Volatile-based Ratiometric Infochemical Communication System Using Polymer-coated Piezoelectric Sensor Arrays

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***Abstract*— In this paper we report upon the implementation and operation of a novel infochemical communication system that uses volatile components to encode, and polymer-coated piezoelectric sensors to decode ratiometric information. Blend information is generated in specific ratios of compounds by a micromachined mixer/evaporator module that is connected to a high-precision multi-channel syringe pump. Eight quartz crystal microbalances (QCM) and four dual surface acoustic wave (SAW) resonator sensors are used for blend detection. Ratiometric communication was demonstrated using seven different blend ratios at two concentrations of isoamyl alcohol and ethyl acetate. The receiver side of the system was able to classify the specific ratios of chemical compounds using simple multivariate data analysis, thus decoding the ratiometric information encoded by the emitter side.**

I. Introduction

The chemical-based form of communication is an emerging branch of information technology employing chemicals alone for possible applications in information transmission, labeling and cellular communication. This type of hemical communication is a ubiquitous way of information exchange in the natural world as animals rely on it for a variety of behaviors such as finding mates, locating food sources, or avoiding predators. Insects in particular rely on blends of several compounds to communicate using their signaling and processing systems.

A novel communication system has been designed and implemented to demonstrate volatile-based ratiometric information encoding, transmission and decoding. The system described here is capable of both detecting – using arrays of polymer-coated piezoelectric sensors – the ratio of predefined volatile compounds and recovering the ratiometric information deployed in the environment. With complementarily tuned sensors and advanced data analysis (e.g. neuromorphic processing), the presented ratiometric system has the potential to function as a method for autonomous communication and control of mobile robot teams.

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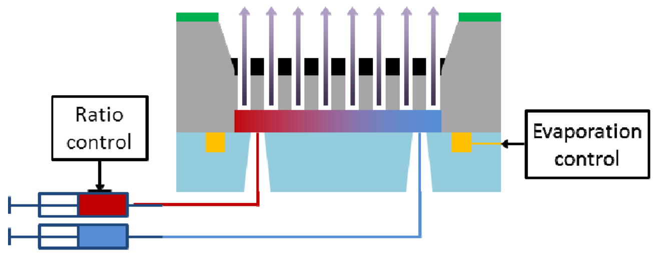

3m	EA	3m	EA	3m	EA	3m	EA	3m	EA	3m	EA	3m	EA
Ratio 1		Ratio 2		Ratio 3		Ratio 4		Ratio 5		Ratio 6		Ratio 7	
0	20	4	16	6.7	13.3	10	10	13.3	6.7	16	4	20	0
Ratio 8		Ratio 9		Ratio 10		Ratio 11		Ratio 12		Ratio 13		Ratio 14	
0	40	8	32	13.3	26.7	20	20	26.7	13.3	32	8	40	0

Figure 1. Schematic diagram showing the basic principle of ratiometric encoding by blend mixing and volatilization by controlled evaporation. The table shows the 14 different ratios of isoamyl alcohol (3m) and ethyl acetate (EA) used for information encoding (all units in µl).

II. Methodology

A. Ratiometric signal generation

The basic principle of ratiometric information encoding by producing a precise mix of predefined compounds in programmable ratios of concentration and volatilizing by controlled thermal evaporation is illustrated in Fig. 1. Mixtures of isoamyl alcohol (3m) and ethyl acetate (EA) – two behaviorally relevant plant compounds detected by many species of insects – are used to encode ratiometric data. The ratios are programmed by a two-channel nanoliter-accuracy syringe pump connected to a bioinspired micromachined evaporator termed 'artificial gland' [1]. As shown in the table in Fig.1, 14 different ratios of the two plant volatiles were produced by this system. Rectangular microfluidic channels deliver the mixtures from the two inlets to the ~0.375 µl reservoir located

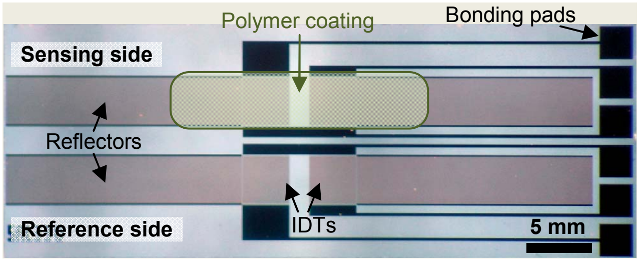

Figure 2. Optical micrograph of a 262 MHz Rayleigh mode dual SAW resonator sensor fabricated on X-propagating ST-cut quartz substrate with aluminum electrodes for gas phase measurements.

under a black silicon covered perforated silicon membrane. The volatile mixtures are passed through the via-holes in the membrane forming small droplets that are released into the environment via thermal evaporation. Each ratio was introduced three times in a random sequence to eliminate the memory effects of the polymer coatings. The small size and the low dead volume of the device enable fast response times for ratiometric encoding. This microevaporator-based technique was found particularly suitable for the generation of precise plume patterns. Moreover, the ability to control release rates by controlling the temperature makes this technique ideal for continuous-flow dynamic measurements.

