Nanomechanical Membrane-Type Surface Stress Sensor: A Case Study on Device Optimization and Food Samples

2023 CNF iREU Intern: Stephanie Wang

2023 CNF iREU Intern Affiliation: Case Western Reserve University

2023 CNF iREU Principal Investigator: Dr. Yoshikawa Genki

2023 CNF iREU Program Location: Olfactory Sensors Group, Research Center for Macromolecules and Biomaterials, National Institute for Materials Science (NIMS), Tsukuba, Ibaraki, Japan

Primary Source of Research Funding: 2023 Cornell NanoScale Facility International Research Experiences for Undergraduates (CNF iREU) Program via National Science Foundation Grant No. NNCI-2025233

Contact: stephanie.x.wang@case.edu, yoshikawa.genki@nims.go.jp

Abstract:

One approach of artificial olfaction looks at the unique combination of vapor molecules that constitute an odor to smell receptors in animals. Nanomechanical sensors, especially those that functionalize microcantilevers, are highly effective in identifying and quantifying these gaseous releases. One of the optimized nanomechanical sensors, the nanomechanical Membrane-Type Surface stress Sensor (MSS) shows promising results. The MSS module was studied to determine optimal measuring parameters due to its highly sensitive nature. The module is affected by environmental factors such as temperature and humidity, so these were monitored and minimized. Signal data from the MSS on various food samples were gathered for comparison as well as future reference.

Summary of Research:

Sensing Technology. The sensing platform on the MSS module comprises of silicon microfabricated sensors. At the edge of the sensors are embedded piezoresistors. As gas molecules flow through the system, they adsorb onto the sensors, causing mechanical deflection. This induces stress on the piezoresistors and changes their electrical resistance. The voltage across this resistance is the output signal, unique to the sample being measured. In this study, eight different receptor layers were used, resulting in eight different output signals from each channel. The module also has humidity and temperature channels.

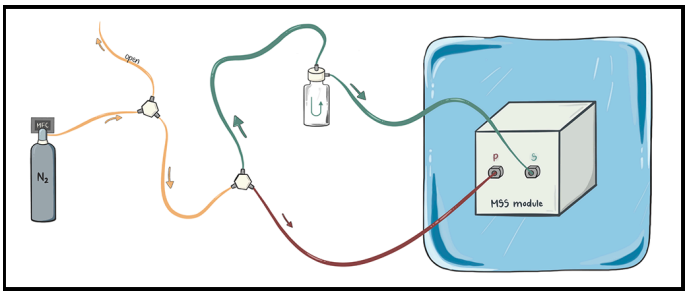

Figure 1: Measurement system depicting connections and airflow.

Measurement System. First, a new measurement system had to be built as shown in the schematic of Figure 1. A line of nitrogen was connected to the system due to its inert nature. It will not react with samples, altering the gaseous composition and skewing data. The flow of nitrogen from a gas tank is set by a mass flow controller (MFC). The sample is connected to the sampling side of the module. Nitrogen is also connected to the purging side of the module. This ensures that molecules adsorbed to the sensors from the previous run can be removed before sampling again. One line is left open from the nitrogen. This ensures that the gas flow from the tank and the settings on the module match up. It also decreases humidity within the system since the MFC is purposely set at a higher flow rate compared to the module. To control temperature, the module is placed in an incubator set at 25°C. All connections were tight and checked often to prevent any air leakages.

Samples. The tested substances were all consumable and ranged from solids to liquids. Water was first tested for practice, ensuring consistent and replicable results. Other liquids included tea, coffee, milk, juice, and soda. Solids tested were matcha powder, coffee grounds, and chocolate.

Measurement Technique. Each substance tested was sampled for one minute. Purging lasted four minutes to ensure that there were no remaining residues from the previous sample. Sampling was repeated four times for each substance. The total testing time per sample was 20 minutes. Multiple trials of the same sample were performed as well. Data was analyzed and graphed using software coded specifically for the MSS module.

