Combined electronic nose and tongue for a flavour sensing system

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SUMMARY

We present a novel, smart sensing system developed for the flavour analysis of liquids. The system comprises both a so-called "electronic tongue" based on shear horizontal surface acoustic wave (SH-SAW) sensors analysing the liquid phase and a so-called "electronic nose" based on chemFET sensors analysing the gaseous phase. Flavour is generally understood to be the overall experience from the combination of oral and nasal stimulation and is principally derived from a combination of the human senses of taste (gustation) and smell (olfaction). Thus, by combining two types of microsensors, an artificial flavour sensing system has been developed. Initial tests conducted with different liquid samples, i.e. water, orange juice and milk (of different fat content), resulted in 100% discrimination using principal components analysis; although it was found that there was little contribution from the electronic nose. Therefore further flavour experiments were designed to demonstrate the potential of the combined electronic nose/tongue flavour system. Consequently, experiments were conducted on low vapour pressure taste-biased solutions and high vapour pressure, smell-biased solutions. Only the combined flavour analysis system could achieve 100% discrimination between all the different liquids. We believe that this is the first report of a SAW-based analysis system that determines flavour through the combination of both liquid and headspace analysis.

Key words: Electronic nose, electronic tongue, Flavour system, chemFET, SH-SAW

1. INTRODUCTION

A challenging problem in the food and beverage processing industries is how to ensure the quality of products. This is achieved by spending significant time and effort on assessing the flavour of samples. Traditionally, panels of trained experts evaluate quality parameters; however this suffers from a number of drawbacks. For example, sensory panels are time consuming, expensive, discrepancies can occur due to human fatigue or stress and clearly cannot be used for online measurements. Thus the development of alternative methods to organoleptic panels for the objective assessment of food products, in a reliable and cost-effective manner, is highly desirable. Consequently, the combination of artificial sensors (for taste and smell) has the potential to reduce the need for flavour panels, since their outcome can be correlated to a human-based sensory experience. Our main aim is to demonstrate the ability of the combined SAW-based system to discriminate between different liquid samples that cannot easily be identified by an electronic nose or tongue individually. Although we refer to this instrumentation as a flavour sensing system, we do acknowledge that the sensors measurements are, in fact, indirect and thus correlated to perceived flavour. However we can think of no better term than flavour sensor even when we are measuring physical properties rather than biological molecules.

2. OPERATING PRINCIPLES AND DESIGN

The electronic tongue employed here is based on surface acoustic wave (SAW) technology. In general, acoustic wave microsensors detect different physical properties, such as mass, density, conductivity, and viscosity of liquids and gases, and offer the benefits of real-time electronic read-out, small size, robustness and low unit

cost. In particular, SAW devices are of considerable interest for sensing applications in gaseous and/or liquid environments.

When a SAW propagates along a piezoelectric crystal surface, its interaction with an adjacent gas or liquid results in a change of its propagation characteristics. Thus, the determination of liquid or gaseous properties can be achieved by measuring simple changes in the velocity, frequency, amplitude or phase of the acoustic wave. Here shear horizontal surface acoustic wave (SH-SAW) devices are employed for the electronic tongue component of the system for analyzing the liquid samples. The devices comprise a dual 2-port delay-line interdigital transducer (IDT) configuration. The configuration utilised allows measurement of both mechanical and electrical characteristics of the liquid under test to discriminate between different samples and also to determine specific properties of the liquids. Unlike electronic tongue devices reported to date [1-5], the devices we have used are based on measuring key physical parameters rather than chemical principles. The main advantage of our SH-SAW devices is that it can measure different properties of a liquid without the need for a taste-sensitive selective membrane. This in turn increases the lifetime and durability of the resultant devices, albeit with a loss of specificity and, in some applications, sensitivity.

Electronic noses, in general, are based on an array of non-selective sensors with overlapping sensitivities. They are not designed to identify specific chemical components within an odour/sample, but the aroma. A single sensor within an array responds to the combined volumes of a number of chemicals, and so a small number of chemical sensors, in an array, can create a chemical fingerprint of an odour. In

electronic nose instruments there are several different sensing technologies, based upon a number of different operating principles, for example metal oxide, conducting polymer, acoustic wave, field-effect, and electrochemical gas sensors. Here, we have employed chemFET sensors with a conducting polymer sensing layer as the gate material. The basic configuration involves the metal (or polysilicon) gate of the FET being replaced with a conducting polymer gas-sensitive layer. These conducting polymers are a combination of an insulating polymer and carbon black particles with diameters of some tens of nanometres. The carbon endows electrical conduction to the resultant mix. This layer is deposited directly on top of the gate oxide and the sensor response is related predominantly to a change in the work function between the polymer and the semiconductor. This in turn modulates the threshold voltage of the MOSFET. It is believed that the change of work function within the polymer is due to either a swelling effect, where the polymer expands or contracts thus altering the average work function, or to a change in the band structure of the polymer from the interaction between the polymer and the test vapour.

2.1 ELECTRONIC NOSE CHEMICAL SENSORS

The chemFET sensors were designed and fabricated in collaboration between the Institute of Microtechnology, University of Neuchatel (Switzerland) and the University of Warwick (UK). The overall devices were designed as arrays of four enhanced *n*-type MOSFET sensors. The individual devices were constructed within *p*-wells on an *n*-type lightly doped silicon substrate to minimise interference between adjacent sensors. The devices have a common gate / drain configuration and thus are operated in the saturated region. The devices were designed with channel dimensions 385 μm × 10 μm. Aluminium tracks were used to contact the drain, source and gate,

though Au was used as the sensing electrode (i.e. the gate connection of the chemFET sensor) for improved ohmic connection to the sensing layer and better chemical inertness. Accessibility to the Au gate was achieved by etching through the silicon nitride passivation layer and subsequent oxide layers. In the sensing chemFETs, the gold was also etched exposing the gate oxide below. Figure 1 shows a cross-section and top view of an individual sensor, showing particularly the common gate and drain and the active polymer material in contact with the gate oxide. The final die size of the device was 4 mm^2 with pads of 300 μm^2 for electrical connection to the sensors. The devices were fabricated with three open gates for sensing and one closed (covered) for reference. The polymers were supplied by Sigma Aldrich (UK) and the carbon black (Black Pearls 2000) was supplied by Cabot Corporation (USA). The carbon nanospheres were supplied as a powder with particle sizes between 50-80 nm, the polymers were either in powder form or small crystals. Three different polymers were used in this study poly(ethylene-co-vinyl acetate), poly(styrene-co-butadiene) and poly(9-vinlycarbazole), mixed at a 80:20 ratio (by weight) with carbon black. The polymers were first dissolved in their respective solvent overnight, with the aid of a magnetic stirrer and at an elevated temperature (50 ^oC). Next, carbon black was added and the mixture sonicated for 10 minutes using a flask shaker (Griffin and George, UK). The mixture was then deposited across the sensor electrodes using an airbrush (HP-BC Iwata, Japan) controlled by a micro-spraying system (RS precision liquid dispenser, UK), through a metal mask with a 1 mm hole. The gates were aligned to the mask using an X-Y stage before deposition occurred. The airbrush was held 10 to 15 cm away from the mask and several passes were sprayed depending on the desired thickness (or resistance). This gave a circular coating of typically 1 mm ±0.1 μm in diameter and 5 ± 1 μm thick that covered the central sensing section (i.e. one film

