

Novel Materials and Applications of Electronic Noses and Tongues

Perena Gouma and Giorgio Sberveglieri,
Guest Editors

Abstract

This introductory article describes the content of the October 2004 issue of *MRS Bulletin* focusing on novel materials and applications of electronic noses and tongues. The articles in this issue review the state of the art in materials, devices, and data processing algorithms used in electronic olfaction and taste systems. The most common gas- and liquid-phase analyte detection tools are presented and compared with traditional chemical analysis instrumentation such as gas chromatography/mass spectroscopy systems. Metal oxides, polymer/polymer composites, and dyes are covered in these articles as key sensing materials. Resistive, optical, electrochemical, and other types of electronic nose and tongue systems are reviewed, and their use in diverse applications, including environmental and food-quality monitoring and medical diagnostics, is discussed.

Keywords: biosensors, chemical detectors, electronic noses, electronic tongues, medical diagnostics, sensor arrays.

An increasing demand for electronic instruments that can mimic human olfactory processes and that may provide low-cost, rapid sensory information to hasten the process of odor evaluation for applications such as food-quality assessment, environmental monitoring, and even forensics has led over the years to the conceptualization of an "electronic nose." This is defined as an intelligent chemical sensor array system for odor classification.^{1,2} Electronic nose systems, including the materials used to build the sensing elements, device architecture, and intelligent signal processing routines, are the focus of this issue of *MRS Bulletin*. Key developments in the domain of artificial olfaction are reviewed through the contributions of leading experts in this field. The most common sensor materials and technologies used, those based on semiconducting oxides, polymers, and dyes, are identified, and a variety of other types of sensor materials and technologies, such as electrochemical detectors used in "electronic tongues" (taste sensor arrays able

to operate in liquid environments), are discussed.

The importance of reviewing the state of electronic nose and tongue technology lies in the current need for advanced detection devices for security (both civilian and military) and health safety applications, such as the detection of explosives and infection monitoring. Furthermore, the need for biochemical detectors that are able to sense the presence of pathogens in humans and that can contribute to the early detection of diseases is high.

Recently, medical applications of electronic noses have been explored. The use of a novel electronic nose to diagnose the presence of pulmonary infection^{3,4} and distinguish between serum and cerebrospinal fluid, as might be encountered in exudates collected from the eye or ear, has been reported.⁵ Osmetech has obtained approval from the U.S. Food and Drug Administration to use its multiple-detector-based device for the detection of urinary tract infections in patients and is currently

seeking approval for use of the device in the diagnosis of bacterial vaginosis.⁶

Clearly, this field is still in its infancy, and the opportunities for developing improved biosensors are abundant. The diverse nature of their applications appeals to the public in general. It is envisioned that handheld electronic nose/tongue devices will be the health monitoring technology of the future, whereby physiological functions may be continually monitored through a simple exhaled breath or nasal-expired air.

At the same time, novel materials such as chemoselective polymers and bio-nanocomposites, carbon nanotubes, and oxide nanobelts and nanowires have revolutionized the field of biochemical sensing.⁷⁻¹⁰ Nanoscience and nanotechnology involve materials with dimensions in the scale of 1–100 nm and rely on the size-dependent properties of these materials. For example, the high surface to volume ratio of nanostructured materials favors gas adsorption on these surfaces, thus enhancing the sensitivity of the sensor. Modifications in the electronic structure of nanoscale semiconductors can affect the optimum temperature for gas sensing. By employing these new materials, advanced sensing systems are being developed that are faster, more selective, and highly sensitive to harmful chemical species and "disease signaling" gases. The issue of the chemical selectivity of each sensor component is currently being compensated for through the use of pattern recognition algorithms and neural network routines that process the signal from nonselective sensing elements and define a spatial distribution of the electronic nose responses to different types of chemicals.

Sensor Materials and Applications for Electronic Noses and Tongues

Traditionally, gas analysis has relied on gas chromatography and mass spectroscopy (GC/MS) systems. GC/MS is used to identify and quantify volatile and semi-volatile organic compounds in complex mixtures. Components of a chemical mixture are separated in the gas chromatograph and identified by their respective masses in the mass spectroscope. Organic compounds must be dissolved in volatile and organic solvents for injection into the gas chromatograph. The duration of a gas chromatographic run is between 20–100 min, and this is the instrument analysis time. GC/MS may identify unknown organic compounds by matching spectra collected with reference spectra or by *a priori* spectral interpretation. Data analysis can take more than 20 h. Such analytical systems, although accurate in identifying volatile

organic compounds, are expensive and bulky and require experienced operators, whereas electronic noses and tongues offer the promise of fast, reliable, portable gas-sensing and/or liquid-detection systems for user-friendly operation. Microfabricated sensor arrays are currently built in small, handheld devices. Each sensor response varies from seconds to minutes. Data processing can be simplified to provide a straightforward reading of the gas chemistry and concentration or the quality of the odorous mixture.

