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A wireless hybrid chemical sensor for detection of environmental volatile organic compounds

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Abstract

A hybrid sensor for monitoring volatile organic compounds (VOCs) in air is developed. The device combines two orthogonal sensing principles, selective molecular binding with a microfabricated quartz tuning fork detector and separation of analytes with a column. The tuning fork detector is functionalized with molecular imprinted polymers for selective binding to benzene, toluene, ethylbenzene, and xylenes (BTEX), and the separation column provides further discrimination of the analytes for real world complex sample analysis. The device is wireless, portable, battery-powered, and cell-phone operated, and it allows reliable detection in parts per billion (ppb) by volume-levels of BTEX in the presence of complex interferents. The hybrid device is suitable for occupational, environmental health, and epidemiological applications.

Index Terms

Air quality monitoring; environmental monitoring; chemical sensors; wireless sensors; environmental sensors; volatile organic compounds; VOCs

I. Introduction

Toxic volatile organic compounds (VOCs), such as aromatic and alkyl hydrocarbons, have serious environmental and health impacts. Because these VOCs are ubiquitously used in industrial and daily activities, detection and quantification of them are critical for many applications, including occupational health, industrial safety, environmental monitoring, and epidemiological studies. Despite advances in chemical sensor technologies over the past decades, a reliable, real-time, and portable device that can reliably detect VOCs remains a scientific and engineering challenge.

Several standard methods have been established for analysis of VOCs in air. One of the most widely used methods uses an absorption tube or a canister with GC/MS equipment to collect the sample on the field, and then send the tube or canister to a lab, where a trained technician analyzes the sample using GC/MS equipment. These methods are labor intensive and time-consuming, involving multiple steps, typically including sample collection, shipping and storage, use of costly equipment, and data analysis [1], [2]. An alternative approach aiming at overcoming the difficulties of these standard reference methods is to use real-time detection devices. One such device is based on photo ionization detection (PID) [3], [4], which is a broadband detector that detects ionized species with a UV lamp [3], [4]. For selective detection of many toxic chemicals, such as benzene (a carcinogen), the PID alone is inadequate and it must be combined with filters and separation tubes. Although the approach offers real-time results, the selectivity and practical detection limits are limited, making PID devices suitable only for certain industrial applications. Moreover, the PID devices are incapable of simultaneous detection and analysis of multiple analytes.

Another real-time detection approach is the use of colorimetric tubes [5], which rely on a specific binding or reaction between an analyte and an appropriate sensing material. The specific binding leads to a change in the color of the sensing material, which is detected optically. Because the reaction is irreversible, this approach is for one-time use only and not suitable for continuous monitoring of analytes in the environment. A more serious limitation of the colorimetric approach is that it cannot be applied to chemicals that do not react to generate color changes. One such example is benzene, which is carcinogenic but does not interact strongly with common sensing materials.

A more versatile approach is based on portable gas chromatography (GC) technologies [6], [7]. High performance portable GCs have been developed, but the instruments are bulky and expensive, which seriously hinder their applications [6], [7]. In addition, the portable GCs often use carrier gas in cylinders, further limiting their portability and usability [6], [8]. In order to miniaturize GCs, various novel detectors [9], [10], such as arrays of chemiresistor, surface acoustic wave (SAW) [11], [12], and metal oxide -based sensors combined with microfabricated preconcentrators and/or GC separation columns [13], [14] have been developed.

In an effort to overcome the difficulties of the current detection technologies, we have recently demonstrated a hybrid approach that integrates specific binding (e.g., colorimetry) and selective separation (e.g. GC) of analytes [15]. The work confirmed the value of the hybrid approach, however, the sensitivity and selectivity fall short of meeting the needs of environmental monitoring. In the present work, we introduce adsorbent packed preconcentrator that selectively collects and releases analytes, a more sensitive microfabricated tuning fork sensor, an automated heat and flow control with a microcontroller-based circuit, and wireless communication with a cell phone. We have integrated all the components into a single unit that weighs 1.2 lbs, tested its analytical performance, and validated its usability in various real world scenarios. The device can reliably detect a few ppbs of BTEX in complex real samples within minutes, more than three orders of magnitude improvement over the previous work [15]. Furthermore, the improvement of selectivity is demonstrated by discriminating BTEX from other hydrocarbons in a complex gasoline vapor sample.

