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Research Article

Characterization Studies of a GC-based SAW Sensor System for Potential Detection and Identification of Toxic Environmental Gases/Vapors

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ABSTRACT

The characterization of a custom-designed GC-based Surface Acoustic Wave (SAW) e-Nose sensor system is presented here to study the sensing ability of the sensor system to detect and identify low medium and high toxic vapors. A semi-automated multi-vapor generator generated vapors of chemical compounds which were then exposed to the sensing system to examine its performance under various concentrations. Time-domain vs. frequency response of GC-SAW sensor was noted for repeated cycles against different chemical compounds like xylene, 1, 2 dibromoethane, dimethyl sulfate, triethyl phosphate, nitrobenzene, phosphorous trichloride being tested. The generated data was examined using a Principal Component Analysis (PCA) technique to detect a unique response for every individual chemical compound. Experimental results were reported.

Keywords: SAW oscillator; SAW sensor; Pre-concentrator; Chemical Warfare Agents; Hazardous Chemicals.

1. INTRODUCTION

The identification of hazardous vapors, chemical compounds and toxic gases in various atmospheric conditions poses a major challenge today. Chemical vapor sensors are eventually required to safeguard humanity from major chemical outbreaks by detecting them proactively. Ion mobility spectroscopy, SAW sensors, infrared spectroscopy, colorimetric sensors, flame photometry, Raman spectroscopy and photoionization detectors are some of the techniques to detect chemical vapors (Arshak *et al.* 2004; Bhasker Raj *et al.* 2013; Venkatesan and Haresh 2013; Haresh *et al.* 2013; Banu Priya *et al.* 2014; Gowdhaman *et al.* 2018; Thirumal *et al.* 2018; Gowdhaman *et al.* 2020). Based on various specifications, each sensor type has its advantage and limitations (Liu *et al.* 2012; Devkota *et al.* 2017; Xu and He 2017). The majority of the sensors produce false-positive readings, which are costly and complicated. For the design to be compact, low cost, high sensitivity, quick response and ability to be used wirelessly, Surface Acoustic Wave (SAW) sensors are considered to be the most dependable chemical vapor sensor as well as gas sensors (Nimal *et al.* 2006; Nimal *et al.* 2009; Gowdhaman *et al.* 2020). SAW sensors use acoustic waves for sensing. When an acoustic wave travels through a piezoelectric substrate, any modification in the path of propagation caused by chemical vapor adsorption on the surface of the piezoelectric substrate alters the wave's amplitude, velocity and frequency in comparison to a reference.

Chemical warfare (CW) agents, toxic industrial chemicals (TICs) and volatile organic compounds (VOCs) have all been used in chemical terrorism and battlefields in recent years (Nimal *et al.* 2006; Nimal *et al.* 2009; Gowdhaman *et al.* 2018; Thirumal *et al.* 2018; Sayago *et al.* 2019; Wang *et al.* 2019; Gowdhaman *et al.* 2020). Identification of the above chemical mixtures is a major concern in the defense sectors (Chauhan *et al.* 2008) and SAW sensors are well-suited for detecting such CW agents (Joo *et al.* 2007; Raj *et al.* 2013a; Pan *et al.* 2015). These are mainly classified as nerve, blood, blister and choking agents (Cheeke and Wang 1999; Haresh *et al.* 2013; Raj *et al.* 2013). Since the production and distribution of CW agents are forbidden, their simulants were used to analyze them in this study. TICs are less lethal than CW agents, yet they are extremely intimidating due to their ease of access and toxicity. Consequently, terrorists can employ TICs as a preferred weapon (Arshak *et al.* 2004; Gongora *et al.* 2018; Bongiovanni Abel *et al.* 2018; Benetti *et al.* 2019; Wang *et al.* 2019; Gowdhaman *et al.* 2020). The hazard index rank (ITF-25) categorizes TICs as high, medium or low hazard, based on the chemical's manufacture, conveyance, stockpiling, poisonous nature and vapor pressure (Wohltjen *et al.* 1979; Chang *et al.* 1998; Cheeke *et al.* 1999; Islam *et al.* 2015; Bui *et al.* 2016; Priya *et al.* 2016; Devkota *et al.* 2017). VOCs that are not immediately dangerous but have long-term effects interfere with the detection of CW agents very often.

