Simultaneous Detection of Multiple Environmental Contaminants Through Advanced Signal Processing of Electrochemical Sensor Signals

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Abstract—The possibility of large-scale attacks using chemical warfare agents (CWAs) has exposed the critical need for fundamental research enabling the reliable, unambiguous, and early detection of trace CWAs and toxic industrial chemicals. This paper presents a unique approach for identification and classification of environmental contaminants by perturbing an electrochemical (EC) sensor with an oscillating potential rather than static voltage levels. The dynamic response, being a function of the degree and mechanism of contamination, is then processed with a symbolic dynamic filter for extraction of representative patterns, which are then classified using a trained neural network. Extraction of statistically rich information from the current response enables identification of characteristics species even when they are mixed with other confounding gases. The approach presented in this paper promises to extend sensing power and sensitivity of these EC sensors by augmenting and complementing the sensor technology with state-of-the-art embedded real time signal processing capabilities.

Index Terms—Electrochemical Sensor, Environmental Contaminants, Symbolic dynamics

I. INTRODUCTION

Defense establishments have long considered the possible use of CWAs in the battlefield and thus have continued efforts to develop sensors for the ambient detection of CWAs to protect troops. However, CWA sensors suitable for civilian use in places such as airports, railroad stations, large public and private office buildings, theaters, sports arenas, etc., have received much less attention. For the detection of CWAs in real environments, in addition to high sensitivity, the sensors must have very few false alarms to avoid unacceptable social and economic disruptions and be capable of discovering low concentrations of target gases including other ambient gases.

Several different approaches have been used to detect organophosphorus compounds, including but not limited to potentiometric methods [1], colorimetric methods [2], surface acoustic wave spectroscopy [3], microcantilever piezoresistive sensor [4], gas chromatography/mass spectrometry [5], conducting polymer arrays [6] and interferometry [7].

Each of the techniques to detect CWAs and other gas phase species, discussed above, has drawbacks and limitations. Infrared spectrometers in addition to being expensive, complex and bulky, offer a limited level of detection. Raman spectroscopy that has successfully been employed for CWA detection in the laboratory conditions is not sufficiently robust for identification of the agents in the real-world environment. Colorimetric indicators, commonly referred to as detector paper, are the fastest, cheapest, lightest, and easiest type of detector to use. However, they detect chemical agents in the liquid form only; they also suffer the high cross-sensitivity to smoke, acetone, gasoline and other interferents. Colorimetric tubes are applicable to both vapors and gases and provide a semi-quantitative indication of the amount of agent present in the environment but has a low speed of response to agents and a large size. Ion mobility spectrometers can detect CWA in real-time, and they are portable and sensitive. This technique, however requires a radioactive source, has poor selectivity in the presence of interferents, is costly, and uses instruments that are too large for some applications. Mass spectrometers combined with gas chromatographs (GC-MS) are the most sensitive and reliable of today’s instruments for CWA detection. However, they are bulky, expensive, require sample preparation and need technically trained personnel.

A subset of chemical sensors are electrochemical sensors, which have two principal modes of electroanalytic measurement: potentiometric and amperometric [8]. Potentiometric analysis uses the equilibrium potential of a given electrode to determine the presence of an ionic species. Alternatively, amperometric measurement techniques monitor the current as a function of the applied potential. By varying the applied potential, multiple species can be detected by one sensor, and typically the detection limits are lower than that of potentiometric techniques [9].

For environmental monitoring applications, multi-specie gas detection is desirable in order to detect and appropriately identify airborne contaminants. Thus, amperometric measurement techniques are of utmost interest for this application. However, due to the variability of contaminant concentration, coupled with the presence of multiple confounding contaminant species and the variation of other key variables such as flow rate, temperature and relative humidity, accurate detection and determination can become quickly convoluted and imprecise. For this reason, devices and methods for multi-contaminant detection have gone underdeveloped.

