

ENHANCED DISCRIMINATION OF COMPLEX ODOURS BASED UPON SPATIO-TEMPORAL SIGNALS FROM A MICRO-MUCOSA

J. W. Gardner¹, S. L. Tan², J. A. Covington¹ and T. C. Pearce³

¹Sensors Research Laboratory, School of Engineering, University of Warwick,
Coventry CV4 7AL, UNITED KINGDOM

(Tel: +44 (0)2476 523695; Email: J.W.Gardner@warwick.ac.uk)

²School of Computer Engineering, Nanyang Technological University SINGAPORE

(Tel: ++65 6790 6706 Email: ASSLTan@ntu.edu.sg)

³Department of Engineering, Leicester University, Leicester LE1 7RH, UNITED KINGDOM

(Tel: +44 (0)116 2231307; Email: t.c.pearce@leicester.ac.uk)

Abstract: We recently reported the novel concept of an artificial olfactory mucosa based upon a set of sensor clusters distributed along a channel coated with a retentive layer. Such a system generates complex signals containing both spatial information (i.e. response magnitude) based upon different types of sensors and temporal information (i.e. delay time like in a GC) based upon retention time differences between identical sensors. Here we report on the development of a micro artificial mucosa or micro e-mucosa. The microsystem comprises of a silicon microsensor array coupled to a true 3D micro-fluidic package fabricated by micro-stereolithography. Results show a differential temporal delay of 96 seconds between simple odours (pulses of toluene and ethanol vapour in air) and improved discrimination of complex odours by combining temporal with spatial data. We believe that this new micro e-mucosa offers a significant advance in the field of machine olfaction.

Keywords: Artificial mucosa, chemical microsystem, odour sensing, biomimetic sensors.

1. INTRODUCTION

In the past decade significant advances have been made in our understanding of the chemosensory mechanisms that govern the response of the human olfactory system. In terms of the development of its artificial electronic counterpart (commonly known as the electronic nose) that crudely mimics this biological system, attention has been mainly focused upon two aspects: namely the choice of sensing materials and data processing techniques [1]. However, little attention has been devoted to mimicking the “front-end” of the biological olfactory system, which may provide added information for the accurate discrimination of complex odours.

The overall aim of the research presented here is to exploit the phenomena commonly referred to as “nasal chromatography” in a new type of chemosensor system. We believe that the resultant spatio-temporal signals have richer information content and so will enable a higher level of discrimination than existing e-nose

systems. Thus we propose a new type of electronic nose that mimics this “nasal chromatograph” effect and we refer to as an artificial olfactory mucosa or e-mucosa for short.

Preliminary work was reported earlier this year and involved the development of a large PCB-based prototypical system [2]. Here we report on the fabrication of a novel micro artificial olfactory mucosa: combining a silicon sensor array with a fluidic micro-package formed by the technique of micro-stereolithography.

2. ARTIFICIAL MUCOSA

Figure 1 shows the basic concept of our artificial mucosa. In our system, when an odour pulse travels down a micro-channel (within the 3D micropackage) it is delayed by an absorbent coating that acts like the mucous layer in the nasal cavity. This absorbent material selectively delays the odour pulse thus creating an odour/coating specific time delay. The partitioning effect is somewhat similar to a traditional gas

chromatograph column. Groupings of different chemical sensors are placed at different distances along the length of the micro-channel, which correspond to the olfactory epithelium in the biological system. These sensors detect the odour pulse as it travels along the channel, thus providing both spatial (within group) and temporal (between group) information. Hence, we incorporate the key aspects of the biological system, namely, odour partitioning along a column and sensor segregation, combined to form an integrated solution.