Results. Initial tests were run with water in the sampling vial. The goal was to achieve reproducibility by generating the almost equivalent signals across different trials of DI H_2O. Identical signals, however, are difficult to output due to sensors' high sensitivity. One major environmental factor is humidity. In Figure 3, the humidity channel shows a significant difference between two trials of chocolate. Temperature, on the other hand, is quite stable across the two trials, ranging from 30.6-30.7°C. The output signal of channel 3 shows that higher humidity increases

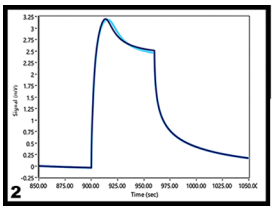
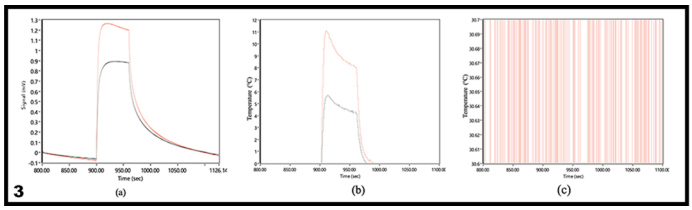
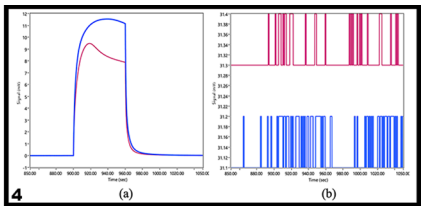

Figure 2: Channel 2 signal of two separate water samples. Figure 3: (a) Channel 3 signal of two different trials testing dark chocolate. (b) Humidity signal corresponding to dark chocolate samples in 4a. (c) Steady temperature signal of above samples. Figure 4: When humidity is stable, but temperature varies, the shape of the voltage signal will vary as shown in Figure 4a.

the voltage magnitude across the resistors due to increased resistance from mechanical deformation. Temperatures are variable and greater than the incubator's set temperature at 25°C. This is due to the pump within the module that generates heat, and it removes contaminated air and location of the sample vials outside the incubator. Samples must be changed out manually, so they cannot be placed inside or else the incubator will have to be opened, defeating its purpose.

The second major factor of concern is temperature. When humidity is stable, but temperature varies, the shape of the voltage signal will vary as shown in Figure 4a. Even a change of 0.3°C can result in a significant change in the output. This is due to samples with different conditions and sensing conditions.

Once more precise signals were achieved, different samples were compared to one another. Principal component analysis (PCA) was performed by placing a cursor on the compilation of signals on the left of Figure 5. Data from every channel for each signal was extracted at the cursor. This data was analyzed using MSS-specific software to generate the principal component graph below. Principal component 1 (PC1) is on the x-axis and PC2 is shown on the y-axis. Here we can distinguish between the different samples tested, which the PCA graph being more readable compared to the output signal information.

Conclusions and Future Work:

We were able to develop a method of extracting data that are reproducible and beneficial in various industrial applications. Using the MSS module, however, still requires a high attention to detail. Regular check-ups on connections within the system are imperative to preventing leaks. Additionally ensuring constant humidity and temperature is crucial for reliable results.

The ability of MSS to test everyday consumables in the form of both liquids and solids is useful in the food industry. Ripeness of produce can be differentiated by the "smell" or hormones released. There are so many applications aside from food, such as testing expiration of make-up products in the beauty industry and testing breath of patients for illness detection in health settings.

Future work can also be done to improve the capability and reliability of the MSS. For example, adding another MSS and having two lines of nitrogen flowing from the source will make sure there is no backflow of air during purging and decreased the humidity. In the current system, samples are not incubated, which caused some temperature fluctuations. Otherwise, the incubator would have to be opened during sample changing. Placing the entire in an incubated glove box could be a solution to this problem.

Acknowledgements:

I would like to acknowledge my PI Yoshikawa Genki for his guidance throughout the summer, the members of the olfactory sensors lab for their advice and encouragement, Dr. Lynn Rathburn for providing this opportunity to research in Japan, and NIMS for providing its facilities.

References:

[1] Minami, Kosuke, et al. Biosensors vol. 12,9 762. 16 Sep. 2022, doi:10.3390/bios12090762.

[2] G. Imamura, K. Shiba, and G. Yoshikawa, Japanese Journal of Applied Physics 55, 1102B1103 (2016).

Figure 5: Left, comparing output signals of many food samples. Right, principal component analysis of same food samples.