The recent explosion in digital computing capabilities and advances in communication promises the possibility of creating a network of small independent battery-powered sensors with associated instrumentation which will take advantage of embedded real-time signal processing techniques for sensor data analysis. These sensors, when deployed at strategic locations in public buildings, or even critical parts of a city,
will report their low-dimensional processed observations to the central computing system, thus forming the backbone of a centralized decision making system to monitor and alert the population in case of an emergency. Redundancy in the number of such sensors can be utilized to make optimal decisions, and also the time trace of events reported to the central console can be used to trace the origin of the contaminant attack using computational fluid dynamics model of air flow in the region of interest. Such optimistic objectives will be part of future research endeavors, but a clear vision of future application is necessary to develop the current proposed technology.

A recent paper by Bhambare [10] developed the formulation and experimental validation of a CO sensor system for fuel cells based on an anomaly detection technique. The CO sensor was shown to be useful in the fuel cell application where the feed stream is reformate with traces of CO. The response of the sensor to a step change in voltage was used to generate the dynamic current response data that was analyzed using a statistical signal processing technique called Symbolic Dynamic Filtering to generate statistical patterns for clean and contaminated conditions. The difference between these patterns provided an indication of the CO content in the reformate stream.

This paper extends these results to include detection of multiple environmental contaminants using dynamic response from similar electro-chemical sensors. Specifically, the contributions of this paper, beyond that reported in [10] are:

- Use of a new preprocessing technique leading to improvement in sensitivity and resolution of the sensor
- Validation of the pattern generation algorithm and classifier capabilities using multiple contaminants
- Prediction of contaminant mixtures

The technical approach is detailed in the next section.

II. TECHNICAL APPROACH

An electrochemical sensor consists of electrodes (e.g., working electrode, counter electrode, etc.). Gaseous species diffuse to the electrode where the species are either oxidized or reduced. The potential difference between the working electrode and the counter electrode (in a two-electrode system, for example) can be quantified by the Nernst equation [11] and depends on the species present at each electrode. If an external circuit controls the potential across the sensor, the resulting electrical current that is passed through the external circuit can be measured. The current response, being highly sensitive to the potential difference across the sensor and therefore to the species present, forms the basis of amperometric species detection techniques, which performs efficiently as long as the target gases have substantially different amperometric signatures.

The novelty of this approach lies in extending sensing power and sensitivity of these chemical sensors by not only improving the mechanism and materials, but augmenting and complementing the sensor technology with state of the art embedded real time signal processing capabilities. At the root of this signal processing technique is the idea of perturbing an electrochemical sensor with an oscillating potential rather than static voltage levels for extraction of statistically rich information from the current response. The dynamic response is a function of the degree and mechanism of contamination by different species, and therefore carries signatures of the operating condition which can be extracted in the form of low-dimensional pattern vectors for correct classification of gases which may not be otherwise possible. Moreover, by using a bank of sensors and obtaining the pattern vectors from each, sensor fusion techniques can be applied to finely resolve even very similar gases. This unique capability promises to greatly extend the capabilities of commercial EC sensors.

The problem of identification of characteristic statistical patterns is formulated in the next section in terms of observation based estimation of process variables, using a recently reported data driven signal processing algorithm called Symbolic Dynamic Filtering (SDF).

A. Pattern Extraction with Symbolic Dynamic Filtering

This section presents a brief summary of the underlying concepts and essential features of a data-driven pattern identification tool called symbolic dynamic filtering (SDF) [13]. The concept of SDF is built upon the principles of several disciplines including Symbolic Dynamics [14], Statistical Pattern Recognition [15], Information Theory [16] and Probabilistic Finite State Machines [17].

While the details are reported in recent publications [13], the essential concepts are very briefly described here for completeness and clarity.

1) Symbolic Dynamic Encoding: In the setting of the contaminant classification problem, let the dynamic response of the EC sensor be formulated as:

$$\frac{dx(t)}{dt} = f(x(t), \theta(t_s)); \ x(0) = x_0, \quad (1)$$

where $t \in [0, \infty)$ denotes time; $x \in \mathbb{R}^n$ is the state vector in the phase space; and $\theta \in \mathbb{R}^t$ is the lumped parameter vector quantifying the different contaminants as well as different concentrations. The closed and bounded region, within which the trajectory of the sensor response is circumscribed, as illustrated in Fig. 1, may be partitioned into a finite number of (mutually exclusive and exhaustive)
cells, so as to obtain a coordinate grid. Let the cell, visited by the trajectory at a time instant, be denoted as a random variable taking a symbol value from the alphabet $\Sigma$. An orbit of the dynamical system is described by the time series data as: $\Xi \equiv \{x_0, x_1, x_2, \ldots \}$, where each $x_k$, which passes through or touches one of the cells of the partition. Each initial state $x_0 \in \Omega$ generates a sequence of symbols defined by a mapping from the phase space into the symbol space:

