

Chapter 22: Environmental Monitoring

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ABSTRACT

In this chapter, we review some of the previous *proof-of-principle* work done in this field. Examples of water, land and air monitoring experiments are examined. Four case studies are then presented. The first three demonstrate the ability of the e-nose to classify odors from animal confinement facilities (odor source determination, odorant threshold detection, and odor abatement evaluation). The fourth case study demonstrates that the e-nose can differentiate between five types of fungi that commonly lower indoor air quality in office buildings and industrial plants. Finally, we conclude that environmental monitoring is a promising application area for electronic nose technology.

1. Introduction

The field of environmental monitoring encompasses a broad range of activities. Contamination of the environment can occur not only by dumping wastes in water, land, and air, but also by generating noise in the audio and communications frequency ranges. Sensing systems have been developed for all of these applications. In this chapter, we focus on efforts to employ an electronic nose (e-nose) to monitor airborne volatile organic compounds that are released when waste products are dumped in water, land or air.

1.1. Water

Water quality is threatened when agricultural and industrial concerns allow their waste products to seep into ground water or to flow into streams or rivers. The e-nose can be used in these applications by collecting samples of the effluent. The headspace of such samples can be tested with an e-nose system, on-line or off-line, to establish the time-course of emission profiles. Boreholes can also be employed to collect samples to test groundwater contamination. Several research groups have studied the e-nose as an instrument for monitoring water quality.

Some teams have utilized metal-oxide sensors for monitoring. Baby et al. [1] used the MOSES II e-nose to measure contaminating residues of insecticides and products from leather manufacture that are often offloaded into streams and rivers. Dewettinck et al. [2] employed an e-nose consisting of 12 metal-oxide sensors to monitor volatile compounds in the effluents of a domestic wastewater treatment plant over a 12-week period. Correlation between the relative overall e-nose output and the parameter "volatile suspended solids" (VSS) was good, indicating adsorption of volatile organic compounds (VOCs) onto the organic particles. This study also concluded that the e-nose has promise in wastewater monitoring applications. In another study

by the same group, Van Hege et al. [3] explored the application of evaporative technology as an alternative desalination technique for wastewater treatment plant effluents. Evaporation completely removed most inorganic and organic contaminants. An e-nose was employed to monitor changes in odor quality and intensity due to volatilization of the VOCs present in the effluent.

Conducting polymers have also been used to analyze wastewater. Di Francesco et al. [4] studied the use of an e-nose with conducting-polymer sensors and fuzzy-logic-based pattern recognition algorithms to test wastewater samples. Fenner and Stuetz [5], Stuetz et al. [6], and Stuetz et al. [7] employed an e-nose with 12 polypyrrole conducting-polymer sensors to monitor quiescent sewage liquors at three wastewater treatment plants over an 8-month period. They evaluated the e-nose as a replacement for human panels in monitoring liquid wastewater samples, wastewater odor, and tainting compounds in water supplies. The study revealed that a strong linear relationship can be expected for site/source-specific odor samples. The study also showed that low levels of organic pollutants can be detected by monitoring water samples with the e-nose. The study also suggested that it might be feasible to use an e-nose to monitor and/or control the biochemical activities of a wastewater treatment process. More recently, Bourgeois and Stuetz [8] reported the use of a similar sensor array to analyze wastewater samples sparged with N₂ gas in a temperature-controlled flow-cell. The headspace gas was then supplied through a temperature-controlled transfer line to the conducting-polymer sensors. They concluded that an externally generated headspace gas could be used to monitor changes in wastewater quality, and could provide a simple non-invasive technique for on-line monitoring of wastewater.

Continuing this avenue of research, Stuetz et al. [9] and Bourgeois et al. [10] examined the use of real-time sensors and array systems for monitoring global organic parameters such as

biochemical oxygen demand (BOD) and total organic carbon (TOC). Stuetz et al. [9] and Stuetz [11] compared the odor profiles of sewage liquids with corresponding BOD, TOC measurements, and determined that a number of different wastewater quality relationships could be formulated from the e-nose analysis of a sewage liquid. They concluded that the organic content of wastewater, as well as the potential of wastewater to produce nuisance odors, can be predicted from a single headspace analysis of a sewage liquid using a sensor array.

Di Natale et al [12] used a sensor array of ion sensitive electrodes to analyze polluted water. The sensor array was processed using chemometrics, non-linear least squares and neural networks. These devices that use sensor arrays to test liquid samples are called electronic tongues rather than electronic noses. See Chapter 14 for more information on electronic tongue devices.

Gardner et al. [13] and Shin et al. [14] developed a system for detecting cyanobacteria (blue-green algae) in potable water. The e-nose system, employing an array of six commercial gas sensors, was able to detect 100% of the unknown toxic cyanobacteria using a multi-layer perceptron (MLP) neural network. The results showed the potential for a neural network-based e-nose, as opposed to more traditional instruments such as liquid chromatography or optical microscopy, to test the quality of potable water.

1.2. Land

Land contamination by toxic and radioactive materials is a chief concern in many countries around the world. Garbage waste dumps are problems all over the world. The e-nose has application in this arena as well. Borehole samples can be placed in sample containers to generate headspace VOCs. Adding specific reagents to some of these samples can accelerate the

generation of VOCs and improve the sensitivity of the e-nose instruments. This is an emerging area for e-nose instrumentation and there should be considerable future growth in this segment of the e-nose market.

There have been few research studies in this area. One example of note is Biey and Verstraete [15]. They investigated the use of a five-Watt UV lamp, generating ozone seven hours per day, to reduce the odors produced by the decomposition of kitchen and vegetable waste. They used an Alpha M.O.S. FOX 3000 e-nose to measure odor levels before and after treatment. They concluded that the UV treatment did indeed reduce the odor levels, and thus would be useful in summer, or year around, in warm climates.

1.3. Air

Air quality has been the primary target of e-nose research projects in environmental monitoring [16, 17]. The e-nose can monitor odorous emissions at their source, such as paper mills, animal production sites, power-plant stacks, vehicle exhaust pipes, compost facilities, wastewater treatment plants, animal rendering plants, paint shops, printing houses, dry cleaning facilities, or sugar factories. The e-nose also holds promise for monitoring emissions from near-source or remote locations, in a populated area. Currently, available sensor arrays have not proven efficient at remotely located sites, due to their lack of adequate sensitivity to many of the offending VOCs in odorant mixtures. However, e-nose measurements made at the source could serve as input to mathematical emission dispersion models that can predict VOC concentrations at remote locations given accurate meteorological data for a specific geographic location [18]. As sensor-array technology improves, the measuring of odorous VOCs at remote locations will become a significant market for hand-held e-nose devices (see Chapter 12).

Although in most cases annoying atmospheric emissions do not menace public health, they do greatly reduce the quality of life [4, 19]. Measuring these odors at the site of complaints is very difficult due to the transient nature of the odorous events. The e-nose offers the promise of being able to make accurate and repeatable measurements of odor profiles at sites of complaint. These e-nose measurements can be correlated with those of human panels in order to calibrate the odor quality and perception scales [20] (see Case Study 3 in this chapter).

Now we discuss several examples of the application of the e-nose to monitoring air quality. Odor abatement and control is a major issue facing municipal sewage treatment facilities. The odors emitted from these facilities can be monitored by an e-nose. Gostelow et al. [21] reviewed various sensory, analytical and e-nose methods for monitoring sewage facility emissions. Stuetz et al. [22] and Stuetz et al. [23] employed a Neotronics NOSE to investigate emissions from ten sewage treatment facilities. Odor levels measured by the NOSE unit were compared with those of an independent human panel, measured in odor units per cubic meter. The effect of biofilters was also considered. A linear relationship was observed between the NOSE measurement and the human panel results for data at each independent site. At low odor levels, the results were also extended to the multiple site case. Hydrogen sulfide (H_2S) concentrations, although commonly used as a measure of odor strength, were also compared with the human panel results and were found not to be a reliable marker compound for measuring sewage odor concentrations.

The perception of the quality of indoor air by building inhabitants is addressed by Schreiber and Fitzner [24, 25]. Delpha et al. [26, 27] investigated the use of an e-nose using metal-oxide TGS sensors for the detection of a leaking refrigerant gas (Forane R134a) in an air-conditioned atmosphere. First the researchers showed that the time response of the TGS sensors

to Forane R134a gas in humidity varying from 0 to 85% can be represented by a double exponential model. The authors then demonstrated the ability to identify the target gas by discriminant factorial analysis even for cases in which the relative humidity or the gas temperature were outside the range of the training database. In a similar study, Sarry and Lumbreras [28] investigated the detection of carbon dioxide, Forane R134a or their mixtures, without a sensor dedicated to carbon dioxide measurement. They used an array of five tin-dioxide sensors. Discriminant factorial analysis was used for processing the data. The authors report a reliable system can be designed for this application.