Metal Oxide Sensors

Semiconducting oxides have been the preferred low-cost sensing elements for the detection and monitoring of permanent gases such as CO. They show high gas sensitivity, fast response to the presence of the gaseous analyte, and good long-term stability. The detection process of oxidizing/reducing gases by semiconducting metal oxides involves the change in oxide conductivity in the presence of the gas due to catalytic reduction/oxidation reactions occurring at the oxide surfaces.¹¹ These catalytic reactions are controlled by the electronic structure of the oxide system used, as well as by the chemical composition, crystal structure, and relative orientation of the surfaces of the oxide phase(s) exposed to the gas.¹² There has been evidence in the literature of selective detection of a particular gaseous analyte in the presence of interfering gas mixtures (i.e., sensor selectivity),^{13,14} which is largely determined by the chosen crystalline polymorph (specific crystallographic phase) of a stoichiometric and pure metal oxide used for sensing. For example, CO and hydrocarbons are sensed by rutile-type structures¹⁵ such as the polymorphs of SnO₂ and TiO₂, while oxides with the perovskite structure may be used to sense oxidizing gases with higher sensitivity. Because a given crystal structure may be sensitive to more than one gas, sensing tests at different temperatures are typically carried out so as to identify the optimum operating temperature for the specific sensor.¹⁶ It is important to remain within the phase stability field of the particular polymorph of the oxide to attain reliable and reproducible sensing properties.

The use of metal oxides in electronic noses is discussed in this issue, in the article by Pardo and Sberveglieri. In this article, the authors review two case studies in which the Pico electronic nose was used for food-quality assessment and environmental monitoring.

There is an increasing trend in chemical sensing to utilize nanostructured oxides, such as SnO₂ nanobelts, as gas-sensing el-

ements.⁸ Nanocrystalline processing may also be used to stabilize oxide polymorphs that would otherwise be energetically unfavorable under normal testing conditions, such as the anatase phase of TiO₂, as opposed to its stable rutile phase.¹⁷ For resistive-type chemical sensors, it was observed that the sensing properties, such as response time and gas sensitivity, appeared to improve when the size of the oxide particles was reduced.^{8,13}

Polymer-Based Sensors

The most common gas-sensing elements rely on sorption-based detector materials, such as conductive polymers or composites.¹⁸ The swelling of polymers due to adsorbed chemical species can change the electrical properties of conductive polymers, as well as the oscillation frequency of polymer-coated cantilever devices. Both conductivity and oscillation frequency can be used as analytical parameters; conductivity is discussed here, while oscillation is covered in the "Other Sensor Materials and Technologies" section. The principle of gas detection by conductivity changes is the adsorption of the volatile analytes on composites consisting of a conductive matrix blended with polymers for which the analytes have variable affinities.^{19–21} As the analytes adsorb to the polymers, the dimensions of the composites change slightly, resulting in small but detectable changes in conductance. Various polymers, especially polyheterocycles such as polypyrroles, have been employed for their capacity to bind volatile analytes.

Although there are clearly differences in the affinities of a number of gas-phase analytes for these polymers, there is little absolute selectivity in the absorptive process on which the detectors depend for recording conductance changes. The responses of these sensing materials depend on their molecular volume, branching of the polymer chain, hydrogen bonding, etc. It is not clear yet what the effect of each of these parameters is on gas selectivity. The absorptive process has been likened to the association of volatiles with an organic solid phase in gas-liquid chromatography. Even with the best-designed composites, the kinetics of adsorption and desorption of the volatile analytes have half-lives on the order of hundreds to thousands of seconds, which suggest relatively long gas-detection times.

Because the composites respond to adsorbed volatile analytes by changes in dimensionality that lead to altered conductance, the choice of polymers may be compromised by the need to achieve a conducting composite. One strategy employs polymers with some intrinsic con-

ductance (several of the polyheterocycles have this property and are therefore favored in the design of composites): this approach limits the range of adsorptive selectivity. Another approach²² employs nonconductive polymers, such as poly(4-vinylphenol), poly(ethylene oxide), and ethyl cellulose, blended with carbon black, which serves as the conducting component of the composite. Both strategies result in detectors that are most sensitive to volatiles with a relatively high vapor pressure, such as fatty acids and related alcohols, rather than permanent gases such as methane, NO₂, or CO. Some of the relatively high-vapor-pressure organic acids and alcohols are products of bacterial or yeast fermentation and are therefore encountered in environments such as the headspace of vessels used in winemaking.

