Novel gas chromatographic microsystem with very large sensor arrays for advanced odour discrimination

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***Abstract*— Here we report on a new technology for electronic noses (e-noses). This combines two large sensor arrays with a micro-package that behaves similarly to a gas chromatograph column, inspired by the nature of the human olfactory mucosa. Our system comprises two silicon based chemo resistive arrays of 300 sensors with a micro-fluidic package, formed by stereo-lithography, fitted between them. In this configuration, the first sensor array provides a "spatial" response to an odour, similar to a traditional e-nose. Then, as the odour pulse travels along the column, chemical components are selectively delayed, thus the second array gives temporal information. Experiments show a time delay of up to 38 s between ethanol and toluene pulses traveling down the column. We believe that this combination of an initial sensor array, producing spatial data with a second array creating spatio-temporal data significantly enhances the ability of an e-nose to discriminate between complex odours.**

I. INTRODUCTION

Although electronic noses (e-nose) have been with us for over 10 years, their ability to detect and identify complex odours still lags behind that of the human olfactory system. E-noses are compared to their biological counterpart, but they neither mimic truly its structure nor its sensor diversity (e.g. *ca*. 350 different olfactory binding proteins). Research into an artificial bio-mimetic mammalian olfactory system has been an active research area since the idea of detecting odours artificially was first proposed in 1920 by Zeaardemarker and Hodewind [1]. The earliest experimental instrument considered was developed by Wilkens and Hartman in 1964 [2]. Almost 20 years later, Persaud and Dodd proposed the possibility of an E-nose with broadly tuned receptors in 1982 [3]. Since then, as developments in electronics, sensors and computing methods have advanced, a number of different types of E-noses mimicking the human sense of smell have been developed.

In the human nose, odours are delivered through the upper nasal passage (superior turbinate) to the olfactory epithelium. The inhaled odorant molecules then pass through a thin (20 μm) mucous layer to reach about 50 million receptor cells that occupy a small part of the nasal epithelium. Previous work has shown that this mucous layer has chromatographic like properties, thus receptor cells in the olfactory epithelium are exposed to time delayed odorant signals, where this latency is dependent on the interaction between different chemicals components within the odour and the mucous layer. Hence, identification can be performed from both spatial and temporal a sensor information [4]. Here we report on the design of a new e-nose system that combines large sensor arrays with a chromatograph column to closely mimic the "nasal chromatograph" effect [5]. Our goal is to create a new generation of low-cost e-nose microsystems for portable applications with enhanced ability to discriminate complex odours by producing these spatio-temporal data.

II. DESIGN AND FABRICATION

Figure 1 shows the concept of our system combining a dual sensor array with a GC like column. The first sensor array (left-side) samples the odour to provide spatial data similar to a traditional e-nose. The odour then passes through a GC like column that delays chemical components within odour dependent on their interaction with a stationary phase coating on the inside of the column. This temporarily delayed odour is the analysed by a second sensor array (right-side), which provides spatio-temporal data.

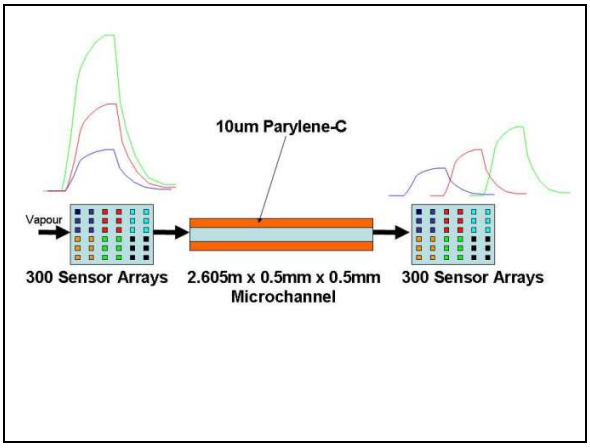

Figure 1. Dual Sensor Array with GC channel Concept

This system has been formed by combining two 300 chemo resistive sensor arrays, employing composite carbon/polymer materials as the sensing films (carbon black, Cabot corp. USA, mixed with 10 different sensing polymers), between which a GC like column has been placed. Figure 2 shows one of the two 300 sensor arrays used in this study. This demonstrator chip has been coated with only 4 different types of polymer-carbon black composite material. Replicate sensors (12 sensors) for the same odorant will improve the redundancy of the system thus giving a more accurate result. The large sensor arrays have been designed and developed specifically to cope with the large number of sensors, with multi-metal structure for individual sensing unit. In this array, like a matrix, every sensing element is assigned an address, comprising a row index and a column index. This array could be considered as a coded address array configuration. An electronic circuit supplies constant current to the system while selecting the exact row and column to be measured. The electronic system can measure up to 25 rows at once. In order to obtain all 300 sensor readings, the circuit which is controlled using Labview software (version 6.1) has to make 12 consecutive readings by selecting one column after another. In order to read 600 sensors, analogue switches are placed between the two sets of sensors. A temperature sensor is also incorporated within the sensor array in order to measure the local environment.