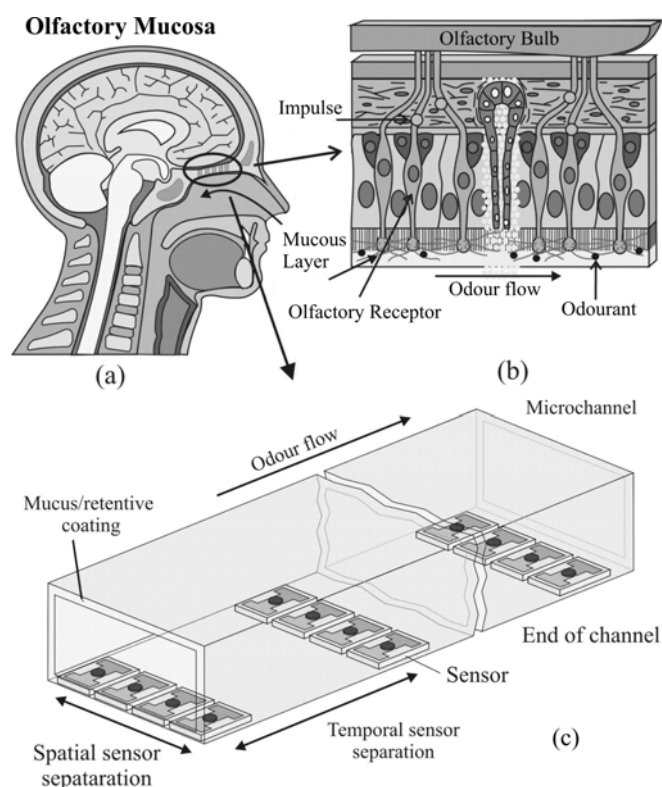


Figure 1 (a) Human head, (b) biological olfactory mucosa and (c) the cartoon of an artificial olfactory mucosa with groupings of four different sensors replicated along the length of the micro-channel.

The sensor array contains a total number of 80 resistive sensing elements, formed through a coplanar pair of gold electrodes ($20\ \mu\text{m}$ apart, aspect ratio of 10, on a Si wafer), and with the opening defined by an SU-8 layer. Each electrode pair is covered by a thin sensing layer made from a composite of polymer and carbon black (carbon black, Cabot corp., USA). Five different non-conducting stationary phase polymer materials

(80:20 ratio mix by weight, polymer to carbon black) were used. These were: polystyrene-co-butadiene (PSB); polyethylene-co-vinyl acetate (PEVA); polyethylene glycol (PEG); polycaprolactone (PCL); and poly4-vinyl phenol (PVPH). More details on the deposition process can be found at [3]. The sensors possess resistances in the range of 2-8 k Ω . Figure 2 shows a photograph of the silicon chip comprising of the 80 sensing elements and coated with polymer sensing layers. This figure also shows the sensor numbering used.

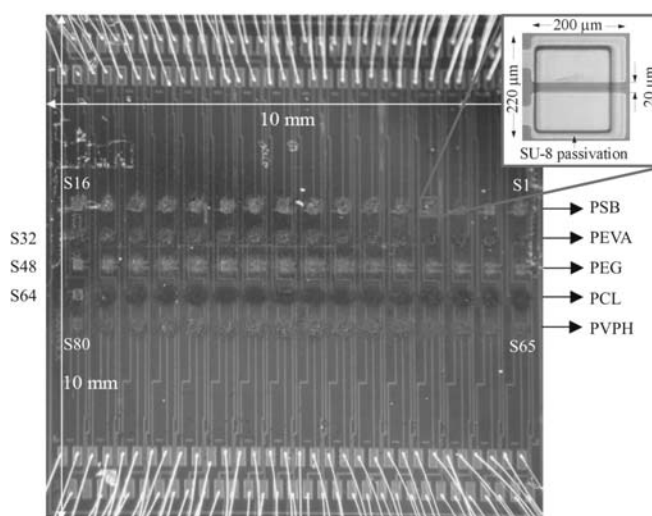


Figure 2. Photograph of silicon micro-sensor array part of e-mucosa.

Figure 3 shows the micro-package design. The package has external dimensions of $7 \times 36 \times 27\ \text{mm}^3$ and contains a channel with dimensions of $0.5\ \text{mm} \times 0.5\ \text{mm} \times 2.4\ \text{m}$. The package is designed to contain multiple channels, stacked in the Y direction, with openings and the top and bottom of each channel. This package is coated with an approximately $10\ \mu\text{m}$ thick layer of Parylene C as the retentive layer (PDS 2010 LabcoaterTM 2 (Specialty Coating Systems, Indianapolis, USA)). The micro-package was fabricated using a Perfactory Mini (Envisiontec, Germany) rapid prototyping machine and the package was made from a photo-sensitive acrylate. The package was designed so that it would encompass blocks of 5 chemical sensors, with each block containing one sensor of each polymer (e.g. sensors S1, S17, S33, S49 and S65

from, figure 2, make up the first block). The remaining openings are there to aid cleaning of any residual resin. After assembly these are sealed to complete the emucosa.