$$x_0 \rightarrow \sigma_{i_0}\sigma_{i_1}\sigma_{i_2} \ldots \quad (2)$$

where each $\sigma_{i_k}$, $k = 0, 1, \ldots$ takes a symbol from the alphabet $\Sigma$. The mapping in Eq. (2) is called Symbolic Dynamics as it attributes a legal (i.e., physically admissible) symbol sequence to the system dynamics starting from an initial state.

B. Analytic Signal Space Partitioning

A crucial step in symbolic time series analysis is partitioning of the phase space for symbol sequence generation [18]. This paper presents a partitioning method, called analytic signal space partitioning (ASSP), for symbolic time series analysis. The underlying concept of ASSP partitioning is built upon Hilbert transform of the observed real-valued data sequence into the corresponding complex-valued analytic signal as explained below.

Hilbert transform [19] of a real-valued signal $x(t)$ is defined as:

$$\tilde{x}(t) = \mathcal{H}[x](t) = \frac{1}{\pi} \int_{-\infty}^{\infty} \frac{x(\tau)}{t-\tau} d\tau \quad (3)$$

That is, $\tilde{x}(t)$ is the convolution of $x(t)$ with $\frac{1}{\pi \tau}$ over the real field $\mathbb{R}$, which is represented in the Fourier domain as:

$$\mathcal{F}[\tilde{x}](\xi) = -i \text{sgn}(\xi) \mathcal{F}[x](\xi) \quad (4)$$

where $\text{sgn}(\xi) = \begin{cases} +1 & \text{if } \xi > 0 \\ -1 & \text{if } \xi < 0 \end{cases}$. The corresponding complex-valued analytic signal is defined as:

$$\mathcal{A}[x](t) = x(t) + i \tilde{x}(t) \quad \text{and} \quad \mathcal{A}[x](t) = A(t) \exp(i\varphi(t)) \quad (5)$$(6)

where $A(t)$ and $\varphi(t)$ are called the instantaneous amplitude and instantaneous phase of $\mathcal{A}[x](t)$, respectively.

Given a set of real-valued time series data, Hilbert transform of this data set yields a pseudo-phase plot that is constructed from the analytic signal by a bijective mapping of the complex domain onto the $\mathbb{R}^2$, i.e., by plotting the real and the imaginary parts of the analytic signal on the $x$ and $y$ axes, respectively. The time-dependent analytic signal in Eq. (5) is now represented as a (one-dimensional) trajectory in the two-dimensional pseudo-phase space.

Let $\Xi$ be a compact region in the pseudo-phase space, which encloses the trajectory. The objective here is to partition $\Xi$ into finitely many mutually exclusive and exhaustive segments, where each segment is labeled with a symbol. The segments are determined by magnitude and phase of the analytic signal and also from density of data points in these segments. That is, if the magnitude and phase of a data point of the analytic signal lies within a segment or on its boundary, then that data point is labeled with the corresponding symbol. This symbol generation process is called analytic signal space partitioning (ASSP) [20], [21] and the resulting set of (finite) symbols is called the alphabet $\Sigma$.

One possible way of partitioning $\Xi$ is to divide the magnitude and phase of the time-dependent analytic signal in Eqn. (5) into uniformly spaced segments between their minimum and maximum values. This is called uniform partitioning. An alternative method, known as maximum entropy partitioning [20], maximizes the entropy of the partition, which imposes a uniform probability distribution on the symbols. In this partitioning, parts of the state space with rich information are partitioned into finer segments than those with sparse information. The ASSP algorithm makes use of either one or both of these partitioning methods.