II. EXPERIMENTAL SETUP

The device consists of three key components: 1) a specific sample collection and preconcentration unit, 2) a sample separation column, and 3) a sensitive, selective and fast sensor (Fig. 1). These components are supported by fluidic and thermal control, signal

processing and data communication. The sensor is based on microfabricated tuning fork coated with molecularly imprinted polymers [16] that can selectively bind with hydrocarbons. The tuning fork together with a low noise circuit allows sensitive detection of analytes. The separation column provides additional selectivity by separating analytes and interferents. Both the sensitivity and selectivity are further enhanced by the selective and efficient preconcentration unit. The synergetic operation of the three components makes it possible to build a miniaturized device with high sensitivity and specificity required for complex environmental monitoring and analysis.

The device is battery-operated and paired wirelessly with a cell phone via Bluetooth connection. To operate the device, a custom application is developed and installed in the cell phone with features such as data analysis, device control, and user interface.

a. Preconcentrator

A preconcentrator is developed and integrated into the hybrid device to increase sensitivity. It also improves the selectivity via selectively accumulating the VOCs of interest at ambient temperature. The targeted VOCs here are hydrocarbons, particularly BTEX, which are highly toxic but difficult to detect with existing portable devices. The preconcentrator consists of a stainless steel tube (1/16-inch diameter and 1-inch length) packed with porous graphitized carbon black, 40/60 mesh Carbopack X (Sigma-Aldrich Co.). It serves as the adsorbent material to provide a large surface area ($250 \text{ m}^2/\text{g}$) with high-density binding sites for the VOCs. A heating wire is wrapped around the stainless steel tube and a circuit to provide joule heating to the wire so that the preconcentrator can reach a temperature of 300°C in one minute to release the analytes adsorbed on the adsorbent.

b. Separation

The device uses a flexibly adjustable column. Depending on applications, the type and length of the column is tuned to maximize the required performance. For example, for fast separation and detection, two short (2 m) columns, UAC-CW (carbowax coated column, Quadrex) and UAC-502 (cyanopropylphenylsilicone coated column, Quadrex) connected in series provide fast and sensitive detection. For separation and discrimination analysis in more challenging scenarios (e.g., presence of complex hydrocarbons interferents), a configuration of a 19-m UAC-502 column was used instead.

c. Detector

One distinctive feature of the hybrid device is the quartz crystal tuning fork detector. In contrast to most GC instruments that use broadband detectors, the tuning fork detector in the present hybrid device is functionalized with a molecularly imprinted polymer (MIP) tuned for selective detection of monoaromatic and alkyl hydrocarbon VOCs [15]-[17]. Briefly, the MIP is a highly cross-linked polystyrene synthesized with biphenyl as template and xylenes as porogen solvent, in which the MIP binding sites are created bind to the target molecules via π - π and van der Waals interactions, leading to high selectivity. The tuning forks are quartz mechanical resonators with a resonant frequency of 32.768 kHz (CITIZEN AMERICA) and a quality factor of about 10000 in ambient air [18]-[20], providing high sensitivity and low detection limit. The crystal is cut such that its thermal expansion is close to zero near room temperature, making the detector relatively immune to temperature changes. The tuning forks are also low power ($1 \mu\text{W}$ maximum) and small ($0.1 \times 0.5 \times 3 \text{ mm}^3$). The intrinsic properties of the tuning forks together with a precision digital counter circuit developed by us can measure a resonant frequency change with 1.8 mHz detection limit, corresponding to $4 \text{ pg}/\text{mm}^2$. This detection limit, combined with the selective MIP, leads to ppb-level detection of VOCs.

d. Fluidic and thermal control

The three components described above are integrated into a single unit and enabled by a fluidic and thermal control system, including a pump (Parker Hannifin Co.), four miniature valves (The Lee Co.), filters, tubing and connectors, and a microcontroller (Texas Instruments Inc.). The hardware components are integrated and optimized to ensure optimal dispersion, separation, and dead volumes associated with the connections between different components. The fluidic and thermal control includes flow rate, valve switching, and temperature control, which are optimized to achieve the best performance. For example, an optimal flow rate of ~ 8 mL/s is found to provide the best sample separation.