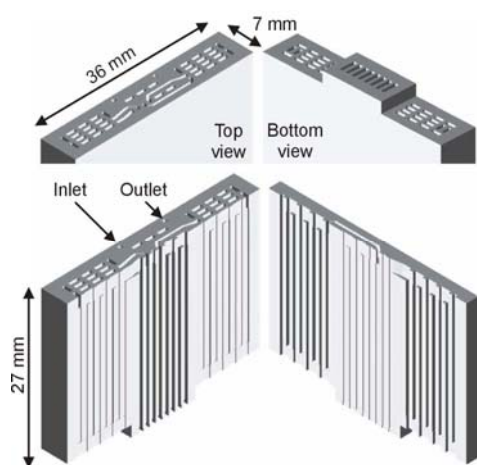


Figure 3. Design of the micro-fluidic package

The fully assembled chemical microsystem or e-mucosa is shown in Figure 4. Here we see the sensor chip bonded onto a PGA socket (Spectrum Semiconductor, USA) and on a custom made printed circuit-board. The micro-package is positioned above the silicon array with micro-channels aligned to the sensing elements; finally it is connected via PTFE tubes to an automated odour delivery system. For testing, the sensor chip was connected to a custom electronic interface where each chemical sensor was used as the feedback resistor of an op-amp (OP277, Analog Devices) in an inverting configuration. The output voltage was then sampled by on-board 16-bit ADCs.

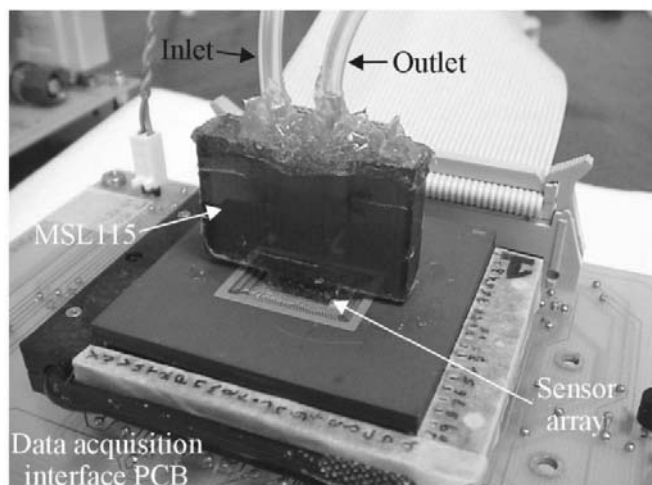


Figure 4. Proposed micro artificial mucosa: an integrated micropackage and silicon sensor array.

3. RESULTS

The artificial mucosa was exposed to a series of pulses of odours and mixtures of odours. The experimental tests were performed at a temperature 30 ± 2 °C, relative humidity of $40 \pm 5\%$ r.h, flow rate 25 ml/min, pulse width 25 s with laboratory air as the carrier gas.

Figure 5 shows the temporal response to ethanol and toluene vapour in air at the beginning and end of the micro-channel (ethanol and toluene vapour concentrations were set to 22 PPT and 12 PPT (Parts Per Thousand) respectively, with PCL coated sensors). The data have been normalised, thus the spatial information has been removed. This shows that toluene vapour has a much longer retention time (~ 168 s) compared to ethanol vapour (~ 72 s). Thus shows the ability of our system to delay selectively different types of vapour.

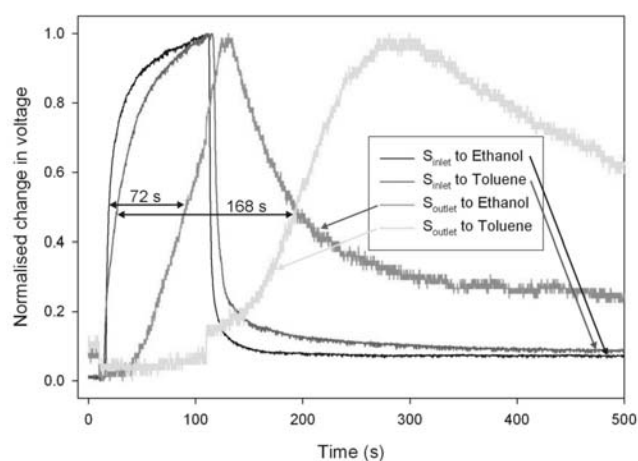


Figure 5. Different responses of sensors to odour pulses within the artificial mucosa.