C. Probabilistic Finite State Machine (PFSM) Construction

Using the symbol sequence generated from the recorded current density time series, the state machine is constructed on the principle of sliding block codes [14]. The window of length $D$ on the symbol sequence $\sigma_{i_0}\sigma_{i_1}\sigma_{i_2} \ldots$ is shifted to the right by one symbol, such that it retains the last $(D-1)$ symbols of the previous state and appends it with the new symbol $\sigma_{i_k}$ at the end. The symbolic permutation in the current window gives rise to a new state. The PFSM constructed in this fashion is called the $D$-Markov machine [13], because of its Markov properties.

**Definition** The probability of transitions from state $q_j$ to state $q_k$ belonging to the set $Q$ of states under a transition $\delta : Q \times \Sigma \rightarrow Q$ is defined as [13]

$$\pi_{jk} = P(\sigma \in \Sigma \mid \delta(q_j, \sigma) \rightarrow q_k) : \sum_k \pi_{jk} = 1; \quad (7)$$

Thus, for a $D$-Markov machine, the irreducible stochastic matrix $\Pi \equiv [\pi_{ij}]$ describes all transition probabilities between states such that it has at most $|\Sigma|^{D+1}$ nonzero entries. The left eigenvector $\mathbf{p}$ corresponding to the unit eigenvalue of $\Pi$ is the state probability vector under the stationary condition of the dynamical system [13].

The time series data at the reference condition, set as a benchmark, generates the state transition matrix $\Pi$ that, in turn, is used to obtain the state probability vector $\mathbf{p}$ whose elements are the stationary probabilities of the state vector, where $\mathbf{p}$ is the left eigenvector of $\Pi$ corresponding to the (unique) unit eigenvalue. The state probability vector $\mathbf{q}$ is obtained from time series data at a (possibly) contaminated condition.

Pattern changes take place in the electrochemical sensor due to parametric variations. These variations are a result of poisoning of the membrane, difference in the diffusion properties of the operating gases, etc. The probability distributions obtained by analyzing the current response for different concentration/composition of contaminants serve as low-dimensional feature vectors which are unique to that particular operating condition.
D. Pattern Classification with Artificial Neural Networks

The Symbolic Dynamic Filtering algorithm extracts a low dimension feature vector for each operating condition. The next task, that is pattern recognition and classification can be completed with a multitude of existing algorithms. For the current purpose, a neural network was trained, so that a particular class of pattern vectors representing a specific operating condition leads to a specific target class. The data set was used to train and test a two-layer feed-forward network, with sigmoid hidden and output neurons. Such networks can classify vectors arbitrarily well, given enough neurons in its hidden layer. The network was trained with scaled conjugate gradient back-propagation [22].

III. RESULTS AND DISCUSSION

In order to validate the concept of the electrochemical sensor based on symbolic dynamic filtering, a set of experiments were performed on an EC-sensor using hydrogen (H₂) contaminated with carbon monoxide (CO) and nitrogen (N₂) respectively at varying contamination levels. CO and N₂ were chosen to probe the ability of the algorithm to leverage both physical and electrochemical differences between contaminants to make outcome determinations. That is, CO was chosen because it is known to chemisorb onto the surface of the platinum in the polymer electrolyte fuel cell (PEFC) catalyst layer [23], even at very low concentrations, thus covering a certain fraction of the available reaction sites on the catalyst layer, but have negligible effects on the diffusion of hydrogen. Conversely, N₂ is known to have no adsorption effect on the platinum catalyst layer within the operating conditions of the PEFC. With 25% N₂ content in the anode gas stream, however, the diffusion of hydrogen to the anode catalyst layer is drastically inhibited. Thus, the N₂ represents a family of contaminants that alter the diffusion of the electrochemically active species (in this case, hydrogen) to the catalyst layer, yet do not adsorb on the catalyst layer.

1) Experimental Setup: Experiments were performed using a 850 C fuel cell test station (Scribner Associates Inc.) and a 5 cm² fuel cell with serpentine channels. A commercially available membrane electrode assembly (MEA) with symmetrical Pt loading of 0.3 mg Pt cm⁻² was used in the cell. The SGL 10 BB type diffusion media were used with microporous layer and hydrophobic treatment. Anode and cathode humidifier temperatures were controlled to meet a specific relative humidity value at the operating temperature.