Ramalho [29] analyzed the characteristics of indoor paints and their effect on perceived indoor air quality. Ten different indoor paints were presented to an e-nose and to 13 trained panelists. Significant differences among panelists were found, whereas the sensors displayed little difference. However, some similarities were found between some sensors and individuals.

Feldhoff et al. [30] compared the ability of an Alpha M.O.S. FOX 4000 and a LDZ Laboratory Smart Nose GA 200 to differentiate between twenty Diesel fuels from three different refineries. The authors reported that both units were able to correctly identify the production site of the 20 samples. However, the Smart Nose uses a mass spectrometer and its data was easier to obtain and was more reproducible. In a similar study, Lauf and Hoffheins [31] illustrated that a selected array of chemical sensors can produce unique signatures for many aviation and automotive fuels. Patterns for aviation fuel are readily identified by visual inspection. The differences among automotive fuels with different octane ratings are subtle but perceptible. Gasohol mixtures have strikingly different signatures from pure gasoline. The results indicate that an e-nose can distinguish between various classes of petroleum-based fuels.

Automotive ventilation may also be monitored and controlled by an e-nose. Menzel and Goschnick [32] investigated methods for improving the time response of an e-nose instrument intended for on-line discrimination applications. Their method combined the classification of the steady-state and transient response via time-series analysis. Rapid signal transients were detected by appropriate digital filters, while steady-state signals were classified by standard statistical methods. To illustrate the method, they investigated automatic control of the ventilation flap of an automobile. Steams of bad air were detected in one to two seconds. The error in the detection of pollutants was reduced from the original 25% to only 10% for their new method.

E-nose systems have also been studied for detection of hazardous materials and gases. For example, Hopkins and Lewis [33] investigated the use of arrays of carbon-black/organic-polymer composite chemiresistive vapor detectors for detecting nerve agents. Chapter 28 of this handbook is devoted to the detection of explosives.

Odorous emission from animal production facilities has been extensively studied over the last few years. We present several case studies in this area later in the chapter. Other research groups have also studied this important problem. Hobbs et al. [20] correlated e-nose measurements of pig manure odors to those of a human panel. Four of the principle odorous compounds in pig manure were selected for the study. Thirty-one different mixtures of hydrogen sulfide, 4-methyl phenol, ammonia and acetic acid were used to simulate the livestock waste odor. A radial-basis-function neural network was used for signal processing. Predictions using a linear regression model were on average 20% less than observed values. The authors reported that this approach using the four main odorants is appropriate for determining the odor concentration of pig manure.

An e-nose can frequently be employed to identify specific VOCs and mixtures of VOCs. Hudon et al. [34] compared the effectiveness of three different e-nose instruments in measuring the odor intensity of n-butanol, CH₃COCH₃, and C₂H₅SH, and binary mixtures of n-butanol and CH₃COCH₃. Two commercial e-nose systems, the AromaScan A32S (conducting-polymer sensors) and the Alpha M.O.S. Fox 3000 (metal-oxide sensors), and an experimental unit with Taguchi-type tin-oxide sensors were employed. The e-nose measurements were processed using linear regression analysis and neural networks. Very strong correlation ($\rho = .99$) was obtained between the sensory data and the two commercial units when using neural network analysis. In a related study, Negri and Reich [35] have used an e-nose with commercially available tin-oxide sensors to analyze a mixture of gases containing carbon monoxide, ethanol, methane and/or isobutane. They modeled the theoretical response function of the array and designed a pattern recognition scheme for the simultaneous identification of a given gas and its concentration in the mixture.

The growth of bacteria and fungi on organic matter generates a broad range of volatile organic compounds and fixed gases. Wessén and Schoeps [36] and Sunesson et al. [37] showed that the presence of certain volatile organic compounds can be used as an indicator of the presence and identity of microorganisms. Holmberg [38], in a dissertation at Linköping University in Sweden, used an e-nose with 15 sensors to classify five types of bacteria (*Escherichia coli*, *Enterococci* sp., *Proteus mirabilis*, *Pseudomonas aeruginosa*, and *Staphylococcus saprophyticus*). The 15 sensors included nine MOSFETs, four Taguchi type devices, one carbon dioxide sensor, and one oxygen monitor. The volatile compounds generated by the bacteria were sampled from agar plates. The results suggested that this e-nose could

successfully classify *Escherichia coli* and *Enterococci* sp. but was less successful with the other bacteria.

Gardner et al. [39] used an e-nose that contained six commercial metal-oxide sensors, a temperature sensor, and a humidity sensor to predict the class and growth phase of two types of bacteria, *Escherichia coli* and *Staphylococcus aureus*. The six sensors were designed to detect hydrocarbons, alcohols, aldehydes/heteroatoms, polar molecules, and nonpolar compounds. The best mathematical model correctly identified 100% of the unknown *S. aureus* samples and 92% of the unknown *E. coli* samples.

Other studies have also found that bacteria can be discriminated using an e-nose. In an evaluation of seven bacterial strains, Vernat-Rossi et al. [40] were able to correctly discriminate 98% of a training set with a cross-validation estimate (test set) of 86% using six semiconductor gas sensors. Studies at AromaScan PLC [unpublished data from Dr. Krishna Persaud] showed that polymer sensors performed well in discriminating multiple samples of five different types of bacteria.

Keshri et al. [41] used an e-nose consisting of 14 polymer sensors to classify six spoilage fungi (four *Eurotium* sp., a *Penicillium* sp. and a *Wallemia* sp.). The headspace was sampled after 24, 48, and 72 hours of growth. The e-nose discriminated the fungi at the 24-hour mark (prior to the visible signs) with an accuracy of 93%. The best results occurred at the 72-hour mark.

Measuring air quality by an e-nose requires a hand-held unit. Several commercial instruments are available as described in Chapters 9 and 12. Nicolas et al. [42] have also developed a portable prototype e-nose based on tin oxide sensors for field applications; that is, generate a warning signal when the malodor level exceeds some given threshold value, identify

the source of an odor detected on site, or identify on-line and monitor levels of an odor in the field. The algorithms used in the instrument are described.

As outlined above, the field of environmental monitoring is very broad. In this chapter, we will focus on case studies in livestock odors and microbial contamination.

2. Special Considerations for Environmental Monitoring

2.1. Sample handling problems

2.1.1. Sample Lifetime. If not properly handled (e.g., long exposure to sunlight), some organic samples may disintegrate or undergo certain chemical reactions. Therefore, considerable effort is required in order to maintain samples in their original state prior to their delivery to the sniffing device.

2.1.2. Humidity. As will be discussed later, it is important that the various odor samples have similar humidity levels. The humidity of the reference sample should also be adjusted to that of the odor samples. This is to ensure minimal response due to humidity when switching from reference to odor inputs. A closed-loop humidity control system for the reference input is offered in some commercial systems for this reason.

2.1.3. Extraction of volatiles. In cases in which the number of volatile molecules is low, one may be required to boost these numbers via some preconcentration, activation, or agitation method. In order to record a meaningful sensor response, the concentration of volatiles in the sample must be above a minimum threshold. Certain agitation methods may be necessary for liquid samples in order to increase the concentration of volatiles in the headspace. Conversely, in

the case of highly volatile molecules (e.g., alcohols), one may need to dilute samples in order to avoid sensor saturation. Chapter 5 covers preconcentration methods.

2.1.4. Tubing system. The acquisition system is generally equipped with a tubing system that delivers volatile compounds from the sample container to the sensor compartment, and then to the exhaust outlet. The material used in the tubing must be inert to the type of odorants that the device handles. In other words, the tubing material should not modify or adsorb the odor of the samples. Similar requirements exist for the sensor compartment, valves, etc.

2.1.5. Temperature. The temperature of the sample, sensor chamber, and sensors must be kept constant to achieve repeatable performance of the e-nose system. A temperature perturbation can cause shift/deformation in the generated patterns, by virtue of changes in concentration or sensor behavior. A constant temperature is usually maintained using a feedback control system. Temperature control is important for all types of sensors.