Chemical detection using piezoelectric sensor arrays

A robust, high-sensitivity system based on polymer-coated surface acoustic wave (SAW) devices and quartz crystal microbalances (QCMs) was developed to detect the ratiometric mixtures. The 8 QCMs are commercial 30 MHz polished crystals with Au electrodes [2]. The SAW resonators (Fig. 2) are designed in a dual configuration to operate at 263 MHz (wavelength of 12 µm) and fabricated on ST-cut quartz substrates using Al electrodes. The interdigitated transducers (IDTs) of the sensors consist of 60.5 finger pairs with 3 µm finger width and have an acoustic aperture of 720 µm. The cavity length is 1764 µm and the overall die size is 9.56 × 3.56 mm^2. The IDTs are surrounded by 550 reflector gratings of 3 µm pitch to create a standing wave pattern. Four polymers (polycaprolactone, poly(9-vinylcarbazole, poly(styrene-co-butadiene) and poly(ethylene-co-vinyl acetate)) were deposited on the sensing side of the SAW sensors by air-brushing while the other side was left blank to serve as reference.

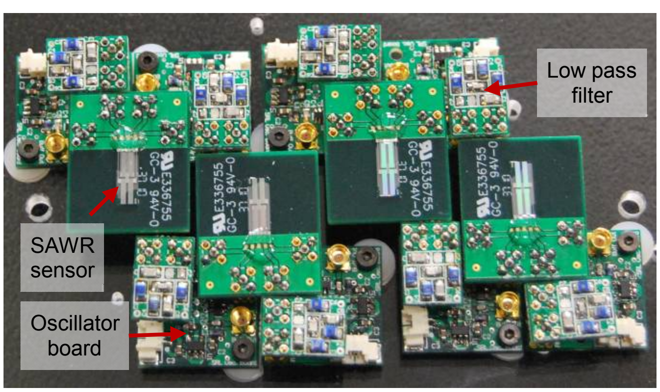

Figure 3. Photograph of a 4-sensor SAW resonator array with the associated radio-frequency oscillator circuits and low pass filters.

The dual configuration of the sensors enables the measurement of the differential outputs between the sensing side and the reference side which ameliorates the common mode variations and ensures that the measured responses are produced purely by the polymer coatings. Analyte induced perturbations of the surface acoustic wave are measured indirectly by using each SAW sensor as a resonating element in the feedback loop of a basic radio-frequency (RF) oscillator circuit shown in Figure 3. In order to suppress oscillations at spurious frequencies, a low-pass filter was added to the feedback loop. The 4-sensor dual SAWR array with the associated RF oscillator circuitries and filters mounted on a Perspex plate are shown in Fig. 3.

B. Infochemical communication system setup

In order to demonstrate ratiometric infocommunication an experimental setup was constructed (shown in Fig. 4) that comprises of the evaporator driven by a neMESYS high-precision multi-channel syringe pump (cetoni GmbH, Korbussen, Germany) and a microcontroller based custom digital evaporation controller, a 14×14×40 cm^3 Perspex odour chamber connected to a membrane venting pump, an SHT75 digital temperature and humidity sensor (Sensirion AG, Staefa, Switzerland) and the piezoelectric sensor arrays.

The QCM sensors are connected to a commercial JLMQ sensor interface (JLM Innovation, Tübingen, Germany) used to drive the sensors and record the frequency output via a USB port of a personal computer. The oscillation frequency of the SAW resonator circuits was measured and recorded using a commercial 4-channel measurement instrument (JLM Innovation, Tübingen, Germany) connected to a personal computer.

III. Results

The typical frequency output (Fig. 5) of a polymer coated dual SAWR to three consecutive injections of 30 µl isoamyl alcohol shows that the SAW sensor response is fast, reproducible and low noise.

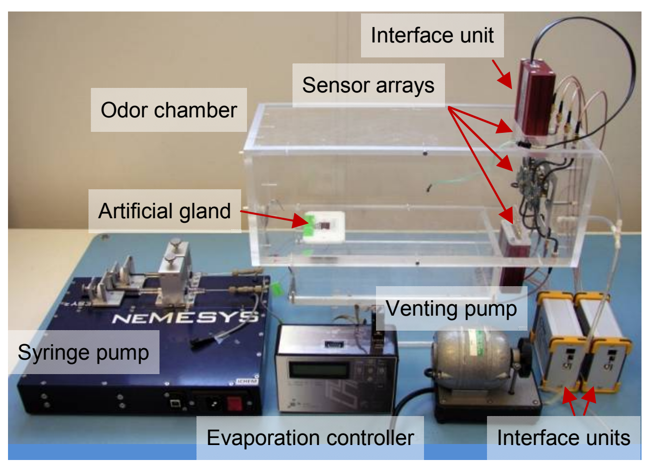

Figure 4. Experimental setup for ratiometric testing consisting of an odor chamber, a venting pump, a high-precision syringe pump, a micromachined artificial gland, QCM and SAW sensor arrays and associated drive and interface circuitries.