Figure 2 shows the experimental arrangement used in the validation of the sensor. Ultra high purity hydrogen and certified hydrogen gas bottles with premixed CO, supplied by Airgas Inc. were used for the experiments. Flow rates were controlled with calibrated mass flow controllers.

2) Measurement and Data Analysis: In all the experiments, the flow rates on the anode and cathode were kept constant at 69 and 250 sccm, respectively, which corresponded to a stoichiometry of 2 and 3 at 1 A cm⁻². During the experiment, the cell temperature was maintained at 65 °C. Humidification temperatures were maintained such that relative humidity at anode and cathode were 88% and 100% RH, respectively.

The polarization curves were obtained by running in galvanostatic mode, dwelling at each current step for 1 minute in order to reduce the small timescale transients. Voltage values at each step were averaged over the duration of the step, and values are reported. Finer steps were used in the low current density region where changes in voltage are more rapid due to kinetic changes. Also, for the contaminated polarization curves, finer steps were used to improve resolution of performance.

In Fig. 3, selected polarization curves indicate the performance of the fuel cell with pure H₂ on the anode, H₂ contaminated with 5 ppm CO, H₂ contaminated with 25% N₂ as well as a mixture of the two classes of contaminants. The four curves indicate the fundamental difference of the two contaminants: CO chemisorbs onto the catalyst surface, severely reducing the performance of the cell. N₂ blocks H₂ gas diffusion to the electrode, which affects the mass-
transport behavior at higher current; as shown, currents higher than 1.4 A cm\(^{-2}\) could not be reached because of the diffusion limitation.

In order to generate the time series data of current for each contamination condition, it is necessary to perturb the EC system using a voltage pulse when the sensor reaches a poisoned steady state. Once the cell reached steady state, it was excited with an input voltage pulse alternated between 0.65 V and 0.45 V repeatedly with a 5-second dwell time at each value. The input voltage cycle was the same for the healthy (i.e., pure H\(_2\)) system, for different CO levels, and for the N\(_2\) mixed condition contamination.

Current response was measured using a Tektronix AMA6302 Hall effect current probe, at a sampling frequency of 30 Hz. A time series containing a total of 10000 data points was collected over 50 cycles. The time series data were analyzed using the symbolic dynamic filtering method to extract the characteristic pattern vector for each contamination level and species.

Figure 4 shows the dynamic current response of the fuel cell when the applied potential was switched between 0.65 V and 0.45 V. In all cases, the uncontaminated cell yields the highest current, as expected. When poisoned with CO, the current is stable but altered. When the anode gas stream is contaminated with N\(_2\), a unique signal is observed. The current is stable when the applied voltage is 0.65 V, indicating the diffusion of hydrogen through the nitrogen environment is sufficient to sustain the demand of the applied potential. With higher concentrations of nitrogen, lower current is expectedly observed, indicating the inhibited diffusion and reduced concentration of hydrogen. At 0.45 V with N\(_2\) contamination, the signal fluctuates and decreases with time because the diffusion of H\(_2\) through the N\(_2\) inert gas cannot sustain the electrochemical reaction. That is, the hydrogen at the catalyst surface is completely consumed, and the mass-transport of hydrogen through nitrogen becomes the limitation. This mass-transport-limited condition was also observed in the polarization curves (Fig. 3). These dynamic responses yield valuable information that can be used for the identification of multiple contaminant gases.

3) Pattern Generation with SDF: Figure 5 exhibits the current response recorded by perturbing the sensor with a voltage pulse of 5 seconds duration. Each column depicts a scenario, where the sensor is subjected to different gases at different concentrations. From left to right, the gases are pure H\(_2\), H\(_2\) mixed with 5 ppm CO, 25% N\(_2\) and 25% N\(_2\)+5 ppm CO contaminated conditions, respectively.

For ASSP of the time series data described in Section II-B, maximum entropy partitioning is employed in the radial direction with |\(\Sigma_{R}\)| = 2, while using uniform partitioning in the angular direction with |\(\Sigma_{A}\)| = 3. Thus, the alphabet size |\(\Sigma\)\(\triangleq|\Sigma_{R}| \times |\Sigma_{A}|\) = 6 and a depth of D = 1 have been selected. The pattern vector obtained by constructing the D-Markov machine is a representation of the dynamical system that characterizes the EC sensor operation under a particular operating condition. The state probability vectors are shown as histograms at the bottom row in Fig. 5. This visualization displays how the structure of the underlying probability distribution changes as the concentration of contaminants or the nature of the poisoning mechanism changes.