The fluidic control includes a key component, a zeroing filter made of activated carbon (Purafil), to generate clean carrier gas from ambient air. This approach eliminates the need of external carrier gas cylinders, which minimizes the weight and size of the hybrid device.

e. Device integration

All of the components mentioned above, together with batteries (three lithium-ion polymer batteries), are integrated into a $12.9 \times 9.9 \times 4.9$ cm³ box. The device weighs a total of 1.2 lbs. It contains a communication circuit that communicates with a cell phone (see Fig. 2). The circuit contains a Bluetooth chip connected to a master microcontroller that reads the sensor signal and other control signals. This Bluetooth chip is paired with a cell phone for communication and data transmission. Fig. 2 shows a prototype hybrid device along with the Smartphone user interface (inset). The integration of all components leads to a truly portable and self-contained hybrid device for trace level VOC detection in complex mixtures.

f. User interface and operation of the device

The user interface is a cell phone with a custom application developed in a Motorola Q phone. The user interface controls the device, receives, processes, displays, and stores data from the device. Once the data are acquired, it also allows further data transmission via seamless wireless cell phone network.

The user interface activates and operates the device according to the following steps: 1) Preconcentration, 2) Desorption, 3) Injection, 4) Analysis, and 5) Cleaning.

1. Preconcentration stage: The pump pulls air sample (e.g. indoor or outdoor air) into the preconcentrator at a constant flow rate, and the VOCs are adsorbed in the preconcentrator. The preconcentration time duration is adjustable, based on the VOC concentration. In general, the preconcentration time is inversely proportional to the analyte concentration. Once the preconcentration period is finished, the valves are switched so that scrubbed clean air is purged into the separation column and the tuning fork detector registers a baseline.
2. Desorption stage: After the preconcentration stage, desorption stage starts with heating the preconcentrator to 300°C for 1 minute, which releases the VOCs to gas phase.
3. Injection stage: After desorption stage, the valves are switched to an injection stage, and scrubbed (clean) air passes through the heated preconcentrator and takes the VOC vapors to the separation column. The injection stage lasts 15 seconds.
4. Analysis stage: After the 15-sec injection, the analysis stage follows with the separation of the sample components in the column via a clean air carrier flow. Concurrently, the tuning fork detector measures the sensor signals and sends them to the cell phone. As the sample components exit the separation column, detection is performed from the recorded peaks in a chromatogram (Fig. 3(b)).

5. Cleaning stage: After the analysis stage is finished, the device is cleaned by heating the preconcentrator again and flushing the system with scrubbed air so that the device is ready for the next testing event.

It is important to mention that the above-described operation is fully automatic, and can be initiated by simply pushing a button in the cell phone application.

III. Results and Discussion

We have built a fully integrated and functional hybrid device, performed analytical validation and calibration of the device, and carried out preliminary field tests in different real world scenarios to demonstrate the device's usability and unique capabilities.

A. Analytical Performance and Calibration Preconcentration

The preconcentrator traps the VOCs in the sample at room temperature and desorbs the trapped VOCs upon heating to $\sim 300^{\circ}\text{C}$. An important parameter that describes how efficient the preconcentrator traps and desorbs the analytes is preconcentration factor. In our hybrid device, the factor is defined as the ratio of the tuning fork sensor response of a sample injection using the preconcentrator to that of a direct sample injection without using the preconcentrator. Fig. 3(a) shows the tuning fork sensor response of a direct injection of 10 ppm BTEX sample, and Fig. 3(b) shows the response of a 20 ppb BTEX injection after 20 minutes of preconcentration. Taking benzene as an example, the response of a direct 10 ppm sample injection is -0.075 Hz , and the response of preconcentrated 20 ppb sample is -0.12 Hz , which is equivalent to a direct 16 ppm sample injection, and the preconcentration factor is then equal to $16\text{ ppm}/20\text{ ppb} = 800$. The corresponding preconcentration factor is around 800 for all the BTEX components. This large preconcentration factor not only reflects the high trapping efficiency, but also is a result of high desorption efficiency. We have tested and found that desorption efficiency is as high as 99.2%, which is important for repeated detection and analysis of samples. Chemical sensors based on specific molecular binding in general lack this capability for repeated measurements.

Separation—The first configuration (with a shorter column) provides a fast gas chromatography measurement, while ensuring sufficient separation capability for detection of BTEX in most samples, such as ambient air. As shown in Fig. 3(a) and (b), such a short-column configuration is capable of separating benzene, toluene, ethylbenzene and isomers of xylene within 200 seconds. It is important to mention that the slight differences in the elution times between Fig. 3(a) and (b) are due to the different injection configurations. Fig. 3(a) is a direct BTEX injection without preconcentration, while Fig. 3(b) is BTEX injection with preconcentration. The theoretical plates number of the short column in our hybrid system is 340 (calculated using the elution time and half peak width of toluene). In some cases, such as petroleum refinery industry, benzene and n-hexane present similar mass percentage in petroleum. They both consist of 6 carbons and their boiling point are close (80.1°C for benzene and 68.9°C for n-hexane), but their impacts on human health and environment are very different. Benzene is a proven carcinogen while n-hexane is hazardous to human nervous system. It is very important for a chemical detector to be able to discriminate these two compounds. In order to meet this challenge, we used a 19 m column in the hybrid device (without increasing the size of the device), which allows us to quantify benzene concentrations in the presence of n-hexane (interferent). Fig. 4 shows the separation of n-hexane and benzene. Fig. 6(a) shows the chromatogram of BTEX mixture using the 19 m configuration. This 19 m column provides about 8000 theoretical plates in this hybrid system (calculated using the elution time and half peak width of toluene).