covered all the chemFETs). The device was mounted in a standard 14-pin DIL package (Spectrum Semiconductors, USA). Figure 2 shows a photograph of a fabricated microsensor array which has been coated with sensing materials.

The chemFETs were operated in a basic configuration with the gate and drain common. This means that the relationship between the drain current I_{DS} and the gate-source voltage V_{GS} is that for the saturated regime, namely

$$I_{DS} = \frac{W\mu_n C_O}{2L}(V_{GS} - V_T)^2 \quad (1)$$

Where W is the channel width, L is the channel length, μ_n is the electron mobility, C_o is the capacitance, V_T is the threshold voltage, and $V_{GS}=V_{DS}=V_{GDS}$.

The response of the chemFET is based upon by a gas-dependent shift in the work function of the polymer-metal gate, which in turn changes the threshold voltage of the transistor. The chemFET can be operated in either a constant current or a constant voltage mode. In the case of driving the chemFET with constant current, the change in gate-source voltage must be exactly equal (from equation (1)) the change in threshold voltage:

$$\Delta V_{GDS} = \Delta V_T \quad (2)$$

So any chemical interactions, which affect the threshold voltage, can be monitored in a simple and direct manner.

There are a number of advantages of operating the chemFET in the constant current rather than constant voltage mode that outweigh the smaller signal:

(1) Unlike ΔI_{DS}, ΔV_{GS} is independent of both channel width and length and so no longer sensitive to geometrical errors in the CMOS process.

(2) Unlike ΔI_{DS}, ΔV_{GS} is independent of device capacitance and so both the dielectric permittivity of the gate oxide and its thickness that can vary with CMOS process.

(3) Unlike ΔI_{DS}, ΔV_{GS} is not dependent upon the electron mobility which is itself strongly dependent upon operating temperature.

It should be noted that V_T still has a strong sensitivity to temperature because the partitioning coefficient of the analyte (i.e. solute) for the polymer (i.e. solvent) coating depends strongly upon the boiling point of the analyte. Further details of the properties of polymer gated FET sensors are provided in references [6 and 17], such as their temperature sensitivity, humidity sensitivity and response to gas mixtures.

2.2 ELECTRONIC TONGUE DEVICE

The electronic tongue devices were developed utilizing shear-horizontal SAW sensors. Here, the surface acoustic waves propagate along the surface of the substrate without coupling too strongly into the liquid, but still being perturbed by the liquid properties [7]. The devices were fabricated on a 36° rotated Y-cut X-propagating $LiTaO_3$ (36YX.LT) 3" wafer substrate. The configuration used is one which allows for simultaneous measurements of both mechanical (elasto-acoustic) properties, and electrical (electro-acoustic) parameters of the liquid under test. This is achieved through a dual delay line configuration, one shorted (metalized and electrically shielded) and the other left free (electrically active). This way, the shorted delay line measures mechanical parameters, predominantly mass loading and viscosity, whilst the free delay line measures in addition the permittivity and conductivity of the liquid under test. The electrical parameters can be indirectly related to certain taste properties, such as saltiness (sodium) and sourness (acetic acid) [8]. Figure 3 shows a

schematic of the basic arrangement of the dual delay line sensing system. The SH-SAW propagating on the surface of 36YX.LT substrate is sustained by both atomic displacements and electrical potentials due to the piezoelectric effect. When the surface of the substrate is metalized and electrically shorted the piezoelectric potential becomes zero at the surface and only the atomic/particle displacements interact with the adjacent liquid. This phenomenon is known as mechanical perturbation (or mechanical interaction) and can be used to detect the mechanical properties of a liquid, e.g. its viscosity and density. The theory related to mechanical perturbation of SH-SAW on the metalized surface was developed by Kondoh *et al*. [9]. In this, the changes in the SH-SAW were derived from Auld's perturbation theory [10] for gases and extended to the liquid phase where the changes of the velocity and the attenuation can be expressed, and therefore measured, as

$$\frac{\Delta v}{v} = -\frac{v}{4\omega P}(v_p{}^{*} \cdot Z_i{}' \cdot v_p - v_p \cdot Z_i \cdot v_p{}^{*}), \tag{3}$$

$$\frac{\Delta \alpha}{k} = -\frac{v}{4\omega P}(v_p{}^{*} \cdot Z_r{}' \cdot v_p + v_p \cdot Z_r \cdot v_p{}^{*}). \tag{4}$$

where, k is the wave number, v the phase velocity, ω the angular frequency, P the flow per unit width, v_p the particle velocity vector, and Z the acoustic metal surface impedance. Also, ´ indicates a perturbed quantity and * indicates a complex conjugate and the subscripts r and i represent the real and imaginary parts of Z.

From the above and assuming that a Newtonian fluid with a viscosity of η and density ρ_l is loaded on the metalized surface, by substituting the surface acoustic impedance Z into equations (3) and (4) we can obtain equations for viscous coupling and mass loading as: ,

$$\frac{\Delta v}{v} = -\frac{v{v_{pc}}^2}{4\omega P}\left(\sqrt{\frac{\omega\eta'\rho_{l'}}{2}} - \sqrt{\frac{\omega\eta\rho_l}{2}}\right) \tag{5}$$

$$\frac{\Delta \alpha}{k} = \frac{v{v_{pc}}^2}{4\omega P}\left(\sqrt{\frac{\omega\eta'\rho_{l'}}{2}} + \sqrt{\frac{\omega\eta\rho_l}{2}}\right) \tag{6}$$

for viscous coupling, where, v_{pc} is the particle velocity component of the shear horizontal mode.

and

$$\frac{\Delta v}{v} = -\frac{uh{v_{pc}}^2}{4P}\left(\rho' - \frac{\mu'}{v^2}\right), \tag{7}$$

$$\frac{\Delta \alpha}{k} = 0\,. \tag{8}$$

for mass loading, where, μ' is the Lamè constant of the film. The above assumes that an isotropic thin liquid film of thickness h and density ρ is uniformly loading the metalized surface and that the liquid properties do not change before and after perturbation.