The principles of polymer-based sensors are discussed in this issue by Dutta et al. This article describes a commercial electronic nose system based on polymer-carbon black composite sensors. This technology relies on a resistive-type detection mode that senses the change in resistivity as the polymer films swell. The detectors used in this electronic nose are nonselective. Thus, the sensor response is not correlated with the concentration of a single gaseous species (chemical compound), but is a combination of all the chemical information contained in the sample, which in this case is the "smell print" formed in the headspace of bacteria solutions.

A different application field for polymer-based sensors is reviewed by Ryan et al. In this case, the application field is primarily the space shuttle environment and space habitats in general. Molecular modeling of the sensor response is also covered in this review.

Other Sensor Materials and Technologies

The LibraNose is an example of an electronic nose based on eight quartz microbalance (QBM) sensor arrays coated with metalloporphyrin compounds as the chemically sensitive materials.²³ The operation principle relies on the variation of the fundamental oscillating frequency (Δf) of a thin quartz crystal as a result of the adsorption of gas analyte molecules on its surface, which changes the oscillating mass (Δm) of the system, as described by the Sauerbrey law.²⁴ Good sensitivity was obtained with this system for aromatic compounds, amines, alcohols, and ketones. LibraNose was used for lung cancer identification by breath analysis in a study involving 60 individuals.²³ Certain volatile compounds found in the exhaled human breath of individuals with lung cancer,

mostly alkenes and benzene derivatives, are considered candidate markers of this disease. Di Natale et al.²³ had their test subjects breathe in a 4l volume disposable bag; the sampled bags were then analyzed on-site with the electronic nose. Using multivariate data analysis to process the obtained data, complete identification of the samples from diseased individuals was possible.²³

Polymer-coated cantilevers (e.g., micro-fabricated beams of silicon) have been considered for use as nanomechanical sensor devices in detecting physical/chemical interactions between the reactive layer on the surface (a polymer film) and the environment.²⁵ Swelling of the polymer upon interaction with volatile species forces the cantilever to bend because of surface stresses when used in static mode. In dynamic mode, the cantilever acts as a micro-balance driven at its resonance frequency. Changes in mass as low as 1 pg (caused by binding reactions) change the resonance frequency of the oscillating cantilever. The addition of biochemically active layers onto the cantilever surface enables the monitoring of mass changes during molecular-recognition reactions.

The reliability of these sensing materials depends on the precision in microfabrication that is required to form structures with reproducibility in resonance frequencies better than a few tenths of a percent. Such cantilever array sensors (consisting of eight polymer-coated cantilevers) were used to detect acetone in exhaled air.²⁵

Another electronic nose technology reviewed in this issue involves an optoelectronic nose using colorimetric sensor arrays, discussed here by Suslick. These colorimetric sensors consist primarily of metal-organic compounds deposited on porous polymer membranes or other inert solid supports. Metalloporphyrins in particular are the sensing elements of choice in this study. The reason for their selection is that mammalian olfactory receptors are metalloproteins and most odorous compounds are excellent ligands for metal ions. Their chemical selectivity and sensitivity depend on the nature of the central metal and peripheral constituents of the porphyrin complex.²⁶

Extending the use of electronic noses to liquid environments, mass-sensitive devices have also been used in electronic tongues. This issue concludes with the article by Winquist et al. reviewing the current state of the art for electronic tongues. Special emphasis is paid to electrochemical detectors and voltammetric devices in particular. Finally, emerging trends in taste sensors include the application of spectroscopic methods in the optical tongue paradigm of Fourier transform infrared-based sensing.²⁷

Pattern Recognition and Multivariate Chemometric Methods

Data analysis and recognition processes are not usual areas for materials scientists to focus their efforts in; however, these are key aspects of electronic olfaction technology. Electronic nose data analysis correlates each tested sample to a vector in multidimensional space by means of classical nonparametric techniques. These are mathematical procedures that make no assumptions about the frequency distributions of the variables being assessed. Principal component analysis (PCA) algorithms are used to project the data sets into two dimensions (principal components). In this way, maximum distinction performance between subject classes is achieved, as well-separated clusters of measurements are projected in principal component space.