2.2. Signal processing challenges

In addition to appropriate sample handling, signal-processing algorithms are required to compensate for the variability of conditions in the field. By including temperature and humidity sensors in the e-nose instrument, it may be possible to compensate for these effects by means of signal processing algorithms. Sensor baseline drift and unwanted concentration effects may also be handled by means of preprocessing algorithms (see Chapter 7).

Due to the large number of sensors and features (e.g. dynamic response recordings), the e-nose is subject to “the curse of dimensionality.” A large number of dimensions can hinder the true (and useful) information, so the use of dimensionality reduction procedures (e.g. feature selection, principal components) is oftentimes required. These signal processing procedures

must be carefully chosen to ensure that memory and CPU requirements do not become prohibitive for an economical (e.g., hand held) device [43].

3. Case Study 1: Livestock Odor Classification [44]

3.1. Background

Livestock industries are expanding rapidly throughout the world, and this expansion is causing environmental concerns. Modern methods of confining thousands of animals in a single facility have led to increased production and profits while creating concerns about odor and water pollution. Odors associated with livestock operations are generated from a mixture of urine, fresh and decomposing feces, and spilled feed. In swine operations, for example, odors emanate from the ventilation air of confinement buildings, waste storage and handling systems including lagoons and field applications of waste. Anaerobic microbial decomposition of livestock waste appears to be the source of the most objectionable smells. Odorous compounds identified in livestock wastes include sulfides, disulfides, volatile organic acids, alcohols, aldehydes, amines, fixed gases, nitrogen heterocycles, mercaptans, carbonyls, and esters. Reduction of odors emanating from livestock operations is necessary to improve the relationship between producers and their neighbors.

Sensitive measurement techniques are important to characterize and document swine odors, as well as evaluate the effectiveness of methods for reducing odors. At present, olfactometry using human odor panels is the most precise approach for quantifying odors, since the human nose can detect compounds at concentrations that cannot be detected by any other method. Human evaluations, however, can be time-consuming, unrepeatable and expensive. In

addition, odor samples degrade rapidly, and thus human panels must perform evaluations shortly after collection for accurate assessment. Since swine odor abatement research is being conducted all around the world on a 24-hour basis, odor testing with human panels is often impractical. Rapid, accurate, cost-effective evaluation of techniques to reduce odor production (such as the manipulation of pig diets to reduce excrement odor) is vitally important to the swine industry. For this reason it would be helpful to determine if an e-nose with conducting-polymer or metal-oxide sensors can substitute for human odor panels in evaluating methods for odor reduction.

3.2. Description of the problem

The objective of the following study was classification of various odorant samples related to a hog farm. The main task was to gauge the accuracy and the precision of an e-nose in identifying the source of unknown odor samples.

3.3. Methods

Odor samples were collected from three locations at a rural hog farm: lagoon, fan, and downwind ambient air. The samples were presented to an e-nose, and signal-processing algorithms were used to classify the data. A cross-validation method was employed to measure the performance of the system. At each step of this cross-validation method, 70% of the data was used to train the system, while the other 30% was used as an unknown sample set. The e-nose used for the experiments of this section was the AromaScan A32S (see Chapter 9). The core of the AromaScan system is an array of 32 conducting-polymer sensors. Depending on the mode of operation, the sensor compartment is exposed to one of the odorant sample, the reference gas, or

the cleansing gas. The reference gas was generated by filtering, dehydrating, and humidifying steps. The humidity of the reference air was set to match that of the odor samples. The cleansing gas (2% n-butanol bubbler) was used to remove (detach) odorants from the sensors after each data acquisition cycle.

Various air-samples from two lagoons, a confinement building exhaust fan, and a downwind site at a hog farm in rural North Carolina were collected using 25-liter Tedlar[®] bags. The downwind-air sample was collected 1,500 feet from the swine operation. These bags were filled using a pump device and sealed barrel under negative pressure. The bags were cleaned using a combination of butanol, methanol, nitrogen, and/or dry air, and reused. The most commonly used cleaning technique was flushing with nitrogen, then a methanol vapor, followed by clean dry air.

A major drawback of this sampling method is the shipping and handling of the filled bags. Since the odors degrade over time, the samples should be processed the same day during which they are collected. Hence, this technique is adequate for sites that are located in close proximity (within 150 miles) to the testing facilities. We have found that holding the bags overnight for processing the following day significantly reduces the odor intensity, and hence the reliability of the sample collection method.

3.4. Signal processing algorithms

The datasets obtained from the electronic nose was analyzed using a set of algorithms listed below. More detailed explanations of the various algorithms can be found in Chapters 7 and 8. The main steps of signal processing in this case study are outlined as follows:

3.4.1. Bias Removal. One of the drawbacks of polymer sensors is their inability to return (within a reasonable time frame) to the baseline after washing. The residual signal will result in a gradual shift in the successive data acquisition cycles. The first step of preprocessing was to remove mathematically the bias. In these experiments, the bias was removed by subtracting the response of each sensor at the first time point from all the other subsequent time points in the dynamic response of that sensor.

3.4.2. Humidity. Another major weakness of some conducting-polymer sensors is their high sensitivity to water molecules. If not controlled, the common-mode response that is caused by humidity could completely overshadow the signal of the odorants. Various approaches have been proposed to counteract humidity and its effects. One is to model the response of the sensors to humidity, and then to subtract it from the composite response. However, due to the low repeatability of the patterns, this was not found to be a suitable approach for the AromaScan A32S polymer sensors. Another approach is to employ the humidity control features of the AromaScan A32S that allow the operator to adjust the humidity of the reference signal to that of the odor sample. We should point out that researchers in this field are developing new types of conducting-polymer sensors that are much less sensitive to changes in sample humidity.

3.4.3. Concentration. One obvious challenge in sample preparation is the control of the volatile concentration. Within certain ranges, the effect of concentration has been shown to be linear. When comparing samples of the same kind, one must be able to either normalize the effect of concentration, or guarantee that samples contain similar concentrations of the odorant of interest. In the experiments of this study, the response of each sensor at each time point was divided by the average response of all sensors at that time point. When the sensors operate in the

linear range, this method has been shown to normalize the response of the sensors with respect to the concentration [44].

3.4.4. Dimensionality Reduction. In the following experiments, every sample produces $30 \times 32 = 960$ data points. Since a single training session may include several dozens of samples, it is evident that the dimensionality could become overwhelming for this problem. Therefore, in lieu of supplying the time series data directly into the processing unit, a reduced set of features was extracted prior to the main analysis.

Data reduction was done in two stages. In the first stage, a series of bell-shaped curves were used to serve as windowing functions. By using windowing functions, the set of 30 time points of the response of each sensor was reduced to four, the number of windowing functions. The next step of data compression was done by Karhunen-Loève (Truncated) Expansion (KLE), a.k.a., Principal Components Analysis (PCA). KLE is known to be the optimal linear method for data compression [45]. Using KLE, a series of features, i.e., the significant eigenvectors, was extracted from the time-windowed traces of each sample. The dimension of the transformed signal was found dynamically by analyzing the relationship between the eigenvalues of the covariance matrix [44].

The set of features extracted from the KLE compression was then directed into an MLP neural network for training and testing. The learning rule of the neural network was based on the Levenberg-Marquardt method [46, 47]. The back-propagation method [48] (with a momentum term and adaptive learning rates) was also used for comparison purposes.

A genetic-algorithm-based supervisor was designed to tune the number of neurons in the hidden layer and the learning parameters of the neural network. The genetic algorithm (GA) was also responsible for choosing all or a subset of the windowed values and/or features.

3.5. Results

The results are depicted in Figure 1. Aside from the difficulties of sample handling, the results appear to be reasonable. The figure shows the histogram of the performance of 100 cross-validated runs. The y-axis is the number of runs and the x-axis is the correct recognition in percent. Note that 97 of the runs gave a perfect 100% correct recognition, while the remaining three cases were 97% correct. The overall correction recognition rate is 99.92%.

3.6. Discussion

Several alternative signal processing methods, e.g., neural networks with back-propagation, with and without the GA supervision, were tried prior to applying the above-mentioned methods. These alternative methods were found to achieve lower performance metrics. The preprocessing steps were found to be necessary for generating repeatable histogram patterns. A neural-network-based classifier with the Levenberg-Marquardt learning rule was found to be appropriate for this particular pattern recognition application. Using genetic algorithms as a supervisor provided a systematic, reliable and automated method for feature selection and architectural tuning of the neural network.