On the other hand, when the surface is free and electrically active both the particle displacements and electrical potentials interact with the liquid. This is an electrical interaction (also known as the acousto-electric interaction/perturbation) with the liquid that affects the velocity and/or attenuation of SH-SAW propagation and it is utilised in sensing the electrical properties of the liquids, e.g. the relative permittivity and conductivity. By employing the perturbation theory proposed by Auld [10], the following acousto-electric interaction (electrical) relationships for changes in velocity and attenuation of the SH-wave in the presence of a liquid are achieved:

$$\frac{\Delta v}{v} = -\frac{K_s^2}{2}\frac{(\sigma'/\omega)^2 + \varepsilon_0(\varepsilon_r' - \varepsilon_r)(\varepsilon_r'\varepsilon_0 + \varepsilon_P^T)}{(\sigma'/\omega)^2 + (\varepsilon_r'\varepsilon_0 + \varepsilon_P^T)^2} \tag{9}$$

$$\frac{\Delta \alpha}{k} = \frac{K_s^2}{2}\frac{(\sigma'/\omega)(\varepsilon_r\varepsilon_0 + \varepsilon_P^T)}{(\sigma'/\omega)^2 + (\varepsilon_r'\varepsilon_0 + \varepsilon_P^T)^2} \tag{10}$$

Here, K_s^2 is the electromechanical coupling coefficient when the reference liquid is loaded on the free surface, *k* is the wave number, ε_P^T is the effective permittivity of the SAW crystal, ε_r is the permittivity of the reference liquid (distilled water), ε_r' and σ' are the permittivity and conductivity (related to loss) of the liquid.

Thus we can conclude that the changes in the velocity of SH-SAW on the two delays lines are functions of several parameters, some of them being common for both lines:

$$\left(\frac{\Delta v}{v}\right)_{free} = f(\varepsilon, \sigma, \eta, \rho, T) \tag{11}$$

$$\left(\frac{\Delta v}{v}\right)_{shorted} = f(\eta, \rho, T) \tag{12}$$

where, ε, σ, η, ρ are the permittivity, conductivity, viscosity and density of the liquid under test and *T* the temperature common to both delay lines. It is these physical parameters that are being used to measure indirectly "taste" in our so-called electronic tongue.

The SH-SAW devices' transmit and receive transducers (i.e. a 2-port device) were optimally designed to have 28 finger pairs with 17 μm width of electrode and 17 μm separation between each electrode, providing. a periodicity of 68 μm. The IDT aperture is 2.0 mm with the IDT centre-to-centre separation of 7.5 mm and a free area of 2.0 mm × 1.5 mm, with overall device dimensions of 8.0 mm by 10.5 mm. The

feature dimentions were determined taking into consideration various constraints in terms of the aperture and the number of finger pairs in the IDTs, in order to achieve trade-offs between the low insertion loss, bandwidth and diffraction losses [11]. The devices were fabricated on a 3 inch $LiTaO_3$ wafer with operating frequency of approximately 60 MHz.

In order to test the sample liquids, the devices were mounted on a custom designed printed circuit board (PCB) below a PTFE cell that contained the liquid under test. The cell is 32 mm in length by 20 mm wide with a central reservoir of 6.8 mm × 2.5 mm × 8.0 mm and a volume of approximately 136 μl. The liquid cell is positioned accurately over the sensing area between the IDTs with the aid of guiding pins that fit into holes in the PCB. The cell rests on the device without any sealant. This enables easy removal of the cell to clean the device and yet holds the liquid without leakage. The top of the liquid cell was machined with a depression to allow the 14 pin DIL package to fit in. The package is mounted face down to detect the sample headspace (pins point up to allow socket connection to control electronics interface). The device was clamped down using a brass fitting across the back of the 14 pin DIL package with bolts on either end. A photograph of the system is shown in Figure 4(a) [12] and a schematic cross-section through its middle provided in Figure 4(b).

3. MEASUREMENT SETUP

The chemFET sensor array was controlled using an instrument that consists of custom interface electronics, a National Instruments card and a software control system written in LabVIEW™. The interface provides a constant current of 10 μA to the sensors and records the resultant voltage drop over the sensors. For the SH-SAW

devices a vector voltmeter / signal generator setup was used. The set-up includes a signal generator (HP 8648C), the dual SH-SAW sensor and a vector voltmeter (HP 8505A). The experimental procedure involved the measurement of both the phase velocity and attenuation of the SH-SAW signals propagating on the delay lines of the sensor. Using this setup, an electrical signal is fed from the signal generator to the input IDTs; the amplitude ratio ΔA and phase difference $\Delta\phi$ between the input and output signals of each delay line and between the output signals of the sensing and reference delay lines were monitored by the vector voltmeter. The fractional velocity shift $\Delta v/v$ and attenuation change $\Delta\alpha/k$ of the SH-SAW can be derived from the phase difference and the amplitude ratio, respectively.

All the experiments were performed under a controlled temperature using a commercial Dri-Bloc™ heater. The sensors were left for 60 minutes before testing at a constant temperature of 30 ± 0.1^{o}C at 50% r.h. A sensor baseline for the chemFET sensors was acquired for reference purposes before the injection of the liquid sample. After injection of the liquid (60 μl) into the micro-cell, using a micro-pipette (Gilson Pipetman P200), the sensor signals were monitored for a period of 15 min. The response of the chemFET sensors was defined as the difference between the baseline signal and the value recorded after 15 minutes of exposure to the liquid. The liquid cell and the SH-SAW devices were cleaned and dried after each measurement using de-ionised water and the sensors were allowed to stabilise to the control environment before conducting the next measurement. These experiments were repeated 5 times for each sample and repeated for two different chemFET sensors each sensor chip having a different polymer.

4. EXPERIMENTAL RESULTS

The initial measurements were conducted to discriminate between different liquid samples (i.e. de-ionised (DI) water, orange juice and milk). Results showing the discrimination of different liquids using principal components analysis (PCA) for the e-tongue are presented in Figure 5. The amplitude ratio and phase difference on each of the delay lines (electrically shorted and free) were used as the four parameters for the principal components analysis. In this case (and in all subsequent analyses), the original data-set was autoscaled to remove the influence of the magnitude of the sensor response. The PCA plot given in Figure 5, as expected, shows excellent spatial separation of the three very low vapour pressure liquids in linear multivariate space. The tight clusters hide the fact that 5 replicate measurements were made (in randomised sampling order) for each of the liquid samples. Previous work on SAW sensors has been carried out that investigated more similar types of drinks and 100% discrimination was observed but with higher within-group variance [13].

