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Polymer-Carbon Composite Films: Reproducibility of Responses in Sensors and Sensing Arrays

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An electronic nose which uses an array of polymer-carbon composite sensors is under development at the Jet Propulsion Laboratory, with future use planned as an event monitor for human habitats in spacecraft. This monitor, the JPL ENose, is being developed with an objective of the ability to identify and quantify 20-30 different compounds at the 24 hour Spacecraft Maximum Allowable Concentration (SMAC) for each compound.[1] Concentrations for common organic solvents and similar compounds tend to be on the order of 10 – 100 ppm. By using a pattern-matching approach to data analysis, compounds causing change in the environment can be identified and quantified if the pattern has been previously recorded.

In order for this device to be useful as an event monitor, the sensors in the array must show repeatable response to a stimulus. That is, two sensors made identically should have identical response, and the response of a sensor to a repeated stimulus should be the same each time. For any software which is developed to identify and quantify changes, sensing films must be made reproducibly. Ideally, if several arrays are fabricated and only one array is used for training data, then the software would still be useable with the other arrays.

These polymer-carbon-black sensing films are made by dissolving the polymer in a solvent (~1.0% wt.) and dispersing carbon black in the polymer solution, 12-20%, by weight, of carbon black to polymer. The sensing films are deposited on ceramic substrates using a pipette; each substrate has eight Au-Pd electrode sets. Each sensing film covers an electrode set with an area of 2mm x 1 mm. The baseline resistance of these films ranges from one to several hundred kΩ, depending on the carbon load, the film thickness, and the polymer identity. The work on making reproducible films focuses on making homogeneous solutions which will result in a homogeneous dispersion of nano-carbon particles in the film, and on developing processing techniques which ensure that each film dries with the polymer left in a similar geometry.

Figure 1 shows the responses of two sensors made from polyepichlorhydrin. Each sensor was deposited by a different person using different polymer solutions. The response to hexane (from 50-1100 ppm) is identical for the two sensors. We have demonstrated this type of reproducibility for over 16 different polymer-composite sensors.

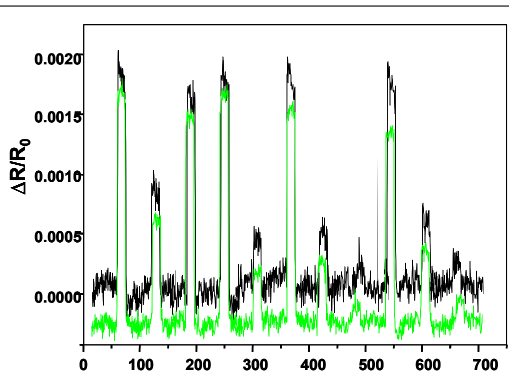

Figure 1. Two Polyepichlorhydrin sensors made from two different solutions and deposited by different people have identical response to hexane (50-1100 ppm).

To test the reproducibility of array responses we fabricated six array sets from the same polymer solutions. Each array consisted of 32 sensors, two sensors each of 16 polymers. One array was selected as the training array, and was trained to 23 different analytes at different background humidities, over an 11 month period. The remaining five arrays were stored in polypropylene containers in ambient conditions. The trained array was then tested for event identification and quantification for all 23 analytes. Event identification and quantification were done using the Levenberg-Marquart Non-linear Least Squares Method.[2,3] For analytes the sensors are most sensitive to, the array and software correctly identified analytes greater than 90% of the time.

From the original sets of arrays, four more array sets were then tested for event identification and quantification for four analytes. Two additional arrays were fabricated 12 months after the original 6, from 16 new polymer solutions. These two arrays were also tested against the same four analytes. Using the parameters from the training set, event identification and quantification was run on the six additional arrays.

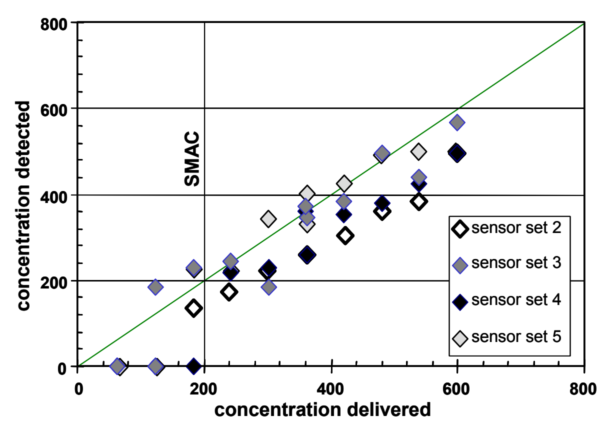

Figure 2. Quantification and identification of acetone using four additional sensor sets.

Event identification and quantification for the additional sensor sets using the training data for the first sensors set was very good, as shown in Figure 2. Because some sensor sets showed a consistent shift in quantification, replacement of the trained set with a second set would require preliminary calibration, but would not require a complete training protocol.

[1] M. A. Ryan, H. Zhou, M. G. Buehler, K. S. Manatt, V. S. Mowrey, S. P. Jackson, A. K. Kisor, A. Shevade, and M. L. Homer, *IEEE Sensors Journal*, **4**, 337 (2004).

[2] M. Lampton, *Computers in Physics,* **11**, 110, (1997).

[3] H. Zhou, M. A. Ryan, M. L. Homer, *IEEE Sensors Journal,* submitted.