The final hybrid GA-NN system proved to serve as an effective signal processing technique for this application. However, regardless of the efficacy of the signal processing method, the quality of the final outcome is a function of the quality of the input data. In general, due to their limited sensitivity, conducting-polymer sensors were found to be more suitable for odor samples containing high concentrations of highly volatile molecules such as those found in fragrances.

4. Case Study 2: Swine Odor Detection Thresholds

4.1. Description of the Problem

The detection threshold for a specific odorant mixture is related in part to the detection thresholds of its individual components. In this study, we select one of the odorous components of hog slurry, acetic acid, and compare the detection thresholds of a human panel and the AromaScan A32S for this compound.

4.2. Methods

Detection threshold concentrations were determined for acetic acid, a major individual constituent in swine odor. In this experiment, twelve serial dilutions of acetic acid that differed by a factor of three and ranged from 5% to 0.0000094% v/v were presented to the human panel at the Taste and Smell Lab at Duke University Medical Center and the AromaScan A32S for evaluation. Both distilled water and odorless mineral oil were used as diluents. The e-nose signals were processed using the same procedure as Case Study 1 above [44, 49]. The employed techniques consisted of a preprocessing stage and a data-compression stage. The preprocessing stage involved shifting each sensor's curve, so that the initial resistance change was initialized to zero. The data compression stage consisted of two steps: windowed time integration and Karhunen-Loève expansion (KLE). The windowed time integration multiplied each sensor curve by four bell-shaped kernels and then computed the area beneath the curves. In this way, each odor sample was reduced from 32 x 45 (sensors x seconds) to 32 x 4 (sensors x windows) features. Then the KLE was performed to extract the principal components in feature space.

4.3. Results

The dilution labels ranged from 13 to 1, for the highest and lowest concentrations, respectively. Due to the cross-sensitivity of the conducting-polymer sensors to water, we decided to determine thresholds for acetic acid for the human panel only for the mineral oil serial dilutions. The resultant two-dimensional KLE scatter plot for the acetic acid dilutions in mineral oil is presented in Figure 2. Note that a detection threshold between labels 9 and 10 can be visually determined.

4.4. Discussion

Our results indicate that the e-nose has a detection threshold that is a factor of three above the detection threshold of the human panel. The detection thresholds for the four human subjects were at Dilutions 9, 8, 8 and 9, whereas the e-nose is between Dilution 9 and 10, as shown in the figure. Since Dilution 10 has an odorant concentration that is three times greater than Dilution 9, and Dilution 9 has an odorant concentration that is three times greater than Dilution 8, on average the human panel's detection level is at a concentration that is three times lower than that of the e-nose. A factor of three in odorant concentration gives the human panel an advantage over the e-nose in this application. However, the e-nose can be deployed on site and can measure emissions over long time periods, characteristics of a monitoring system that are not practical for human panel implementation. Hence we conclude that the e-nose can compete with a human panel in detecting odorous emissions from swine facilities.

5. Case Study 3: Biofilter Evaluation [50]

5.1. Description of the Problem

The objective of this study was two-fold. First, to develop an experimental procedure to evaluate biofilters for odor remediation in the ventilation exhaust fans of hog confinement buildings. Second, to determine if the AromaScan A32S could be utilized to predict the human panel olfactory ratings of malodors before and after bioremediation.

5.2. Methods

In order to rapidly screen the performance of various odor remediation materials, a bench-top biofilter setup was developed at NC State. The biofilter material consisted of earth, wood chips, small twigs and straw. This material was placed in a one-inch diameter PVC tube, which was cut to a length of 3.9 inches. This length was selected because of the requirement to have the air reside within the filter for 15 seconds. A 15-second residence time matches the specifications of field units at the NC State Animal and Poultry Waste Management Center. The tube was cemented on each end to a PVC fitting which had screw threads and an O-ring to produce an airtight seal with the connecting piece. Wire mesh was placed on each end of the cemented tube fitting to prevent the biofilter material from spilling out of the tube.

To test this biofilter setup, we conducted an odor remediation experiment with a synthetic slurry following the concoction of Persaud et al. [51]. Serial dilutions (1/1, 1/3, 1/9, 1/27 and 1/81) of the headspace above the slurry, as well as serial dilutions of the biofiltered synthetic slurry and biofiltered blank room air (as a control) were presented to both the Duke human panel and the e-nose. The experimental setup is depicted in Figure 3.

To measure the human perception to the different odors and dilutions, the panelists were asked to generate scores for intensity, irritation, and pleasantness using the 9-point scale shown

in Table 1. The e-nose signals were preprocessed by computing the fractional change in resistance of each sensor with respect to its baseline resistance in reference air (steady-state $\Delta R/R$). The steady-state response of each sensor was extracted to form a 32-dimensional feature vector.

5.3. Results

The average response of the human panel and the 32 conducting-polymer sensors in the e-nose for each of the 15 dilutions (five dilutions for each of three odor sources) is shown in Figure 4. Note that for the human panel, biofiltering reduced the intensity, irritation, and unpleasantness of the odor. In addition, the panel's ratings of the biofiltered slurry and blank air were quite similar.

In order to establish whether the e-nose could be used to replace a human panel in odor-remediation scenarios, we performed Partial Least Squares (PLS) regression [52] to predict the average response of the human panel from the 32-dimensional average response of the e-nose. To establish the predictive accuracy of this model, we performed cross-validation in which one of the fifteen dilutions was removed from the training data and predicted only after the PLS model had been obtained. Figure 5 shows the performance of the model on test data for the fifteen leave-one-out validation runs. The correlation coefficient ρ between predictions and true values on test data for intensity, irritation and pleasantness are 0.90, 0.94 and 0.86, respectively.

Given the notorious cross-sensitivity of conducting polymers to moisture, we decided to analyze the response of the built-in humidity sensor of the AromaScan A32S to the different odors and dilution ratios. The transient response of odor and humidity sensors to the fifteen samples is shown in Figure 6. Two observations can be made. First, looking at the humidity

sensor response to the slurry before and after biofiltration, it can be concluded that the biofilter material is increasing the relative humidity of the samples. Second, as a result of serial dilutions, the humidity of the samples is significantly reduced.

Based on these results, it is necessary to determine if humidity is dominating the e-nose response. A closer look at the data shows one that the response of the sensor array to the synthetic slurry has a unique dynamic signature that is different from the exponential decay to the biofiltered samples. This indicates that, in spite of relative humidity changes, the odor sensors are able to detect the synthetic slurry. In addition, if the odor sensors were responding only to the humidity, the largest response of the sensor array would then occur with the 1/1 biofiltered blank since this sample has the highest response on the humidity sensor.

To further rule out the possibility that the e-nose is just detecting differences in moisture, we attempted to predict the human olfactory ratings from the humidity sensor response alone. The results are summarized in Figure 7. The correlation coefficients between these single sensor predictions and true values by the human panel on test data for intensity, irritation and pleasantness drop down to 0.40, 0.31 and 0.29, respectively. Hence, the conducting-polymer sensor array is giving much better performance, proving that the response of the odor sensors contains information related to the presence of synthetic slurry.

5.4. Discussion

The main findings of this study are that the AromaScan® A32S can differentiate between different dilutions of the components of swine odor, and between synthetic slurry and biofiltered slurry/blank samples. The sensor array response can be used to predict the intensity and pleasantness olfactory ratings from a human panel. Moisture is shown to be a major interferent

since biofiltration increases the relative humidity of the samples. However, the signal processing routines were able to mediate this interference. In the future, this interference might be reduced further by performing serial dilutions with a carrier gas having the same relative humidity as the odor samples.

6. Case Study 4: Mold Detection [53]

6.1. Background

Microbial contamination of our environment is an area of increasing concern. An e-nose has the potential to identify and classify microorganisms, including bacteria and fungi. When conditions are favorable and a nutrition source is present, microbial organisms such as fungi and bacteria can grow almost anywhere. Microorganisms have been shown to generate volatile organic (VOCs) while metabolizing nutrients, and these VOCs have been used as indicators of microbial growth. Colonies of microorganisms not only generate airborne contamination in the form of VOCs, but also generate toxins, conidia (spores), and bacterial cells.

