Polydimethylsiloxane Connection for Quartz Microchips in a High-Pressure System

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An interconnecting technique, the "PDMS connection method", for quartz microchips in a high-pressure system is presented. The connection between quartz microchips is an essential technology for modular microfluidic devices, such as microchip-HPLC. PDMS was applied to the seal material, being spread on the seal side of the chips, and set into the metal housing. The characteristics of the PDMS connection method concerning pressure resistance and the extension of the peak were examined. The experimental results showed a good seal at 5 MPa, which seem to be sufficient for realizing microchip-HPLC utilizing a monolithic silica capillary column as a separation medium. The influence of the extra column effect on chromatographic separation was almost the same as in the case using a commercial union fitting. In addition, the PDMS connection enabled the detachability of chip-based modules with user-friendliness. Our experimental findings suggest that the novel PDMS connection method can possibly be applied as a generic technology in high-pressure μ TAS.

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Introduction

Recently, micro total-analysis-systems (μ -TAS) have attracted a great deal of attention as a possible means for increasing the throughput and mass sensitivity of analytical procedures via miniaturization.^{1,2} A variety of microdevices have been constructed. Compared to conventional instrumentations, microdevices offer the means for handling small sample amounts with no excessive dead volumes. The microchip devices used in recent studies are roughly classified into two types: (1) a total system on a chip; and (2) an integrated system of several modules, each of which individually has specific functions. The typical system of a device-on-a-chip is microchip electrophoresis, where a huge number of studies have been reported.³⁻⁸ However, in the case of miniaturizing further complicated systems, such as high-performance liquid chromatography (HPLC), the approach of integrating several modules having specific functions is considered of value. To integrate modular microchips, the interconnection method is one of the key technologies. Several studies have been reported for miniaturizing HPLC.⁹⁻¹² A common technique for integrating modules is to connect fused-silica capillary tubes between modules. However, in the case of interconnections between microfluidic devices by capillary tubes, they have the disadvantage of a dead volume, which leads to reduce the chromatographic separability in HPLC analysis.

In this paper, we describe a novel "PDMS connection

method" for coupling quartz microchips. The essential features of our method include (1) resistance to high pressure; (2) detachability of chip-based modules with user-friendliness; and (3) no dead volume inside the seal part. First, the PDMS connection method was characterized, and then they were applied to integrate modules for realizing a micro-HPLC.

Experimental

Chemicals and reagents

Ethylbenzene was supplied by Kishida Chemicals (Osaka, Japan). Butylbenzene and amylbenzene were purchased from Tokyo Kasei Kogyo (Tokyo, Japan). Polydimethylsiloxane elastomer (PDMS; Sylgard 184) was obtained from Dow Corning Asia (Tokyo, Japan). Acetonitrile used for the eluent was of HPLC grade, and was purchased from Kishida Chemicals. Deionized water was prepared with a Milli-Q system (Nihon Millipore Kogyo, Tokyo, Japan). All other reagents were of analytical grade, and were obtained from Kishida Chemicals (Osaka, Japan).

Apparatus

Micro-HPLC analyses were performed using a MP-711 micro-flow pump (GL Sciences, Tokyo Japan) and a MU-701 UV-Vis detector (GL Sciences) that was equipped with a long-optical-path flow cell (4 mm of optical path length). The sample injector was a Model C4-1004-05 injection valve (sample volume, 0.05 μ L; Valco, Schenkon, Switzerland). Chromatographic separation was typically performed on a MonoCap for a fast-flow monolithic silica capillary column

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Fig. 1 Schematic diagram of modular microchips integrated by the PDMS connection method.

Fig. 2 Schematic diagram of modular microchips for examining the influence in the case where chips were connected by the PDMS connection method.

(150 × 0.2 mm i.d.; through-pore size, 2 μm; meso-pore size, 15 nm; skeleton size, 1 μm; GL Sciences). A Model P779-01 nanotight union (0.05 inch of thru-hole and 8 nL of inner volume; Upchurch Scientific, USA) was used to connect capillaries, and Model F230 poly ether ether ketone (PEEK) tubing sleeves (Upchurch Scientific) to connect capillary to a standard 10–32 port. Data acquisition was performed using Elite (GL Sciences).