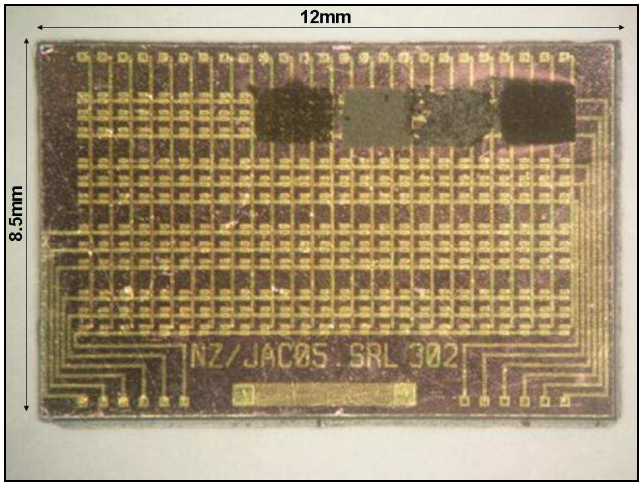

Figure 2. Partly Coated 300 Sensor Array Chip

The GC column component is shown in Figure 3. Here the column has been combined with the sensor chamber to reduce dead volume within the system thus making the system more efficient. The length of the column is 2.605 m with a channel cross-section of 0.5 mm × 0.5 mm. The column was fabricated using an Envisiontec Perfactory Mini micro-stereolithography machine. The 3D design for a standalone chamber, as well as combination of micro-channel chamber, was designed in Solidworks and then exported to the Envisiontec Perfactory Machine where the microchannel was formed.

The micro channel was built unsealed to aid cleaning and then coated with a 10 µm thick Parylene C as the non polar stationary phase material (SCS/PDS 2010 Specialty Coating Systems). The Parylene depositor is based upon physical condensation under vacuum at room temperature. The advantage of using this machine is it can provide a uniform layer of coating on the component. After coating, the column is then sealed using glass slides coated by a thin layer of adhesive. In order to verify a tight seal, the column was immersed in water while air was pumped through it.

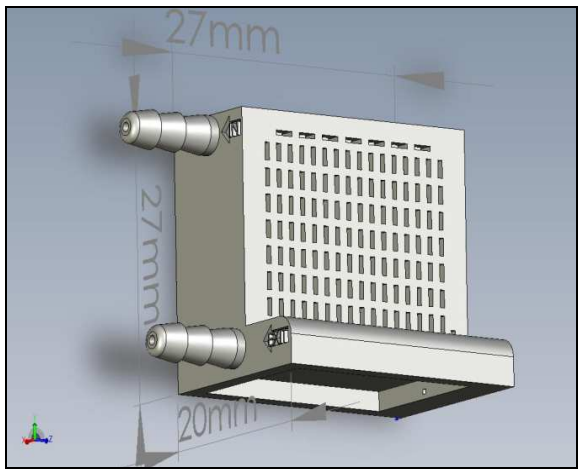

Figure 3. 3-D CAD diagram of GC Column and Chamber

III. SYSTEM INTEGRATION

Figure 4 shows the arrangement of both the micro-channel with the integrated chamber and an additional standalone chamber. Adhesive was used to seal both chambers onto the sensor package making it hermetically sealed.

The tested vapour (in air) is pulled through a flow sensor then through the first chamber (standalone chamber). Responses of the 600 sensors are measured continuously during the test. From the first chamber the vapour will flow through the column, then into the second chamber (column-chamber combination).