Figure 6 shows the transient response of a chemFET sensor to the three liquids, and although a visual difference can be noticed in the responses in the plot, it is actually very small (~ 10 μV). The parameters used were the change in gate/drain-source voltage (ΔV_{GDS}) of each of the four chemFETs along with the attenuation and phase data for each delay line of the SH-SAW devices, as mentioned earlier. PCA analysis again revealed good multivariate separation, but with reduced clustering compared to electronic tongue data alone.

Further experiments were performed on milk samples with different fat content, (whole milk with 4% fat, semi-skimmed with 2% fat and skimmed milk with no fat).

The same procedure was followed for these experiments as described earlier. Results, showing the discrimination of different milk samples, using principal components analysis for the SH-SAW device, are presented in Figure 7a. This plot again shows very good discrimination and tight clustering of the different milks samples. Some discrimination can also be observed from the response of the chemFET sensors, however not as promising as in the case of SH-SAW devices. The transient response of the one of the chemFET sensors is presented in Figure 8, with the combined PCA plot shown in Figure 7b. Here, again the clustering is not as tight compared to the SH-SAW devices alone - as was the case with the different liquids (water, orange juice and milk). Thus from the results of the two sets of experiments mentioned above, it can be concluded that the chemFET based electronic nose does not add much to the discriminating power of the system for these particular samples. This was to be expected as the liquids used in the experiments have a very low vapour pressure and thus do not generate significant headspaces (i.e. very low vapour concentrations) for the electronic nose to detect. The small variations in the responses of the electronic nose devices could be due to the difference in water content in the different samples.

Following the results gathered from the above experiments it was decided to perform experiments using a number of samples that are taste-biased and a number that are smell-biased. It was anticipated that these experiments would give encouraging results to discriminate between complex solutions that contain substances that may not be detected by the electronic tongue but would be picked up by the nose and vice versa. The details of the different synthetic samples selected for these experiments and results obtained are discussed in the following section.

Experiments with nose/tongue biased samples

Knowing that the SH-SAW electronic tongue devices respond well to ionic solutions (with very low vapour pressure) three different taste solutions were chosen along with two organic solutions (high vapour pressure) that are known to give good response from the chemFET devices. The solutions with their respective concentrations are given in Table 1 below.

Distilled DI water was used as the reference liquid sample and all the solutions were prepared or diluted (in the case of the organic samples) using the DI water. The same setup was used as for the previous experiments within the same environmental conditions. Both the chemFET and SH-SAW sensors were controlled at 30 ± 0.1°C using a Dri-Bloc™ heater and at approximately 34% r.h. As before a sensor baseline for the chemFET devices was acquired before the injection of the liquid sample for reference purposes. The electronic nose device used for this experiment was coated with composite polymer - poly(ethylene-co-vinyl acetate). After injection of the liquid (60 μl) into the micro-cell using the micro-pipette the measurements were recorded for 15 min. The liquid cell and the SH-SAW devices were cleaned and dried after each measurement, using DI water, and the sensors were allowed to stabilise to the control environment for approximately 15 min before conducting the next measurement.

Five replicate measurements were performed on each analyte and the results analysed using principal components analysis. As before, the four parameters (amplitude ratio and phase difference from both delay-lines) were used for the analysis of the electronic tongue data. For the electronic nose devices the voltage differences for each of the four chemFETs were used to describe the raw sensor

response. (N.b. as stated above the data-set was autoscaled to enable each sensor to be put on an equal footing).

Figure 9 shows a 3D principal components plot for the electronic tongue data from the five samples (see Table 1) with DI water added as the reference liquid. Three components were used here, as a 2D analysis showed an overlap between DI water and estery samples. It was expected that the tongue would not respond very well to the high vapour pressure organic solutions of relatively low concentrations (1000 ppm) and this explains the estery solution clustering close to DI water. However, the tongue still shows good separation of the ethery compound. For more information about the concentration dependence of these SAW sensors and their ability to discriminate between liquids, please see references [16].

Figure 10 gives four plots of the voltage (V_{GDS}) against time for the five different flavours and DI water, for each of the four chemFETs in the electronic nose device. (Note: the time series plots are not given for the SAW sensors because they respond almost instantaneously to the liquid (milliseconds) and the sampling rate of the frequency measurement is 1 second.) The plots for all four chemFETs show clear differences and large responses for both ethanol and ethyl acetate that have a high vapour pressure (the fourth sensor has a solid Au gate, thus giving a much smaller response). Conversely, DI water, sucrose, NaCl and quinine responses were very small with small differences in the voltage change and hence almost no discrimination. 2-D PCA plot for the electronic nose voltage difference data could not separate all the flavours as demonstrated by the transient responses. Ethanol and ethyl acetate are clearly separated from the rest of the analytes, as expected, due to their high vapour pressure, hence easily sensed by the electronic nose. The effect of

introducing a third principal component to improve the separation is demonstrated in a 3-D PCA plot (see Figure 11). It can be concluded that the electronic nose could not discriminate between all the flavours, however it clearly separates the high vapour pressure ethereal and estery samples. For more information about the behaviour of these polymer FET devices, such as temperature and humidity dependence, please see reference [17].

Finally, in order to determine the discriminating power of the combined system, PCA was performed on normalised electronic nose and tongue data together. Figure 12 shows the 3D PCA analysis of the combined nose/tongue data where good separation was achieved using a third principal component. From a plot of the sensor loadings given in Figure 13, the relative weighting of the e-nose and e-tongue can be determined. The electronic nose did not separate out the low vapour pressure flavours and water however it gave very distinct responses to the high vapour pressure estery and ethereal compounds (n.b. sensor loadings were smaller and three FET devices were close together). The electronic tongue did not respond very well to the high vapour pressure organic liquids, however it gave very good separation between low vapour pressure samples (n.b. SAW sensor loadings were better distributed than FETs). Thus a combination of the responses from both the nose and tongue devices, as expected, gave total separation between all the samples demonstrating promising application of this combined system in the flavour analysis of liquids.

5. Conclusions

A combined tongue/nose flavour system comprising of the SH-SAW liquid sensors and the chemFET gas sensors has been developed and thus differs from a previously

reported system using electrochemical cells and metal oxide gas sensors [14,15]. The uncoated SAW devices should not only be more robust than electrochemical sensors but also respond almost instantaneously. Moreover, polymer coated gas sensors are attractive in that they operate at room temperature (low-power) and do not suffer sensitivity to common headspace gases such as oxygen, methane, and hydrogen.