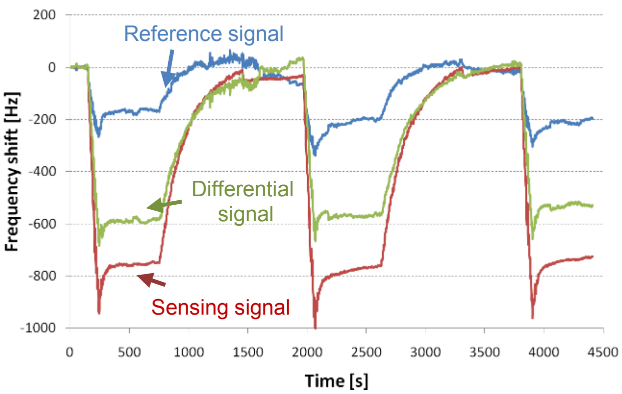

Figure 5. Typical frequency output of a dual SAWR sensor in response to three sequential injections of a volatile compound.

In a similar manner, the frequency response of the polymer coated SAWR sensors was measured to various levels of fruit volatile concentrations to establish the minimum sensitivity and the linearity of the sensors. In random repeated sequences, 10, 20 and 30 µl of ethyl acetate and 5, 10, 20 and 30 µl isoamyl alcohol were introduced into the odor chamber using the microevaporator. The average differential SAW responses were found to be 1.3-5 Hz/µl to ethyl acetate and 6-17 Hz/µl to isoamyl alcohol. The response of the sensors scaled linearly with volatile concentration confirming that the sensors operated in the non-saturated regime.

In an attempt to detect and decode ratiometric information, the frequency response of the sensors to each ratiometric mixture listed in Fig. 1. was measured. The encoded and transmitted ratiometric information was successfully recovered by performing principal component analysis of the steady state sensor responses to the blend mixtures as shown in Fig. 6. Each of the 14 different ratios representing a specific infochemical message forms a well-defined and tight cluster in the plot. The clusters corresponding to mixtures of a given volume are distributed evenly between the clusters corresponding to the pure components (R1 and R8 for ethyl acetate, and R7 and R14 for isoamyl alcohol). Moreover, clusters of the same blend ratios but of different concentrations are clearly separable as well.

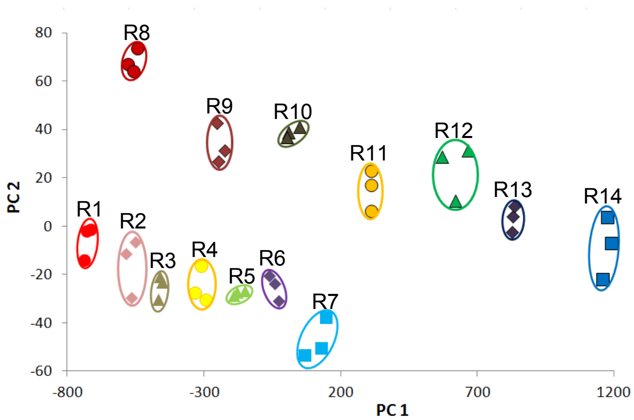

Figure 6. Decoding of ratiometrically encoded information by principal component analysis of sensor responses to blend mixtures.

IV. Conclusions

A volatile-based communications system based on a micro-evaporator and polymer-coated piezoelectric sensors was constructed in order to prove the concept of infochemical based ratiometric communication. Using mixtures of two plant compounds, ethyl acetate and isoamyl alcohol, information was ratiometrically encoded and released into the environment, then successfully detected and recovered using sensor arrays and performing principal component analysis.

Although the results presented here are based on static data analysis, the developed communication system can facilitate high-resolution real-time measurements and signal processing. The sensors outputs can be directly fed into a neuromorphic model implemented in a field-programmable gate array for dynamic feature extraction and data analysis. An implementation of an insect-based model has been tested and was shown to recover blend ratio information from the early transient phase of the sensor responses [3]. The combination of fast piezoelectric sensors and neuromorphic signal processing is a promising utility for rapid and efficient ratiometric chemical blend processing and infochemical communication.

In the future, we aim to develop a compact and low power portable chemical sensors system by integrating the surface acoustic wave sensors and the associated circuitry into a single monolithic microelectronic system. As part of this integration, an application-specific integrated circuit comprising a high-frequency analog front-end and a digital controller block was designed and fabricated [4].

In conclusion, we have demonstrated that the combination of precise spatiotemporal signal generation using plant volatiles with highly sensitive detection and signal processing provides a powerful tool for small-scale, yet high-throughput infochemical communication.

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