When microorganisms infest buildings, they can produce a potentially hazardous environment. Individuals exposed to environments that contain high concentrations of airborne contaminants from microbial organisms report health symptoms including eye and sinus irritation, headaches, nausea, fatigue, congestion, sore throat, and even toxic poisoning. Sick Building Syndrome (SBS), which includes health symptoms arising from poor indoor air quality, has been correlated with the presence of fungi [54]. A study of two households reporting indoor environmental complaints correlated the presence of excessive VOC's with the presence of fungal contamination [55]. Typical signs of microbial contamination include water damage, high

levels of humidity, and physical presence. However, these signs are not always present, and therefore cannot be utilized as sole indicators of microbial contamination.

Current methods for detecting microbial contamination include air and material sampling with culture analysis, air sampling coupled with gas chromatography/mass spectrometry, and visual inspection [56, 57]. These methods, however, can be inconclusive as well as time consuming and expensive. Thus, rapid detection of the presence of microbial contamination is needed in order to minimize its impact.

6.2. Description of the Problem

In this study, we explored the ability of the NC State E-Nose, a prototype electronic system with 15 metal-oxide sensors, to detect fungi at various stages of growth. Fungi that are typically found in indoor air-conditioning systems were chosen for experimentation. The purpose of the experiment was to demonstrate that an e-nose system is capable of diagnosing the presence of these fungal types in commercial buildings and residential housing units.

6.3. The NC State E-Nose

An e-nose instrument was designed and constructed at North Carolina State University [44, 49] that uses an array of metal-oxide sensors for measuring odor in air samples (see Figure 8). The e-nose consists of a sampling unit, a sensor array, and a signal processing system. The sampling unit, which consists of a pump and a mass-flow controller, directs the air sample containing the odorant under investigation across the sensor array. The current configuration allows for sampling from a set of 12 odorants, a reference sample (filtered odorless dry ambient air), and a washing agent (ambient air bubbled through a 2% n-butanol solution). The tubing and

sensor chamber are made of stainless steel. The sensor chamber is designed to minimize dead volume (see Figure 9). The sensor array is composed of 15 different metal-oxide sensors. Twelve of the 15 metal-oxide sensors are manufactured by Capteur (Didcot, UK) and include sensors for isopropyl alcohol, toluene, hydrogen sulfide, nitrogen dioxide, chlorine, butane, propane, hydrogen, carbon monoxide, heptane, ozone and general VOCs. The remaining three metal-oxide sensors are produced by Figaro USA (Glenview, IL) and include a methane, a combustible gas, and a general air contaminant sensor. All of the sensor response patterns are digitized and recorded using a National Instruments® Data Acquisition Card controlled by LabVIEW®.

The operation cycle for the NC State E-Nose consists of three phases: wash, reference, and sample. The solenoid valves are normally closed. Solenoid valve s_1 (exhaust) and an appropriate inlet solenoid valve (s_2 to s_{15}) are opened at the beginning of each phase and closed afterwards. The mass flow controller must also be set at the beginning of each cycle to the appropriate set point (between 0.0 and 1.0 liters per minute). A brief explanation of each phase follows:

Wash: solenoid valves s_1 and s_2 are opened. Room air is passed through a charcoal filter (to remove residual ambient odors) and a bubbler with 2% diluted n-butanol in distilled water. The resulting gas is used to flush tubing and sensors and remove traces of odorants from previous gas samples.

Reference: solenoid valves s_1 and s_3 are opened. Room air is passed through a charcoal filter (to remove residual odors) and a moisture trap. The resulting odor-free dry air is used as a reference gas to force the sensor resistances back to their baseline values.

Sample: solenoid valve s_1 and one other valve (s_4 to s_{15}) are opened. The odorous sample is passed through the e-nose. Return to *Wash*.

6.4. Methods

Five fungi (*Aspergillus flavus*, *Aspergillus niger*, *Penicillium chrysogenum*, *Cladosporium cladosporioides*, and *Stachybotrys chartarum*) were incubated at 28°C on 150 mm Petri dishes containing Potato Dextrous Agar (PDA), a complex media rich in nutrients, and Czapek-Dox Agar (CZ), a minimal media. These two types of media were used in order to provide two different growth environments and to produce different growth rates. Twenty four Petri dishes of each media were inoculated with 0.5 ml of an individual spore suspension containing 10,000 conidia/ml from each fungus, respectively. The suspensions were prepared using a Spencer hemacytometer with improved Neubauer ruling. Using the autosampler functions of the NC State E-Nose, air samples from the headspace of each Petri dish containing one species on each medium were randomly sampled ten times each after 24 hours and every other day thereafter for two weeks. The headspace above each fungus was sampled through a small hole in the center of the lid of the Petri dish using a PVC tube and an inline two micron filter for removing conidia (spores).

The data were analyzed with MATLAB® using signal processing algorithms developed by Kermani [44] and Gutierrez-Osuna [49]. More specifically, the raw data were first compressed using windowing functions that produced a set of four features for each sensor. Linear discriminant analysis (LDA) was then applied to the compressed data to maximize class separability. Sixty percent of the compressed data was randomly selected to form a training set for the classification algorithms. K-nearest-neighbors (KNN) and least-squares (LS) techniques were both employed to classify the remaining 40% of the compressed data [58]. This process was repeated 100 times, and the average score was used as the final classification score.

6.5. Results

The data were analyzed using two classification protocols. In the first protocol, the data were grouped into 12 classes: five fungal species grown on PDA and CZ respectively plus two controls (the two media PDA and CZ without fungal growth). The results are shown in Table 2. After 24 hours of growth, the percent classification was 90% for KNN, and 76% for LS. Classification for the 12 classes reached a maximum after five days of growth, with an accuracy of 96% for KNN and 94% for LS. After day 5, the percent classification began to decrease slowly. By day 15, the percent classification was reduced to 89% for KNN and 69% for LS.

In the second classification protocol, the data were grouped into seven classes: five fungal species (independent of media used for growth) plus two controls (the two media PDA and CZ without fungal growth). In other words, each of the fungi grown in PDA and CZ were combined into a single class. After 24 hours of growth, the percent classification was 89% for KNN, and 79% for LS. Classification reached a maximum after five days of growth, with an accuracy of 94% for KNN and 93% for LS. After day 5, the percent classification oscillated around an average percent classification of 92% with a standard deviation of 2%. The results are shown in Table 3.

6.6. Discussion

This experiment with five fungi showed that the NC State E-Nose using metal-oxide sensors can detect and classify microorganisms on the basis of volatile emissions. The classification was independent of the media used to grow the fungi. Furthermore, correct classification was achieved early in the experiment at 24 hours of growth. Thus e-nose

instruments of this type have the potential to be used for early detection of microbial contamination in office buildings and manufacturing facilities.

7. Future Directions

The success of laboratory instruments in classifying environmental odors has been demonstrated by many research groups around the world. This success must now be leveraged to build new portable instruments for field use. These portable units must operate in real time, recording odor concentration profiles at specific time intervals tailored to individual environmental monitoring applications. These devices must be able to detect odors at very low (ppb) levels. Hence, more sensitive gas sensors and preconcentration units must be included in instruments that will be used in onsite, real-time environmental measurements. Chapters 9 and 12 have illustrated some progress by the instrument makers towards reaching these goals. Improvements in signal processing algorithms can offer some assistance. Low-power, embedded microprocessors are continually being improved by the electronics industry. Incorporating more powerful real-time data processing algorithms onboard these portable instruments will differentiate the different commercial models. If the e-nose manufacturers can “break” into the environmental monitoring market in a significant way, the future of this technology will be guaranteed.

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Table 1. Hedonic tone odor rating scales.

Scale	Odor Intensity	Irritation Intensity	Pleasantness
8	Maximal	Maximal	Extremely Unpleasant
7	Very Strong	Very Strong	Very Unpleasant
6	Strong	Strong	Moderately Unpleasant
5	Moderately Strong	Moderately Strong	Slightly Unpleasant
4	Moderate	Moderate	Neutral
3	Moderately Weak	Moderately Weak	Slightly Pleasant
2	Weak	Weak	Moderately Pleasant
1	Very Weak	Very Weak	Very Pleasant
0	None at all	None at all	Extremely Pleasant

Table 2. Percent classification for 12 classes (five fungal species on two different media and two control media) [53]

Classification Method	Day of Growth							
	1	3	5	7	9	11	13	15
KNN	90%	91%	96%	94%	89%	93%	93%	89%
LS	76%	90%	94%	90%	93%	86%	80%	69%

Table 3. Percent classification of seven classes (five fungal species and two control media) [53]

Classification Method	Day of Growth							
	1	3	5	7	9	11	13	15
KNN	89%	90%	94%	93%	89%	94%	94%	92%
LS	79%	88%	93%	91%	95%	90%	92%	86%

Figure Captions

Figure 1. Histogram showing test results of 100 runs of training/testing of hog-farm samples using the hybrid of neural-network and genetic-algorithms in conjunction with the AromaScan A32S. The number of runs is given on the y-axis, and the percent correct recognition is given on the x-axis. On 97 of the runs, there was a perfect 100% correct recognition, while there was 97% correct recognition for the remaining three cases.