Results from initial experiments performed using our combined flavour system on complex samples (such as milk samples with different fat content) and different solutions (such as water and orange juice) showed little contribution from the electronic nose devices. However, the use of the combined nose/tongue system was justified with further experiments where three taste-biased solutions with low vapour pressure and two organic high vapour pressure smell-biased solutions were tested with the system. The results of this investigation concluded that, unlike the case of individual devices, the combination of the electronic nose and electronic tongue produced 100% discrimination between all the different flavours. These results are encouraging and indicate the potential of the flavour sensing system to analyse complex solutions in the food and beverage industries where the sole use of an electronic tongue or electronic nose has become inadequate. Furthermore, the choice of polymer based FET devices and SAW devices enables room temperature operation and so a low-power solution for handheld, battery-operated devices.

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FIGURES

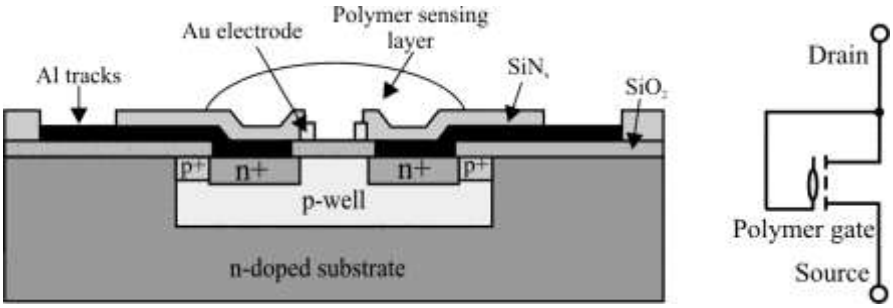


Figure 1: Schematic overview of chemFET sensor and configuration

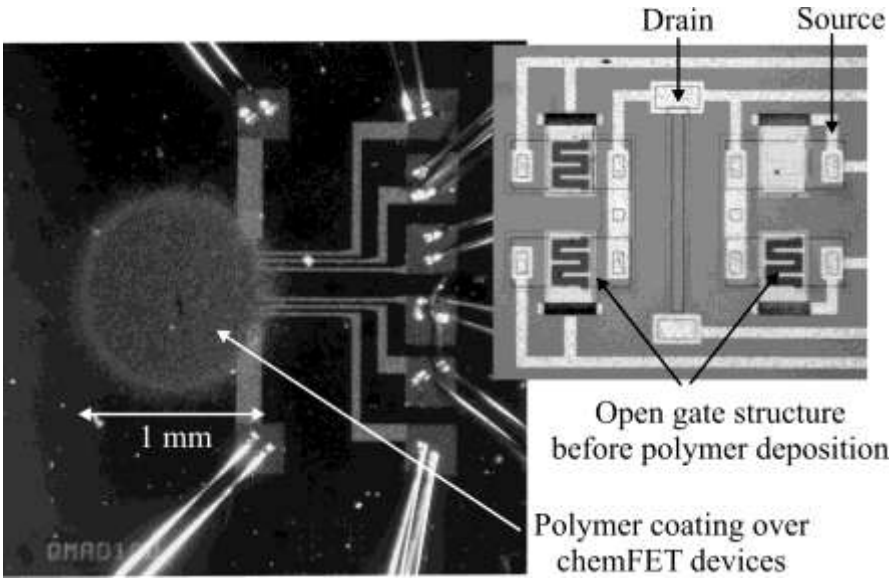


Figure 2: Photographs of uncoated and coated chemFET sensor

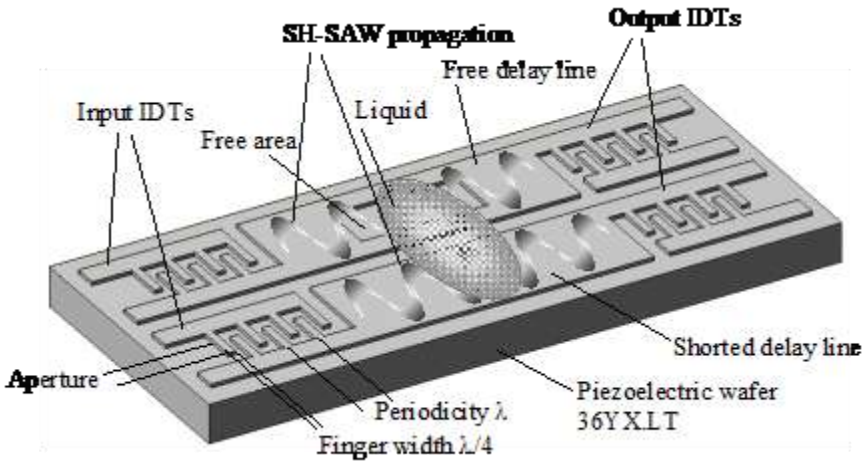
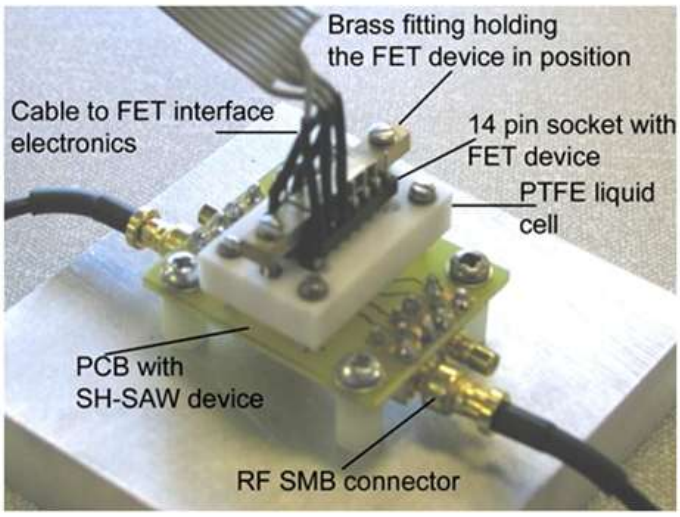


Figure 3: Schematic of dual-delay line SH-SAW taste sensor

(a)



(b)

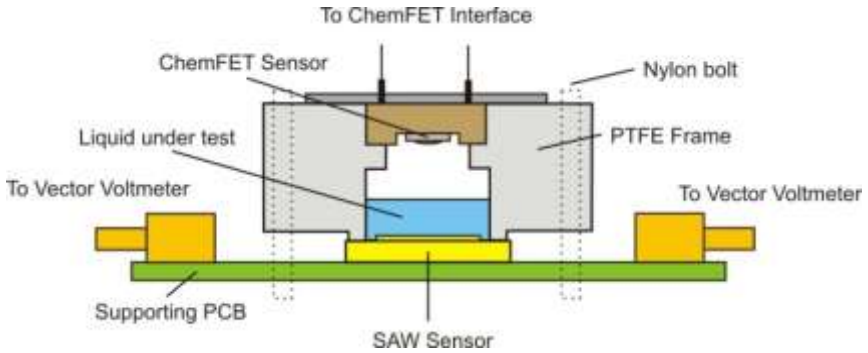


Figure 4: (a) Photograph of miniature total analysis taste system or electronic tongue and (b) schematic cross-section of the flavour sensing system showing the SAW sensor (bottom), liquid chamber (32 mm × 20 mm with a central reservoir of approximately 136 µl volume) and FET sensors (top).