Preparation of PDMS for the connection of microchips

In order to connect chips, PDMS was prepared according to the following procedure: a 11:1 combination of PDMS elastomer and curing agent was sufficiently mixed, and then thoroughly degassed. The polymer mixture was cast into a mould, or was spread on the seal side of the chips, and then cured for 6 h at 120°C in an oven.

Fabrication of quartz microchips

Techno Quartz Inc. (Yamagata, Japan) kindly produced and provided all of the quartz microchips used in this study. The microchannels (typically 100 μm width, 100 μm depth) were diced into a quartz wafer by using a dicing saw; taper-connection ports were made by using a drilling machine. Two quartz wafers were welded by the following procedure: each grinding side of the chips was matched, and then heated at high pressure in an electric furnace.

Results and Discussion

Connection between quartz microchips by the “PDMS connection method”

A new interconnecting method between quartz microchips,

Fig. 3 Comparison of the peak shapes between the PDMS connection method and the union fitting. The chromatographic condition is as follows: separation column, Monocap® for fast flow (150 × 0.1 mm i.d.); eluent, 80% methanol; flow rate, 2 μL/min; column temperature, ambient; detection, UV 210 nm; sample injection volume, 0.05 μL; sample, each 0.11 μL/mL of ethylbenzene ($k = 1.4$), butylbenzene ($k = 2.0$) and propylbenzene ($k = 2.6$).

the “PDMS connection method”, was characterized. Figure 1 shows the typical experimental setup. A mixture of PDMS elastomer and curing agent was spread on each seal side of the chips at a proportion of 15 mg/cm². They were then baked in an oven. Next, the integrated chips were set into a brass housing. Four housing blocks were fixed by tightening screws. Two capillaries with PEEK sleeves were inserted into the each side of the brass blocks for connecting a pump and an UV detector, respectively. The liquid leakage was estimated by pumped pressured water, where the pressure in the system was monitored. The experimental results indicated a good seal under high pressure (> 5 MPa). In addition, the seal performance was not changed after conducting the connection/disconnection procedure twenty times.

Influence of the “PDMS connection method” to a modular microchip device

In the case of integrating modular microchip devices to be employed as a microchip-HPLC, the dead volume and effluent are the essential factors. The influence of the PDMS connection method concerning the extra column effect was evaluated. The influence of the PDMS connection and typical union fittings for micro-HPLC was examined based on the case where an injector, a column and a UV detector were directly connected. Figure 2 shows the experimental setup. Microchip A, which has a microchannel (0.1 mm width, 0.1 mm depth, 30 mm length) and a port, and Microchip B, which was equipped with a monolithic silica capillary column, were coupled by PDMS. Then, a capillary tube was inserted into Microchip A to connect a sample injector. A mixture sample of alkylbenzenes ($R = 2, 3, 5$) was injected for the evaluation. A typical chromatogram is shown in Fig. 3. A value of 50% width and a theoretical plate in the case where the PDMS connection, or the union fitting, were employed as the connection tools, as indicated in Fig. 4. The 50% width and the theoretical plate of the PDMS connection technique were slightly decreased compared with the case using a union fitting. The value of the plate decreased by 5% at 1.4–2.6 of the retention factor (k). However, consideration of the advantage of a one-touch connection with user-friendliness, the PDMS connection method, from a practical viewpoint, obviously has a possibility to be used as the

Fig. 4 Evaluation of the extra column effect by various connection methods.

fundamental technology of micro-total analytical system.

Conclusion

We have developed a simple and effective connection technology, the "PDMS connection method", to hold and interconnect modular microfluidic devices, which is one of the essential technologies for the modular microfluidic devices, such as microchip-HPLC. The obtained results suggest that the interconnection of microchips by a PDMS connection has the ability of 5 MPa proof pressure, which is a sufficient value to realize microchip-HPLC utilizing a monolithic silica capillary column as the separation medium. In addition, the PDMS connection technology enabled a detachability of chip-based modules with user-friendliness. In addition, the influence of the PDMS connection technique to the extra column effect was similar to that of the common HPLC fittings. Our experimental findings suggest that the novel PDMS connection method has an application possibility as generic technology in a high-pressure μ TAS. Further, the PDMS connection could be applied to microchip-HPLC with a multichannel column.

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