Figure 2. Principal Component Analysis for dilutions of acetic acid in mineral oil. The two-dimensional scatter plot shows that a detection threshold occurs between labels 9 and 10.

Figure 3. Experimental setup for malodor biofiltration assessment. Air from the synthetic hog slurry and the room air control is filtered and delivered to the human sensory panel and e-nose (AromaScan A32S) for analysis.

Figure 4. Average human and e-nose response versus dilution number in the biofiltration experiment. The labels on the abscissa for the serial dilutions are defined as follows: 5 (1/1 dilution), 4 (1/3 dilution), 3 (1/9 dilution), 2 (1/27 dilution), and 1 (1/81 dilution). As expected, both human and e-nose (AromaScan A32S) responses decrease with increasing dilution.

Figure 5. True vs. predicted human panel ratings for intensity, irritation, and pleasantness using the odor sensor array based on the performance of the model on test data for the fifteen leave-one-out validation runs. The correlation coefficient ρ between predictions and true values on test data for intensity, irritation and pleasantness are 0.90, 0.94 and 0.86, respectively.

Figure 6. Transient response of the gas sensor array and the humidity sensor to five serial dilutions per odor using the AromaScan A32S. The waveforms in both the upper and lower portions of the figure show the time response of the odor and humidity sensors

for each dilution (labeled in the center of the figure). Note that the humidity sensor response indicates that the biofilter material is increasing the relative humidity of the samples. Also serial dilutions with dry air reduce the humidity of the samples.

Figure 7. True vs. predicted human panel ratings using only the humidity sensor. The correlation coefficients between the true and predicted values are reduced to 0.40, 0.31 and 0.29, respectively, as compared with 0.90, 0.94 and 0.86 in Figure 5. Thus, the conducting-polymer sensor array gives much better performance than humidity alone.

Figure 8. System configuration for the NC State E-Nose. The exhaust pump pulls air samples through the system. The mass flow controller and exhaust pump can be separated from the system by solenoid valve S_1 . The system has 14 sample input ports controlled by solenoid valves S_2 to S_{15} . Ports S_2 and S_3 are assigned the washing (cleaning) and reference functions, respectively. Ports S_4 through S_{15} are designated as odor sample handling inputs. The system includes an inline pressure sensor, a combined temperature/humidity sensor, and 15 metal-oxide odor sensors.

Figure 9. The sensor chamber; (a) airflow pattern; (b) photograph. Commercially available metal-oxide sensors are mounted in a stainless steel chamber. The electrical leads of the sensors are soldered to printed circuit boards with attached ribbon cables that relay the sensor responses to interfacing electronics. From the top of the chamber, air enters a cylindrical tube with holes that “jet” the odor samples directly onto each odor sensor. After passing over the sensors, the air streams merge and exit the chamber.

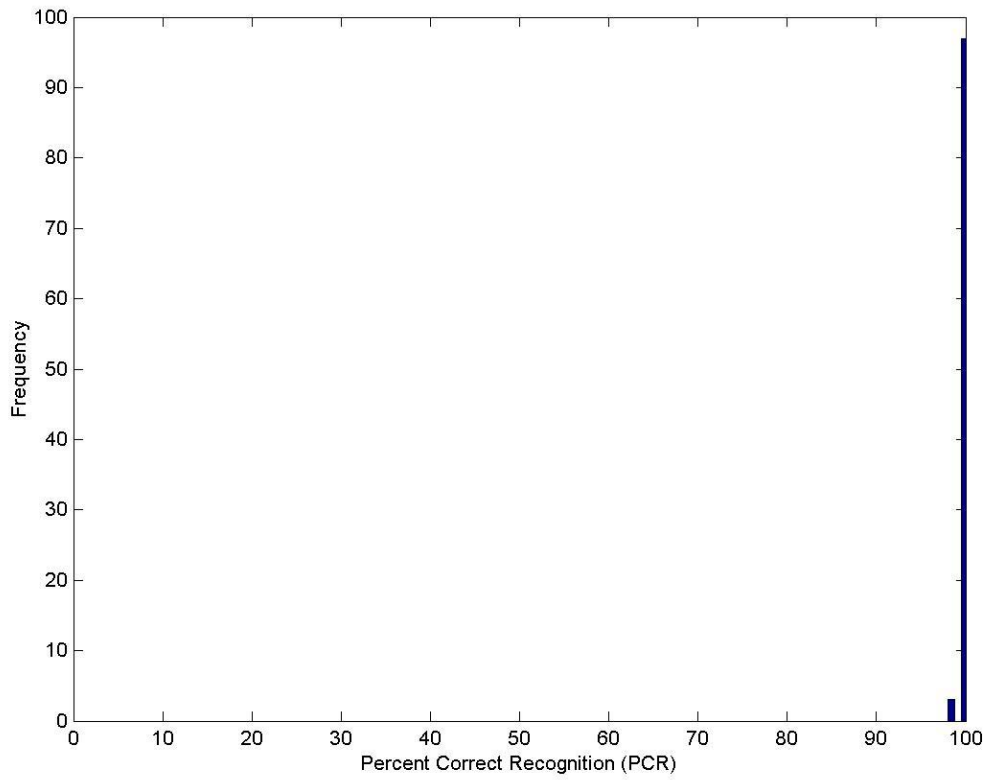


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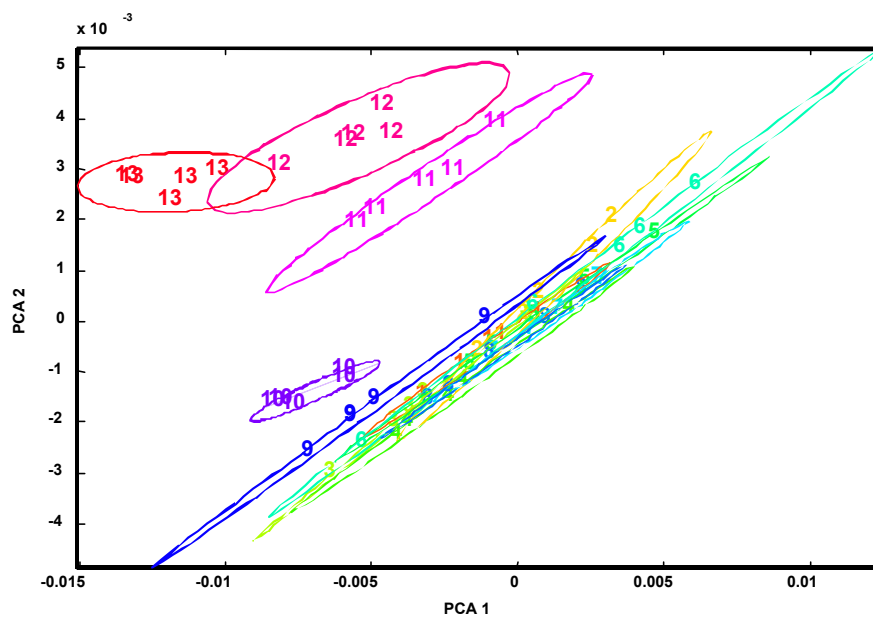