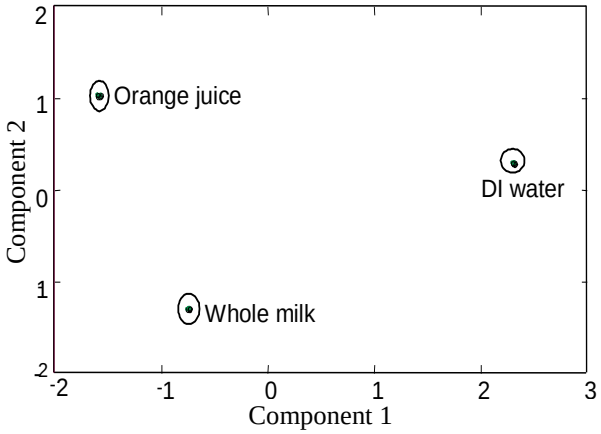


Figure 5: PCA of liquids with the e-tongue showing excellent linear discrimination. Each cluster is made from 5 replicate samples.

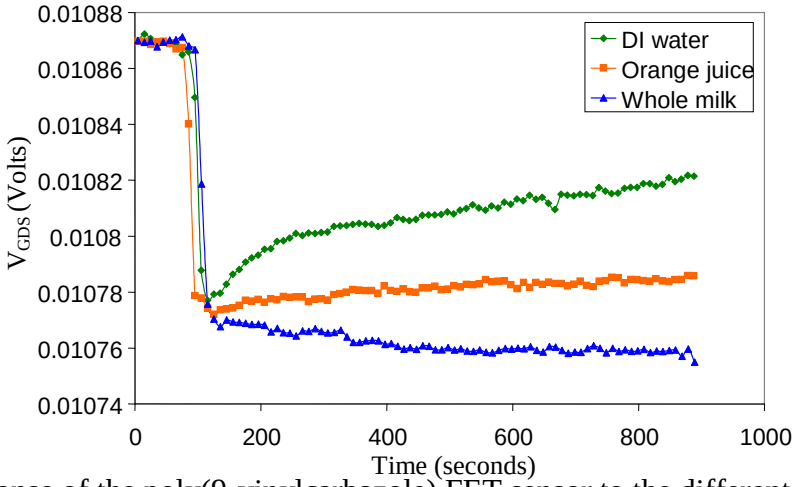


Figure 6: Response of the poly(9-vinylcarbazole) FET sensor to the different liquids

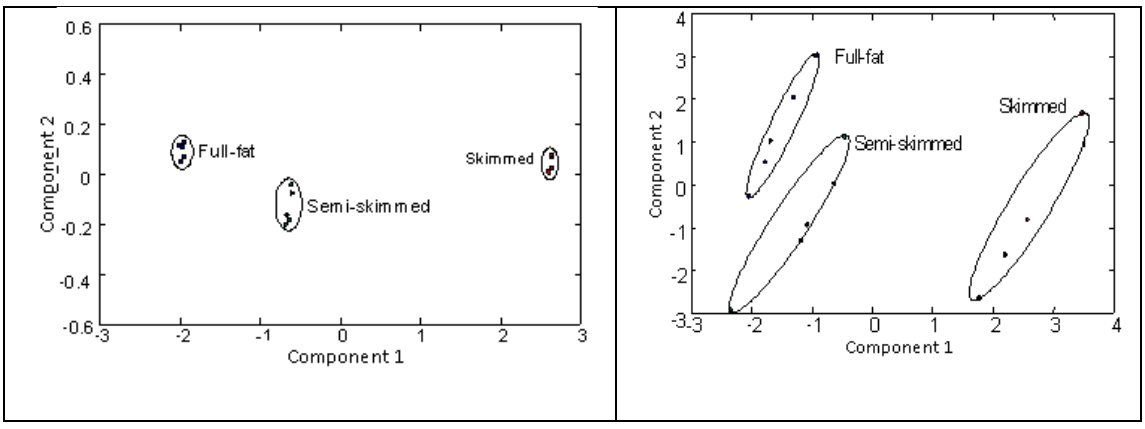


Figure 7: PCA analysis of (a) electronic tongue data and (b) combined electronic nose and tongue to milks of different fat content.

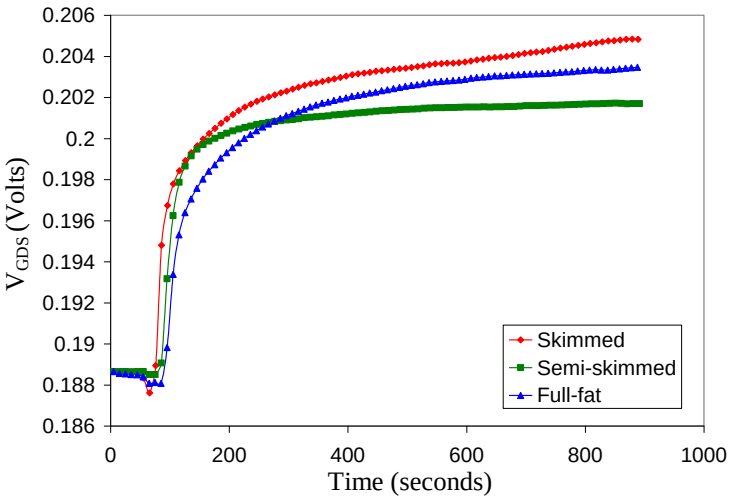


Figure 8: Transient response of the poly(styrene-co-butadiene) FET sensor to milks of different fat content (full fat is 4%, semi-skimmed 2%, skimmed 0.3%).