Detection—The dimensions of the tuning fork sensors used in the present work are 2.48 mm × 0.27 mm × 0.12 mm, which are smaller than the ones used in our previous work [15], which have dimensions of 4 mm × 0.6 mm × 0.35 mm. The smaller tuning forks provide 10 times better mass sensitivity because of the increased surface-to-volume ratio. The use of more sensitive tuning fork sensors, together with a low noise detection circuit and adsorbent packed preconcentrator allow us to achieve ppb-level detection limit for BTEX components, which is more than three orders of magnitude improvement over our previous results [15]. Fig. 5(a) shows two repeated measurements of 250 ppb xylene, where xylene sample was injected for 1 minute, and then the sensor was purged with clean air for 2 minutes in each of the measurements. The detector shows fast response, rapid desorption, and excellent reproducibility (Fig. 5), which are critical for integrating its selective binding with the chromatographic separation principle. Note that the tuning fork sensors functionalized with the molecular imprinted polymers also eliminate effects due to common interferents, including acetone, ethanol, and ammonia. We have tested these interferents at high concentrations (~40 ppm) and observed no response from the tuning fork sensors [15], [17]. This level of interference immunity is necessary to minimize false positive for many real world applications.

Device Calibration—We have calibrated the hybrid device using BTEX samples of various concentrations. The BTEX samples are prepared and the concentration of each compound is tested with a Selected Ion Flow Tube – Mass Spectrometer (Instrument Science, UK). Fig. 6(a) shows a chromatogram for one of BTEX calibration sample. 19 m column is used in this calibration test. The BTEX components elute at the expected elution sequence: benzene, toluene, ethylbenzene, and xylenes, consecutively. Also note that the xylene isomers which are m-xylene, p-xylene, and o-xylene are also well separated. The peak height of each component is proportional to the analyte concentration (Fig. 6(b)). The sensitivities determined from the slopes of the linear plots are 0.57 mHz/ppb, 0.50 mHz/ppb, and 1.15 mHz/ppb for benzene, toluene, and xylene, respectively. The detection limits are a few ppbs for all of the above compounds. It is important to mention that a short preconcentration period of 45 seconds is used here; and the detection limits can be further lowered to about 1 ppb by increasing the preconcentration time to 5 minutes. The Occupational Safety and Health Administration's permissible exposure limit (OSHA PEL) for benzene, toluene and xylenes are 1 ppm, 200 ppm and 100 ppm, respectively [21], [22]. In an EPA study, the ambient benzene concentrations in the U.S. were found to be within ppb range [23]. The large linear calibration range and low detection limit levels of our device are useful for most outdoor and indoor environmental air monitoring scenarios as well as industrial applications. Once the calibration is established, unknown BTEX concentrations can be determined using the peak height of the chromatograms.

B. Field tests

In order to test the robustness of the hybrid device in the field, preliminary field tests under different scenarios are carried out and the findings are summarized below.

a. BTEX detection in gasoline vapors—Gasoline consists of a complex mixture of aliphatic and aromatic hydrocarbons, which presents one of the most complicated and challenging samples for analysis [24]. The ability to detect BTEX from gasoline is also essential for detecting traffic related environmental, safety and health applications. Vapors from a car gasoline tank are detected and analyzed with the hybrid device configured with a 19-m separation column (UAC-502). The sample shows over 20 different hydrocarbons (Fig. 7(a)), including the individual isomers of xylenes. The Fig. 7(b) shows the peak of benzene, where the side peaks could be due to other components in gasoline vapor, such as cyclopentane and 2-methyl-2-butene. The experiment shows clearly that the hybrid device is

highly selective for BTEX components, and allows for the detection of benzene at ppb level in the presence of complex interferents. The hybrid device demonstrates a clear advantage over current existing commercial and reference methods for use in the field and real-time assessment of health and environmental hazards in air, such as BTEX from gasoline (incomplete combustion or leaks from various sources).