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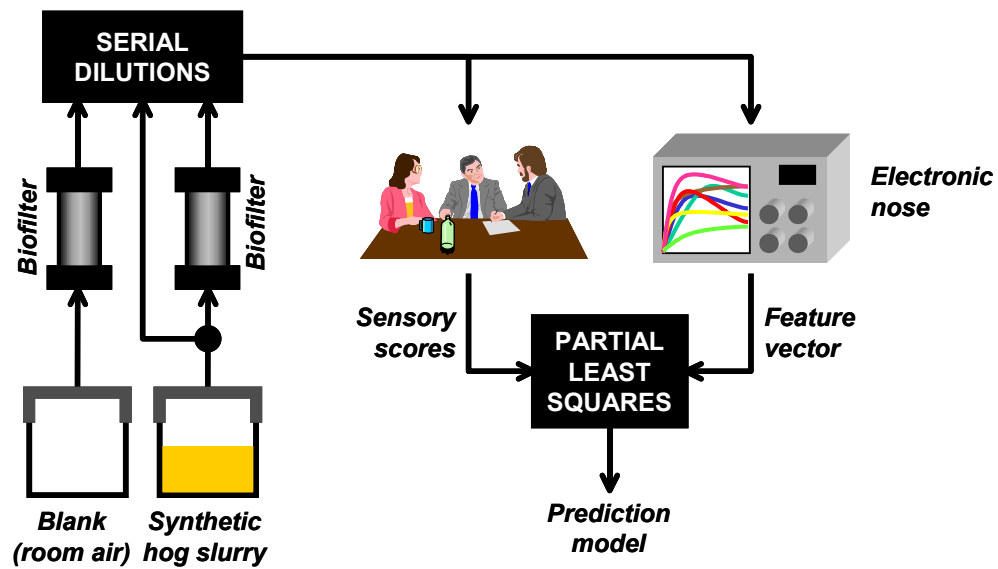


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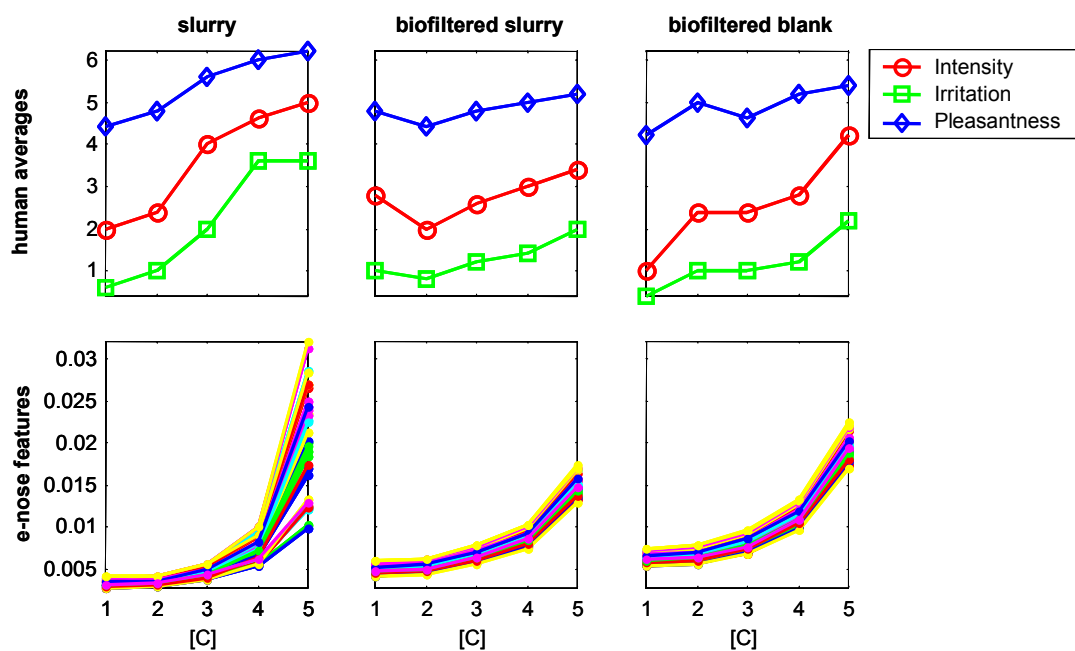


Figure 4. Average human and e-nose response versus dilution number in the biofiltration experiment. The labels on the abscissa for the serial dilutions are defined as follows: 5 (1/1 dilution), 4 (1/3 dilution), 3 (1/9 dilution), 2 (1/27 dilution), and 1 (1/81 dilution). As expected, both human and e-nose (AromaScan A32) responses decrease with increasing dilution.

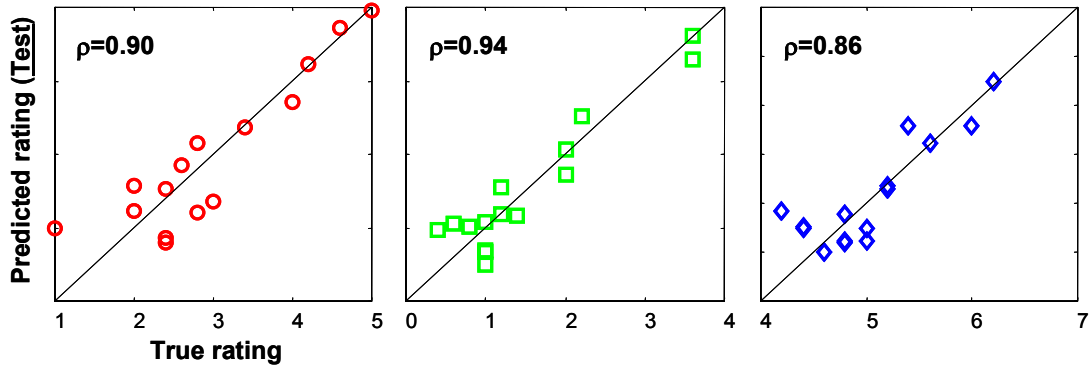


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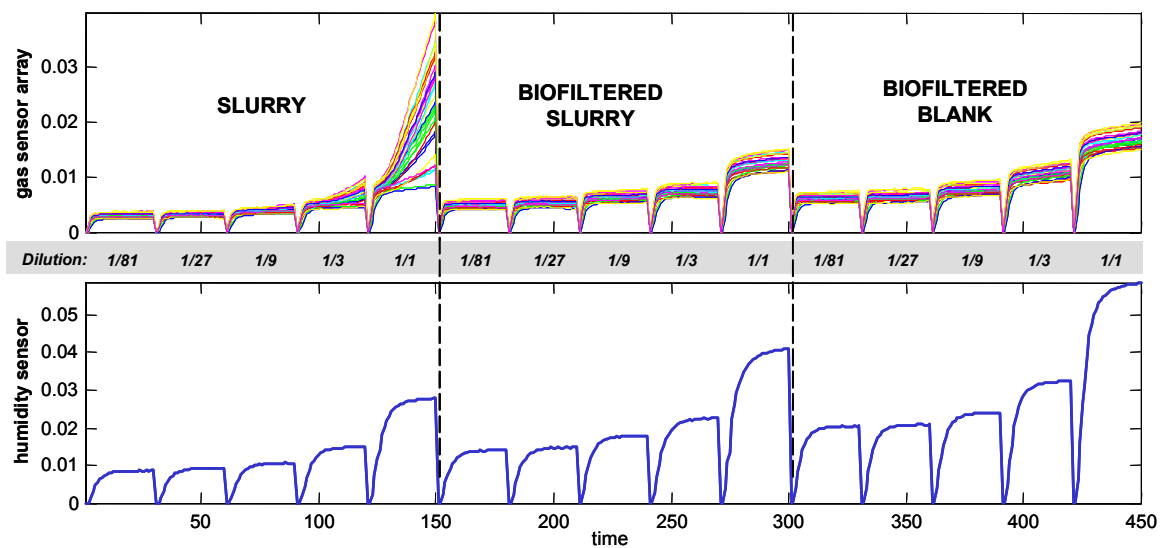


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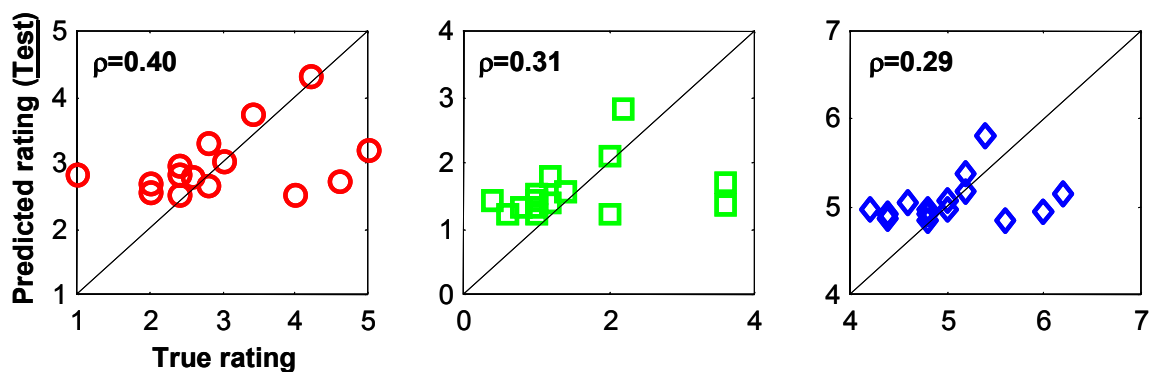


Figure 7. True vs. predicted human panel ratings using only the humidity sensor. The correlation coefficients between the true and predicted values are reduced to 0.40, 0.31 and 0.29, respectively, as compared with 0.90, 0.94 and 0.86 in Figure 5. Thus, the conducting-polymer sensor array gives much better performance than humidity alone.