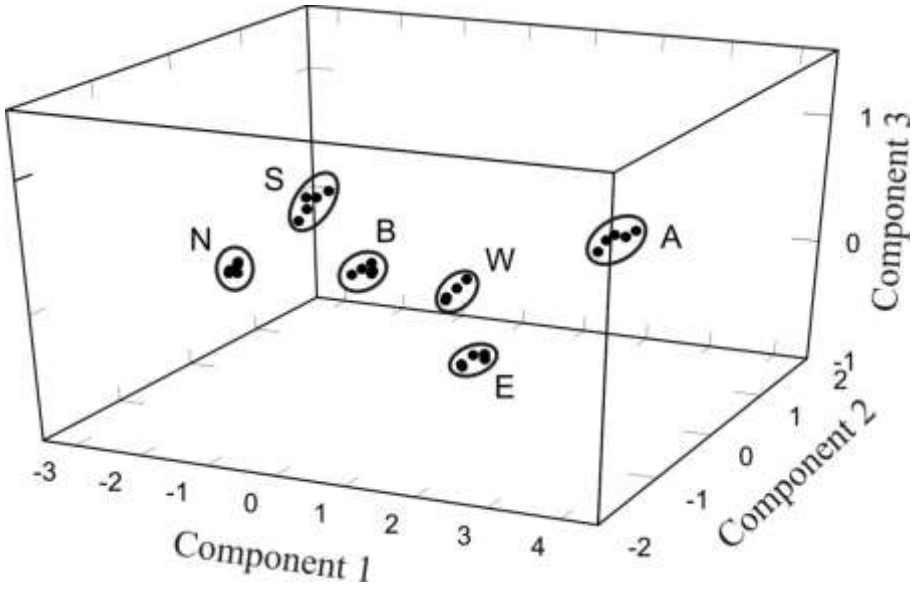


Figure 9: 3-D PCA plot on the attenuation and phase data of the SH-SAW sensor.

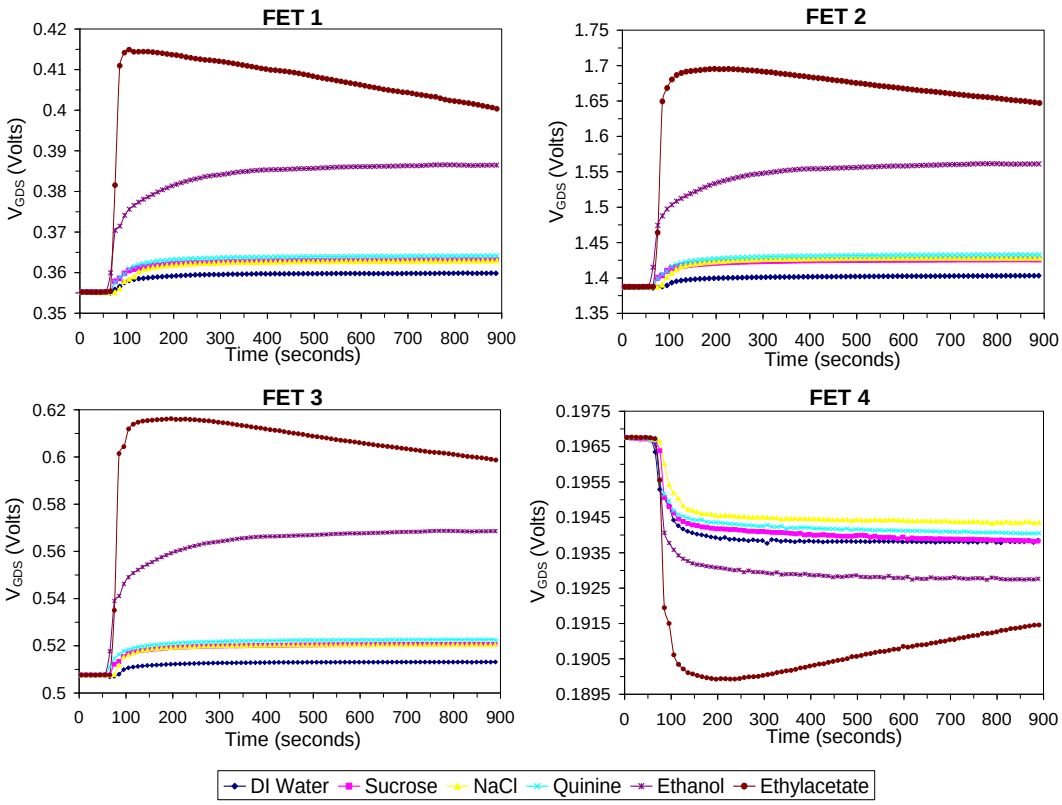


Figure 10: Transient responses of the 4 different ChemFETs in the array to each of the flavour samples