In another approach, multivariate analysis is used to extract the maximum amount of information from the sensor array. Such parametric statistical methods assume that the distribution of the variables being assessed have certain characteristics [e.g., analysis of variance (ANOVA) assumes that the data obtained are normally distributed].

Artificial neural networks are also used for further processing of the data from the electronic noses for improving the analyte identification rate. These networks consist of hierarchically organized layers of information processing elements similar to the biological nervous system and have a learning ability that sets them apart from the other classification methods.^{1,2} The articles in this issue present the different levels of complexity in the subject data analysis and recognition process of electronic noses and tongues.

Considering the future of electronic nose technology, there are two approaches that seem to naturally evolve. One is the use of hybrid (or orthogonal) electronic olfaction and taste systems composed of more than one type of sensor (e.g., metal oxide-resistive and polymer composite-resistive) or arrays of hybrid sensing elements (e.g., chemoselective membranes deposited on metal oxide sensing films). The second approach is to use small detector sets (2–3 sensor arrays) with high specificity, targeted for a given application. In both cases, emphasis is placed on improving the semiselective nature of sensor materials and reducing the need for complex algorithms for signal discrimination.

Nanotechnology is expected to have a major impact on shaping the future of the fields of biochemical detection as new materials, structures, and devices with improved properties are developed. For example, novel nanomanufacturing processes

such as electrospinning²⁸ produce self-standing pure or composite material nanostructures (membranes) with high surface areas for enhanced chemical attachment of analyte species and biocatalytic processes that are suitable for on-line monitoring and/or advanced power systems (e.g. bio-fuel cells). Therefore, the electronic noses of the future are envisioned to be tiny devices that will fit in a wristwatch or a toothbrush that will alert us to health problems and protect us from exposure to allergens and pollutants. Similarly, miniaturized electronic tongues will taste the freshness of our food and the purity of the water we drink. All of these devices will be inexpensive and easy to operate. It is this kind of improvement in human welfare through novel technology that materials scientists are striving for.

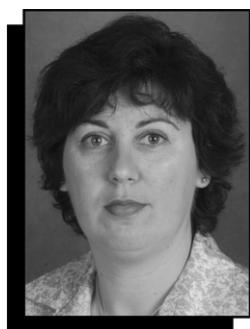
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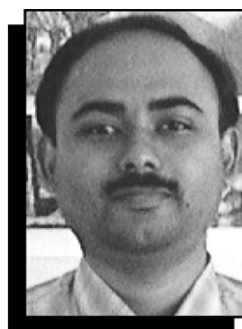


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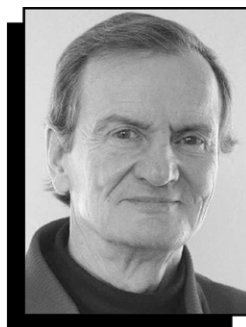
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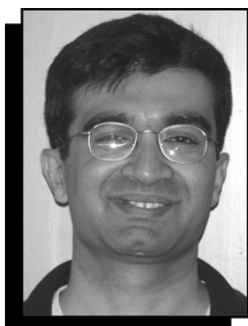
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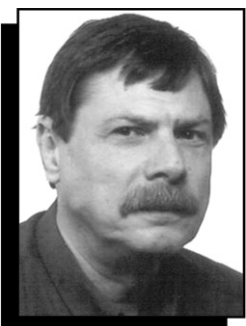
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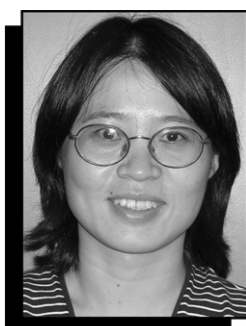
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Suslick's recent interest in electronic nose technology led to the founding of ChemSensing Inc. in 2002. He has published more than 250 papers, edited four books, and holds 16 patents. He is the recipient of the Materials Research Society Medal, the CS Nobel Laureate Signature Award for Graduate Education, the ACS Senior Cope Scholar Award, the Wolfgang Göpel

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physics. Initially, he studied thermometric sensors in combination with enzymes and later on developed an ammonia-gas-sensitive field-effect semiconductor structure that was also used for biosensor applications. Further development of these types of semiconductor structures eventually were applied to the concept of electronic noses. He has also developed an electronic tongue based on voltammetry. Winquist's current interests concern sensor systems, artificial senses, multivariate data analysis, imaging ellipsometry, and biosensors.

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