MICRO PRECONCENTRATOR FOR HANDHELD MONITORING OF WATER QUALITY

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ABSTRACT

The detection limit of analytical systems can be enhanced by utilizing a preconcentration step. We report on the development of a 12 mm × 12 mm silicon-on-glass micro preconcentrator (µPC) consisting of more than 3600 square shaped µ-pillar structures embedded within a microcavity for handheld water quality monitoring systems. The µPC was coated with a novel cobweb configuration of Tenax-TA adsorbent films. The µPC was evaluated experimentally by flowing (1, 5, and 10 mL) water samples spiked with 800 ppm benzene at room temperature. Results demonstrated that the µPC was capable of extracting and concentrating benzene from water samples.

KEYWORDS: Water monitoring, MEMS, preconcentrator, µGC, Tenax TA

INTRODUCTION

Water resources are susceptible to both natural and man-made contamination. Water contaminants are grouped into microbiological, radioactive, inorganic, synthetic organic, and volatile organic compounds (VOCs) [1]. These contaminants, specifically VOCs, pose serious health effects which include liver and nervous system diseases and cancer [2]. Chemical analysis of water supplies requires samples to be transported to off-site laboratories. The cost of such analysis can range between $100 to $1000 per sample and the integrity of the analyses can be compromised during sample collection, transport, and storage [1]. Thus, there is a need for inexpensive and accurate handheld water monitoring systems.

Several technologies for handheld environmental monitoring have been reported which include spectroscopic [3, 4] and chromatographic systems [5-9] for analyzing gaseous and liquid samples on-site. However, reports on systems that can process liquid samples are very limited. This paper report on the development of micro preconcentrator (µPC) consisting of square shaped µ-pillar structures embedded within a microcavity, as shown in Fig. 1, aimed for handheld water quality monitoring systems.

![Figure 1: 3D rendering of the silicon-glass µPC](image)

FABRICATION

The fabrication process of the presented device is shown in Fig. 2. A 6 µm-thick layer of AZ9260 photoresist was used to pattern a 500 µm silicon wafer with the µPC layout. Then, the wafer was anisotropically etched using deep reactive ion etching (DRIE) technique to achieve vertical microstructures (pillars). The etching process parameters were configured to achieve a depth of 300 µm. The wafer was then exposed to oxygen plasma for 20 minutes to remove any residual passivation polymers from the etching process. This is an important step since any fluorocarbon residuals on the sidewall can compromise the adsorbent coating stability.

Adsorbent deposition was achieved by utilizing our recently reported adsorbent deposition method to deposit Tenax-TA on the µ-pillars [10]. The cavity was filled with Tenax-TA solution (25 mg/mL in dichloromethane). Then, before the solvent evaporates, drops of 2-propanol were added into the cavity. Cobweb Tenax-TA evolves when 2-propanol contacts Tenax-TA solution. The cobweb structure anchors to the µ-pillars once the solvent evaporates as shown in Fig. 3. The µPC was then sealed by anodic bonding the silicon substrate to a Pyrex wafer under 1250 V of applied electric field at 250 °C to avoid damaging the polymer.
The µPC was evaluated experimentally by flowing water samples spiked with benzene through it. It is noteworthy that benzene has been classified by the US Department of Health and Human Services (DHHS) as a human carcinogen. Benzene has 0.8 g/L solubility in water at room temperature. Thus, benzene was added to deionized water and the solution was left for 24 hours to equilibrate. Then, water samples were extracted from the bottom of the container to avoid the insoluble benzene floating on the top of the water surface. A 1 µL sample of the solution was injected directly to a flame ionization detector (FID) to determine the detector response to the unpreconcentrated sample. It should be noted that the FID is water insensitive detector. Subsequently, the µPCs chip was connected to the FID through a zero dead volume valve. While the valve in waste position, the chip was loaded with three water samples volumes (1, 5, and 10 mL) at room temperature. After every load, the chip was dried by purging it with helium for 5 minutes. Then, the valve was switched to FID position and the device was heated to 250 °C at 100 °C/sec to desorb the collected benzene from the different volumes of the water sample. The flow rate through the µPC was set to 1 mL/min.

**RESULTS AND DISCUSSION**

Fig. 4 shows the expected FID response to the concentrated benzene from the 10 mL water sample. The area under the curve of the FID signal is directly proportional to the concentration of the desorbed sample. The peak width at half height

![Figure 4: FID response to the concentrated benzene from the 10mL water sample.](image-url)
was about 10 seconds. Fig. 5 compares the FID response to the three different sample volumes including the 1 \( \mu \)L sample of unpreconcentrated water sample. The amount of benzene collected by the device was proportional to water volume flown through it. The experimental results showed that the \( \mu \)PC was capable of extracting and concentrating benzene from water samples.

![Figure 5: FID response (peak area) to the three different sample volumes along with the 1 \( \mu \)L sample of unpreconcentrated water sample](image)

**CONCLUSION**

The reported MEMS device was capable of capturing VOCs from water. This development would be of significant benefit to local authorities, environmental agencies, and environmental researchers as it promises sensitive and quantitative on-site monitoring at a low cost.

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**REFERENCES**


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