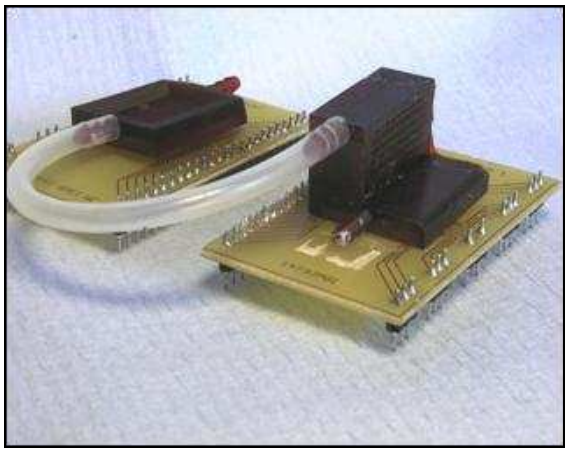

Figure 4. Sensors Array with unsealed Micro-GC Column and Chamber

During this test cycle, the data acquisition system measures the resistance of all 600 sensors continuously. A 300 seconds flush of the system is performed before testing another vapour to enable the sensor signals to stabilize.

Ethanol and toluene vapour in laboratory air were tested at flow rate 20 sccm and temperature 20 ±1 ˚C. A 10 s pre-test flush was performed before passing 60 s of ethanol or toluene vapour. Then, a 100 s flush with laboratory air was carried out to purge the system.

IV. Results and Discussion

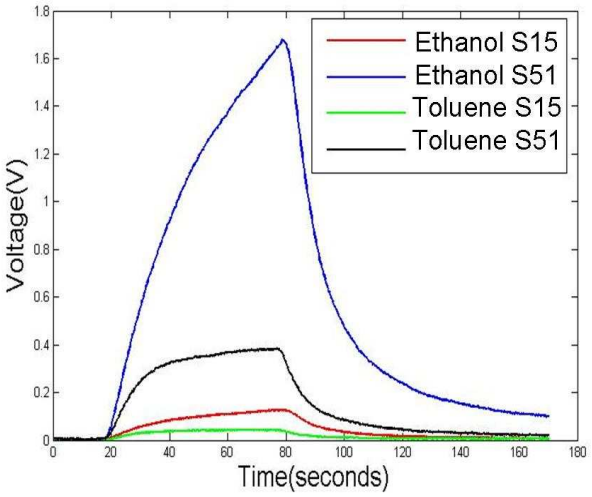

Figure 5. Spatial Information for Polystylene-co-butadiene (bottom two curves) and Polyethylene-co-vinyl acetate (top two curves)

Figure 5 shows the spatial information collected by sensor S15 and S51 in the first sensor array to toluene and ethanol vapour. As shown in the figure, the first sensor array provides spatial information just like a conventional electronic nose in which carbon black-polymer coatings react differently to each odour.

The temporal information caused by the micro channel is shown in Figure 6. The toluene pulse was delayed by 38 s compared to 20 s for ethanol (taken from time to reach 50% of its final value). These shows significant temporal difference that would increase when a longer micro-channel is used.

These two responses show the ability of our system to create spatio-temporal data that we believe could be used to enhance odour discrimination.

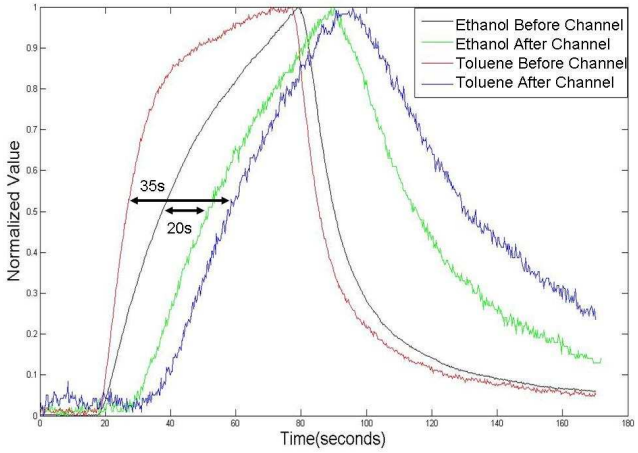

Figure 6. Difference between Temporal Information for Sensor S51- Poly (ethylene-co-vinyl acetate) towards ethanol and toluene pulses

V. Conclusion

We have demonstrated the successful integration of large chemo sensor arrays consisting of 600 sensors and a micro gas chromatograph channel to imitate the behaviour of the olfactory mucosa. In addition to the diversity of the 300 sensors coated with 10 different polymer composite films, this system provides temporal information that can be used to enhance odour discrimination. We are currently trying a variety of different stationary phase coatings as well as longer micro channel to improve the temporal information of the system. We are also experimenting with two gas chromatographic columns to increase the discriminating power of the system.

VI. References

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