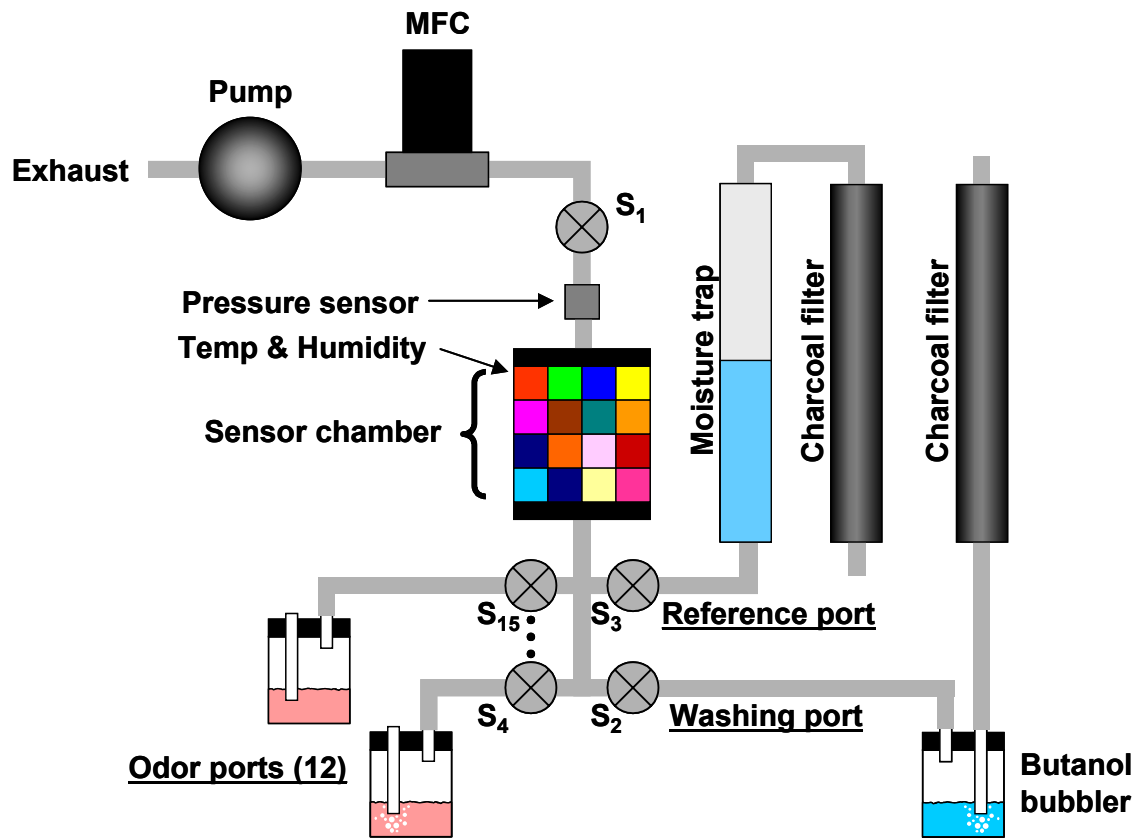
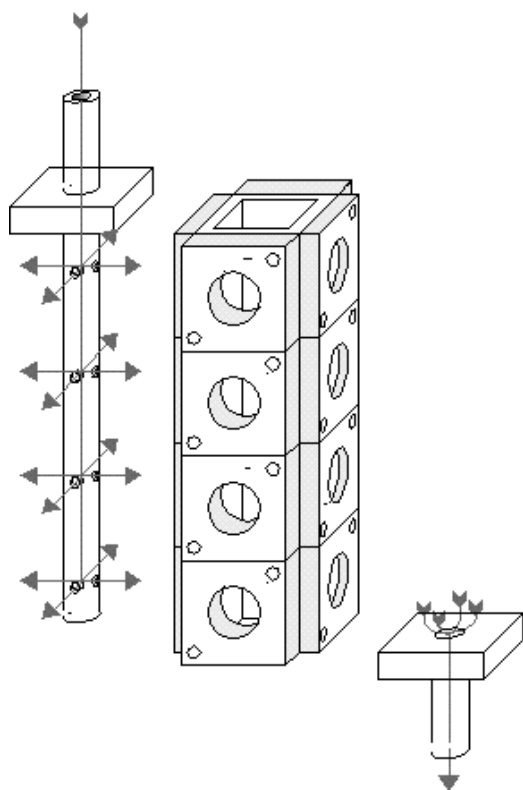
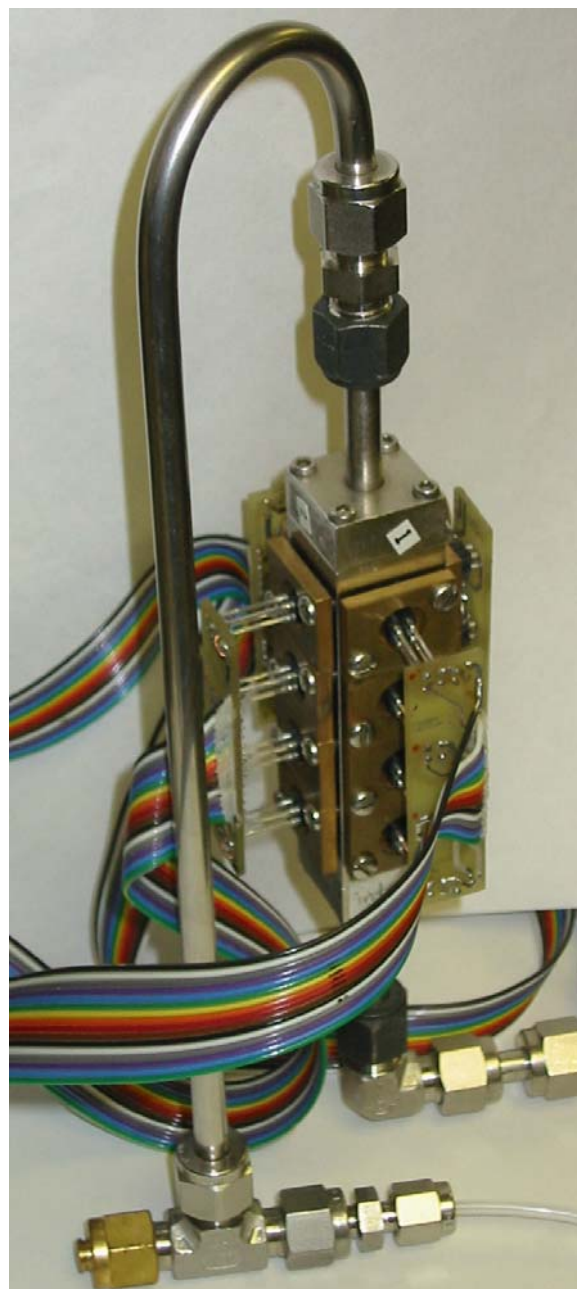


Figure 8. System Configuration for the NC State Electronic Nose. The exhaust pump pulls air samples through the system. The mass flow controller and exhaust pump can be isolated from the system by solenoid valve S₁. The system has 14 sample input ports controlled by solenoid valves S₂ to S₁₅. Ports S₂ and S₃ are assigned the washing (cleaning) and reference functions, respectively. Ports S₄ through S₁₅ are designated as odor sample handling inputs. The system includes an inline pressure sensor, a combined temperature and humidity sensors, and 15 metal-oxide odor sensors.



(a)



(b)

Figure 9. The sensor chamber; (a) airflow pattern; (b) photograph. Commercially available Figaro® and Capteur® metal oxide sensors are mounted into a stainless steel chamber. The electrical leads of the sensors are soldered to printed circuit boards with attached to ribbon cables that relay the sensor responses to the interfacing electronics. From the top of the chamber, air enters a cylindrical tube with holes that jet the odor samples directly onto each odor sensor. After passing over the sensors, the air streams merge and exit the chamber.