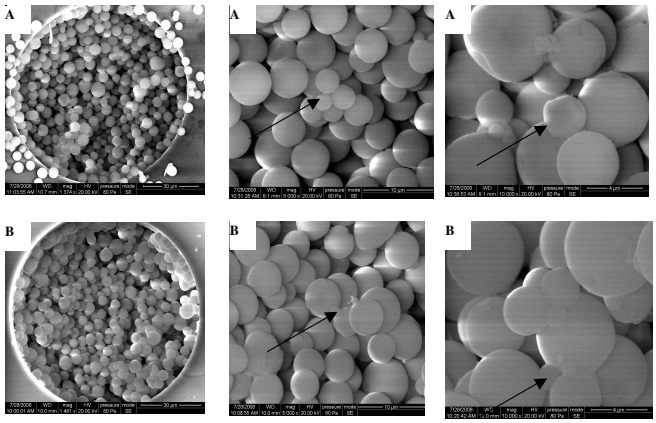

Fig. 9. SEM photopgraphs of the fritless columns in different magnifications

sol-gel and organic polymers, there were some noticeable changes observed on the surface at sphere-sphere (see the arrowhead in Fig. 9A and 9B) and sphere-capillary wall contact points where inorganic or organic polymer "bridges" were observed, which "webbed" them together. Furthermore, some small pits were apparent on the surface of the microspheres, which was believed to be caused by the removal of adjacent microspheres when preparing the cross-section sample for SEM (Fig. 9A). The formed polymers on the surface of ODS particles with a size less than 2 μm could both embedded the voids among the ODS microspheres and interconnected within the capillary tube, so that the fritless columns were stable and strong enough to be operated continuously up to a pressure of 35 MPa without column damage. On the other hand, the prepared capillaries could be cut to any desired length for both electro-driven and pressure-driven separations with the minimized alternation of the properties of ODS sorbent[27,29].

Conclusion

An easy and fast method to retain the C_{18} particles in place using MAPMS and polymethacrylate-based matrix has been initially attempted, with the polymerization time to be shorten to several minutes. Bubble formation is not observed during any of the chromatographic and electrophoretic runs. The resulting separation beddings are well-suited for the separation of a variety of neutral compounds in both micro-HPLC and CEC. Our intention in this technical note is simply to demonstrate that our approach is valid to prepare the fritless capillary column quickly. It exhibited good potential instead of the traditional thermal and UV light initiation methods. The main advantage of microwave irradiation as an energy source is its short polymerization time, simplicity and no need to use brisk and expensive UV transparent capillaries. Furthermore, some application such as the immobilized material for HPLC-MS and solid phase micro-extraction are going on in our laboratory.

ACKNOWLEDGEMENTS

The financial supports by the Scientific Research Foundation for the Returned Overseas Chinese Scholars, State Education Ministry and the National Natural Science Foundation of China (Nos. 30070506, 30771436) are gratefully acknowledged.

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CARBON PIONEERS WIN NOBEL

Stockholm - Two Russian-born scientists, Andre Geim and Konstantin Novoselov, won the 2010 Nobel Physics Prize Tuesday for pioneering work on graphene, touted as the wonder material of the 21st century.

Both laureates began their careers as physicists in Russia but now work at the University of Manchester in Britain. Geim holds Dutch nationality and Novoselov is both a British and Russian national.

The Swedish Academy of Sciences hailed graphene - “the perfect atomic lattice” - for its glittering potential in computers, home gadgets and transport.

It lauded Geim, 51, and Novoselov, 36, for having “shown that carbon in such a flat form has exceptional properties that originate from the remarkable world of quantum physics.”

The prize honors a breakthrough that paved the way to graphene, a form of carbon touted as the next-generation super-material.

Just one atom thick, it is the world’s thinnest and strongest nano-material, almost transparent and able to conduct electricity and heat.