4) Classification: Each experiment was performed several times to generate 21 sets of data with different contaminants. As an initial benchmark test, 16 of the 21 pattern vectors were randomly selected and used for training the neural net. The remaining 5 were used for validation. This was repeated several times to generate a statistic about the true positive rate and the false positive rate of the classifier. The results were uniformly consistent and the classifier could correctly classify the 4 kinds of gas mixtures. However, the main focus of this work is to investigate the efficacy of the algorithm in predicting combination of gaseous contaminants while being trained with only single contaminants. This characterizes a very important scenario in real world CWA monitoring, since even though a classifier may be trained for recognizing a single CWA, it may be needed to identify that gas in the presence of a myriad of other toxic or non-toxic gases. Even presence of dust can act as a deterrent to correct classification. For that purpose, in the second phase, the neural net was only trained with patterns corresponding to Class 1,2 and 3, i.e. pure H\(_2\), H\(_2\)+5ppm CO and H\(_2\)+25% N\(_2\). In the testing phase, however, in addition to all these gases, pattern vectors characterizing mixtures of H\(_2\), 5ppm Co and 25% N\(_2\) were also fed as input to the trained neural net. The outputs of the net is displayed in table I.
TABLE I
MEMBERSHIP INDICATORS FOR THE 4 CONTAMINANT CLASSES

<table>
<thead>
<tr>
<th>Data Set</th>
<th>Pure H2</th>
<th>H2 + Synth CO</th>
<th>H2 + 25% N2</th>
<th>Inference Class</th>
<th>Target Class</th>
</tr>
</thead>
<tbody>
<tr>
<td>Set + 5ppm CO + 25% N2</td>
<td>0.0000</td>
<td>0.9993</td>
<td>0.0000</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>+5ppm CO +25% N2</td>
<td>0.9993</td>
<td>0.0000</td>
<td>0.0000</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Class 1</td>
<td>0.0000</td>
<td>1.0000</td>
<td>0.0000</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Class 2</td>
<td>0.0000</td>
<td>0.0000</td>
<td>1.0000</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Class 3</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Class 4</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
<td>2</td>
<td>2</td>
</tr>
</tbody>
</table>

It may be noted that for data-sets 1 through 16, the outputs of the neural net unambiguously predict the class to which they should belong, indicated by \( \sim 1 \) for one and only one class, while being \( \sim 0 \) for all other classes. However, for sets ranging from 17 to 21, the outputs predict membership in both classes 2 and 3, which implies that the pattern contains characteristics derived from both CO and N\(_2\). Only for data-set 20, the classifier puts unequal weight in Class 3 compared to Class 2. This can be overcome using thresholds. In short, this method is able to correctly predict contaminant mixtures while having been trained with only pure contaminants.

IV. SUMMARY, CONCLUSIONS AND FUTURE WORK

The work presented in this paper reports the use of a stochastic signal processing tool called Symbolic Dynamic Filtering (SDF) for pattern extraction from the EC sensor dynamic response. Perturbing the sensor with an oscillating potential rather than static voltage levels enabled extraction of statistically rich information from the current response. The shape of the dynamic response is a function of the degree and mechanism of contamination by different species, and therefore carries signatures of the operating condition which were then extracted in the form of low-dimensional pattern vectors for correct classification of gases. The reported work is a step toward building a real-time data-driven tool for detecting chemical warfare agents. Further theoretical, computational, and experimental work is necessary before the SDF-based CWA detection tool can be considered for incorporation into the instrumentation and control system of large public buildings or for deployment in the field.

Specifically, more experiments with real CWA agents have to be carried out. Again, the effect of operating conditions on the performance of these EC sensors have to be carefully investigated.

In a broader context, the sensors developed here could be networked into large coordinated clusters. The redundancy would greatly increase veracity of the sensor output, and could also be used to dynamically identify the source location, spread and timing of contamination in urban environments.

REFERENCES