These experiments were repeated for different complex odours and binary mixtures thereof. Table 1 summarises the experimental results from our set of measurements on milk, cream, peppermint and vanilla samples. For this analysis 5 different sensors were used placed along the channel S34 (PEG sensor 30 mm from inlet), specifically S38 (PEG sensor 1060 mm from inlet), S57 (PCL sensor 2100 mm from inlet), S27 (PEVA sensor, 2160 mm from inlet), and S30 (PEVA sensor, 2200 mm from inlet).

Table 1. Spatial and temporal responses of five different sensors to six different stimuli.

Spatial data (ΔV)	Milk	Cream	Milk + Cream	Pep	Van	Van + Pep
S34 Av.	-0.385	-0.423	-0.338	-0.513	-0.474	-0.411
s.d.	0.014	0.016	0.006	0.061	0.026	0.008
S28 Av.	-0.534	-0.584	-0.462	-0.992	-0.743	-0.705
s.d.	0.037	0.031	0.007	0.133	0.067	0.025
S57 Av.	0.070	0.079	0.056	0.186	0.098	0.104
s.d.	0.005	0.006	0.002	0.010	0.001	0.002
S27 Av.	0.117	0.112	0.109	1.619	0.280	0.586
s.d.	0.010	0.007	0.019	0.079	0.004	0.013
S30 Av.	0.077	0.082	0.069	0.892	0.157	0.324
s.d.	0.008	0.009	0.005	0.036	0.005	0.007
Temp. data (sec)						
S34 Av.	110.13	87.04	113.68	80.28	94.18	98.71
s.d.	23.67	7.77	5.10	26.24	4.71	5.29
S28 Av.	103.99	83.80	106.50	29.25	68.14	78.53
s.d.	16.06	7.41	14.29	3.92	17.94	10.78
S57 Av.	66.22	47.93	78.48	31.54	42.63	51.14
s.d.	2.71	2.99	1.23	3.23	1.92	1.22
S27 Av.	32.08	18.59	38.86	57.76	51.39	67.51
s.d.	4.86	1.17	10.20	2.31	25.13	2.38
S30 Av.	74.68	65.00	93.46	112.49	95.70	113.51
s.d.	17.91	9.30	9.61	2.74	1.52	1.80

Principal component analysis (PCA) was performed on the spatial data (i.e. response magnitude), temporal data (delay time, time to reach 50% of its maximum value) and the combined spatial and temporal data for a set of four different odours and two mixtures. Figure 6 shows the PCA plot for the combined data-sets.

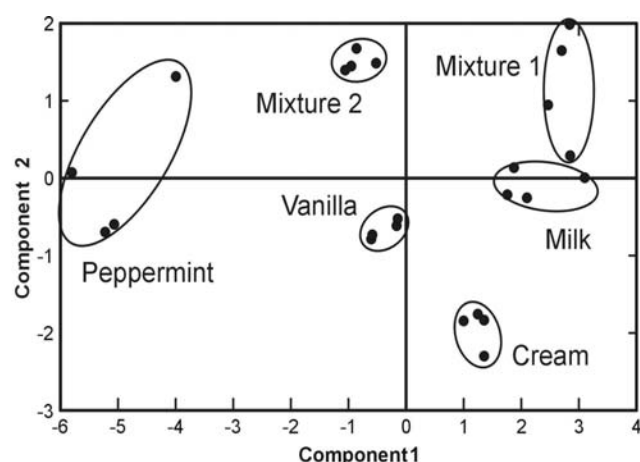


Figure 6. PCA of single and complex odours measured from the e-mucosa. Mixture 1=50:50 mix of Milk and Cream, Mixture 2=50:50 mix of Vanilla and Peppermint.

From the PCA it was found that the distance between the complex odours, as represented in multivariate vector space, was greater for the spatial-temporal data than that for the spatial data or temporal data alone; only the combined data set giving complete separation. A more detailed parametric study of the responses is being carried out using both linear and non-linear classification methods, such as discriminant and radial basis function analyses. The results of this study will be published elsewhere.

4. CONCLUSIONS

In conclusion, the combined spatio-temporal data produced by our simple artificial mucosa gave superior performance by providing complete linear separation in multivariate space of the different odours tested here. More precisely, the artificial mucosa offers better separation than that observed from using either the spatial data (i.e. conventional e-nose) or the temporal data (i.e. type of basic GC column). We believe that our concept of an artificial olfactory mucosa is not only novel but may also lead to the development of a new and better generation of odour recognition systems based upon biomimetic principles. Such systems could employ much more advanced time-dependent signal processing algorithms and address more challenging odour discrimination problems like segmentation.

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