Preconcentration and Detection of Chlorinated Alkanes and Alkenes

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Abstract
Remote and automated detection of chlorinated compounds in subsurface aquifers is crucial to superfund monitoring and environmental remediation. Current monitoring techniques involve the use of expensive laboratory instruments and trained personnel. The use of a chemcapacitive detector array presents an attractive option for the unattended monitoring of these compounds. Five preconcentrator materials were exposed to common target compounds of subsurface remediation projects (trichloroethene, trichloroethene, dichloroethene, benzene, and perchloroethene). Rapid heating of the tube caused the collected, concentrated effluent to pass over the surface of a chemcapacitive detector array coated with four different sorbent polymers.

A system containing a porous layer polymer and the sensor array was subsequently used to sample the analytes injected onto sand in a laboratory test, simulating a subsurface environment. With extended collection times, effective detection limits of 5 x 3 ppbv were achieved for trichloroethene in the vapor phase. The effects of the preconcentrator material structure, the collection time, and sensor material on the system performance were observed. The resultant system presents a solution for remote, near-real-time monitoring of chlorinated organic compounds and other volatile organic compounds in a soil matrix.

Experimental: Test System

| Analytes Tested: | • benzene | • 1,1,2-dichloroethylene (1,1,2-DCE) | • trichloroethylene (TCE) | • 1,1,2-trichloroethene (TCA) | • perchloroethylene (PCE) | • All tests in dry air |

Experimental: Preconcentrator

<table>
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<th>Solvent materials used in Preconcentrators</th>
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<td>PIM 1</td>
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</table>

Extended collection times

| TCA (ppbv) | 6.2 ± 1.4 | 6.2 ± 1.4 | 6.2 ± 1.4 |
| PCE | 1.0 ± 0.3 | 1.0 ± 0.3 |

Experimental: Detectors

Detector coatings:
- PVAC: poly(vinylacetate)
- PEI: poly(ethylene imine)
- PEVA6E: poly(ethylene co-vinylacetate)
- PCH: poly(epichlorohydrin)
- ADIOL: difluoroalcohol siloxane
- HC: hyperbranched difluoroalcohol carbosilane
- PIB: Polyisobutylene

Results: Preconcentration

Comparison of preconcentrator materials
- TCE (127 ppM) collected for 8 min.
- Comparison of 5 analytes using PM-1: TCE (127 ppM), benzene (160 ppM), PCE (28 ppM).

Results: Chromatography

Prototype Mini-GC: uses air as the carrier gas, directly heated column, and chemcapacitive array as the detector.

Summary
This work represents the initial efforts towards an unattended sensor to track subsurface contamination of chlorinated organic compounds in near real-time while logging and transmitting contaminant concentrations. Such a system would be used in tandem with geophysical and other methods, to provide comprehensive contamination management. Use of preconcentrator and chemcapacitive sensor array with an improved preconcentrator packing material and sensor materials resulted in the detection the target analytes at levels below regulated levels. The modular nature of the system will allow future systems to be tailored to other analytes by changing the structure of the preconcentrator material and the sensor array coatings. In addition, the preconcentrator may be integrated with more complex devices for solving other environmental monitoring problems, such as vapor intrusion monitoring or fence-line monitoring.

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