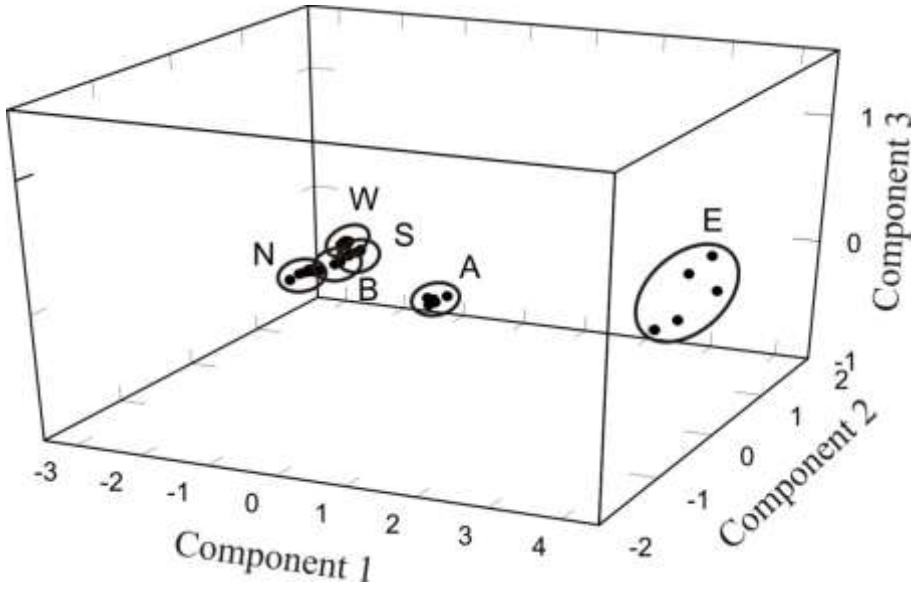


Figure 11: 3-D PCA plot for the ΔV_{GDS} data from the 4 chemFETs

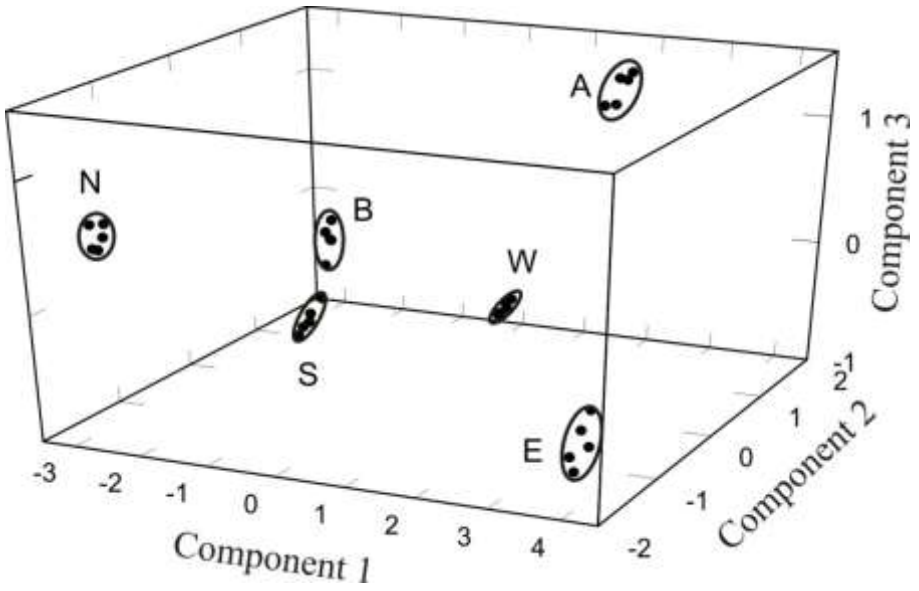


Figure 12: 3-D PCA plot of the combined electronic nose/tongue data

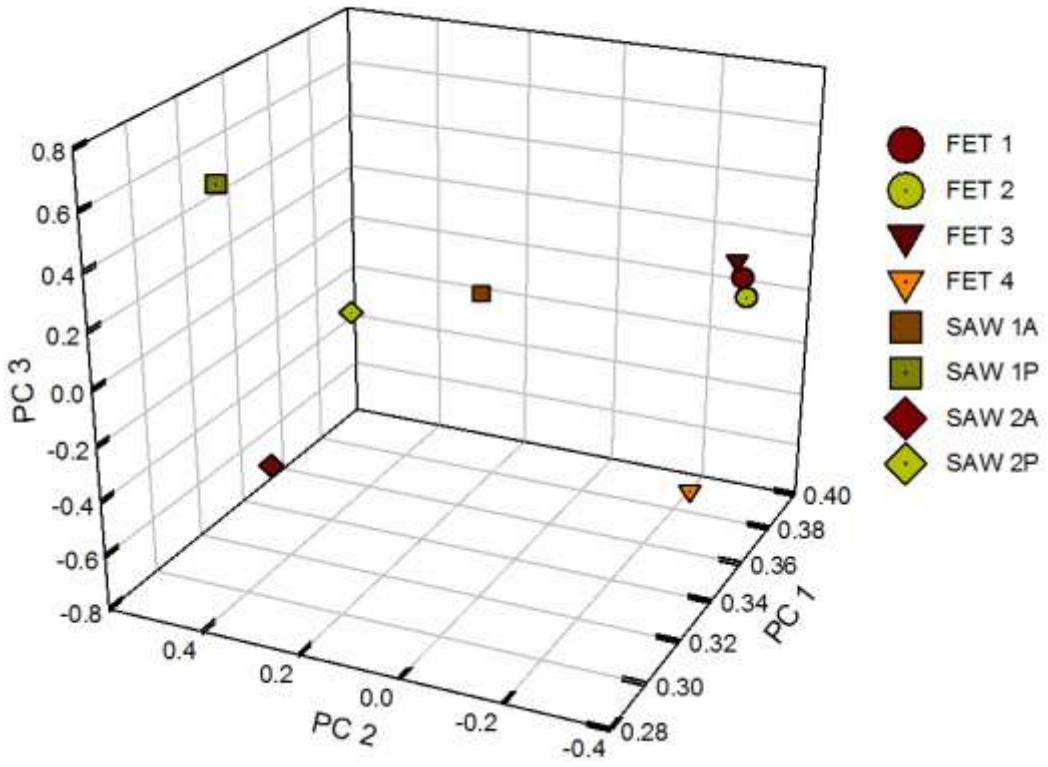


Figure 13: 3-D PCA plot showing the loadings of each of the 8 sensors for the first three PC components using the combined electronic nose/tongue data-set.

Biographies

Marina Cole BSc, PhD, MIEEE, received her BSc degree from the University of Montenegro (Yugoslavia) and the PhD from Coventry University (UK). She joined the School of Engineering at Warwick University in 1996 as a postdoctoral research assistant and in 1998 she was appointed to a lectureship in electronic engineering. Her main research interests are integrated silicon-based sensors, SAW-based sensors, analogue and mixed signal ASICs, smart sensors, actuators and Microsystems.

James Covington BEng, MRes, PhD, MIET is presently an associate professor in the School of Engineering at the University of Warwick. He received his BEng in 1996 in Electronic Engineering and remained there receiving his PhD in 2000. His PhD was on the development of CMOS and SOI CMOS gas sensors for room temperature and high temperature operation. He worked as a research fellow for both Warwick University and Cambridge University on the development of gas and chemical sensors and was appointment as a lecturer in 2002, being promoted to associate professor in 2006. Current research interests focus on the development of silicon devices with novel materials using CMOS and SOI ASIC technology (nose-on-a-chip), and biologically inspired neuromorphic devices with applications based on environmental and biomedical engineering.

Julian Gardner *BSc PhD DSc FREng FIEE SMIEEE* is Professor of Electronic Engineering in the School of Engineering at Warwick University. He is author or co-author of over 450 technical papers and patents as well as six technical books in the area of microsensors and machine olfaction. He is a fellow of the IEE and senior member of the IEEE and has served on many advisory panels on sensors, e.g. for EPSRC, DTI and IEE Professional Network on Microsystems and Nanotechnology. He has worked with over 20 companies in the past 15 years developing commercial e-nose instruments and a consultant for various companies. He is also Head of the Sensors Research Laboratory and Director of the Centre for Cognitive & Neural Systems. He was elected a Fellow of the Royal Academy of Engineering in 2006 and awarded the JJ Thomson Medal for Outstanding Achievement in Electronics by the Institute of Engineering & Technology in 2007. His research interests include the modelling of silicon microsensors, chemical sensor array devices, biomimetic MEMS devices and electronic noses.