b. Air pollution due to the Gulf oil spill—The Gulf oil spill in 2010 contaminated the ocean with over 160 billion gallons of crude oil, and also released unknown amount of VOCs into the air in the Gulf of Mexico, which potentially affects the residents living in the region and workers engaged in oil remediation and cleaning activities [25]. Conventional monitoring equipments showed limited sensitivity and slow response time for real time monitoring of VOCs evaporated into air [26]. Taking advantage of the portability of the hybrid device, our team rented a shrimp boat and tested air quality at multiple locations in an area located 69 miles from the spill site. For this field test, we used a single 2-m column (UAC-502) and with 5 minutes preconcentration time.

A typical data set is shown in Fig. 8 as a blue curve. For comparison, a 50-ppb BTEX calibration curve (black line) is also plotted in the figure. The results reveal two important findings. First, peaks associated with BTEX are absent, which indicate their levels are below 1 ppb. This finding is in agreement with the data reported by EPA's mobile units, which were deployed in the area at the time of the analysis (June 13th, 2010). The absence of BTEX in that area is presumably due to the high volatility of the BTEX compounds. Second, a large peak between where peaks of toluene and ethylbenzene are expected is found. This peak is due to an alkyl hydrocarbon, and its concentration is estimated to be ~50 ppb. We have confirmed the finding by analyzing water samples collected at the site with gas chromatography-mass spectrometry (GC-MS). The GC-MS data is shown as inset in Fig. 8, which reveals a peak between where toluene and ethylbenzene are expected. This peak is identified as a hexane derivative, which is in excellent agreement with the finding of our hybrid device.

IV. Conclusion

We have successfully developed a hybrid sensor device that combines selective preconcentration, separation, and tuning fork-based detection principles into a single integrated wireless device. The device is portable, battery operated, and wirelessly connected to a user-friendly cell phone application. It can detect and discriminate toxic VOCs in the presence of complex interferents with a detection limit of ~ 1 ppb.

We have demonstrated the device's applications in real world environments, such as outdoor air quality and gasoline vapor monitoring, and detected carcinogenic benzene in the presence of complicated interferents. We have also demonstrated the usability of the hybrid device in environmental accidents and disasters that demand rapid assessment of air quality in microenvironments, such as oil spill that took place in the Gulf of Mexico in 2010.

The portability, selectivity, and sensitivity of the hybrid device for detecting toxic hydrocarbons are unprecedented, and expected to lead to many applications, including environmental, occupational health and safety, and epidemiological studies.

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Fig. 1. Key components of wireless hybrid sensor. A preconcentration unit selectively collects and pre-concentrates analytes from air, a separation unit separates the analytes released from the pro-concentrator, and a detection unit comprises a microfabricated tuning fork array functionalized with molecular imprinted polymers detects the analytes.

Fig. 2.
Photos of a hybrid device together with a cell phone. Inset: Cell phone-based user interface.

Fig. 3. Chromatograms of BTEX sample with and without preconcentration. (a) BTEX sample direct injection without preconcentration, concentration: 10 ppm. (b) BTEX sample injection with 20 minutes preconcentration time, concentration: 20 ppb.

Fig. 4. Black curve: 5 ppm n-hexane only, preconcentration time: 1 minute. Red curve: Separation of 5 ppm n-hexane and 5 ppb benzene with the hydrid device. Preconcentration time: 1 minutes.

Fig. 5. Sensitivity and reproducibility: highly selective tuning-fork based detector of the hybrid device (a) Response of tuning fork sensor to 250 ppb xylenes. Detection limit: 4.4 ppb xylenes. (b) Reproducibility study: results of 12 measurements of the same xylenes sample using the tuning fork sensor.

Fig. 6.

(a) Separation chromatogram of BTEX mixtures. [B] is benzene, [T] is toluene, and [X] is Ethylbenzene + xylenes. Concentration: 40 ppm, preconcentration time: 20 seconds (b) Calibration curve for BTEX components. Peak height is taken as the response. Linear response is obtained even at high concentrations. Preconcentration time of each test: 45 seconds.

Fig. 7.

(a) Result from gasoline vapors taken from a car tank showing benzene, toluene and xylenes. Preconcentration time: 5 minutes. (b) Zoom in to benzene.

Fig. 8.

(a) The testing result of gulf coast (blue line), the typical response of a laboratory prepared BTEX mixture (black line) and GC-MS result (insert). (b) Map showing test location and oil spill area, date: 6-13-2010.