The aim of our present work is to characterize a GC-based SAW electronic nose (e-Nose) sensor by exposing it to various chemical agent vapors like VOCs, simulants and TICs. In this study, the characterization of the sensor was performed for different vapors at different temperature and their sensing capabilities as a potential chemical vapor sensor were tested. The data obtained for the sensing response of each chemical vapor at different concentrations were analyzed using custom-developed data analysis software. Further, principal component analysis (PCA) - a software tool was used to analyze the obtained data and discriminate the chemical compounds.

2. WORKING PRINCIPLE AND COMPONENTS OF SAW-BASED E-NOSE DEVICE

SAW electronic nose (e-Nose) device utilizes piezoelectric effect to convert mechanical energy into electrical energy and vice versa. The sensing process in this sensor device starts with creating SAW and detecting variation in the wave propagation by means of frequency variations (Gowdhaman *et al.* 2020). Interdigital transducers - specially designed metallic strips on a piezoelectric substrate, were used to generate SAW. The variation in the wave propagation was measured due to physical and chemical factors (Morgan *et al.* 1998; Cheeke *et al.* 1999; Alizadeh *et al.* 2008; Islam et al. 2015; Raj *et al.* 2015; Mittal *et al.* 2015). The changes in SAW propagation were generated by the changes in mass loading, viscoelasticity and conductivity of a sensing layer coated in their path when exposed to sample vapors in chemical sensing applications; these changes can be measured in terms of frequency, phase and insertion loss of corresponding electric signal (Devkota *et al.* 2017).

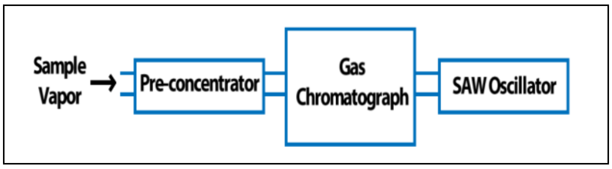

Fig. 1: Schematic diagram of a GC-based SAW e-Nose sensor

The detection of chemical vapor at a low concentration of the sample is achieved by the pre-concentrator. The Pre-concentrator and gas chromatograph (GC) in SAW e-Nose sensor set-up, shown in Fig. 1 were used to enhance the detection limit and selectivity to particular vapor at lower concentrations. These components were used to concentrate the chemical sample by holding it for some time and allowing it for detection after reaching the detection limit of the SAW sensor. Pre-concentrator concentrates the sample by isolation and separation techniques. The concentrated sample vapor was allowed into a gas chromatograph to distinguish the chemical compounds of a mixture of sample vapor. The different chemicals reach the GC outlet at different time intervals because they travel at different speeds within the GC column. Separated sample vapors were allowed into SAW e-Nose sensor for further detection and identification (Bui *et al.* 2016; Priya *et al.* 2016; Singh *et al.* 2016; Devkota *et al.* 2017).

SAW sensors in the e-Nose device use the surface acoustic waves, which were generated by utilizing interdigital transducers (IDT). IDTs are periodic metal electrodes deposited on a piezoelectric substrate. The polarities of the IDT electrodes were changed by applying an alternate electric field that causes periodic vibrations in the piezoelectric material. The generated wave traverses on both sides on the surface of the substrate and they are perpendicular to the IDTs. Finally, these waves were transferred as an electric signal. The SAW velocity in the piezoelectric substrate is 105 times smaller than the electromagnetic radiation, making it suitable for use in portable devices (Liu *et al.* 2016; Mujahid *et al.* 2017).

SAW resonators are used for the generation and detection of surface acoustic waves. There are two types of SAW resonators, i) One-port resonator and ii) Two-port resonator. In this work, one port SAW resonator was employed for its high Q-value. One port SAW resonator consists of one IDT in the center and two reflectors on both sides of IDT, as shown in Fig. 2.

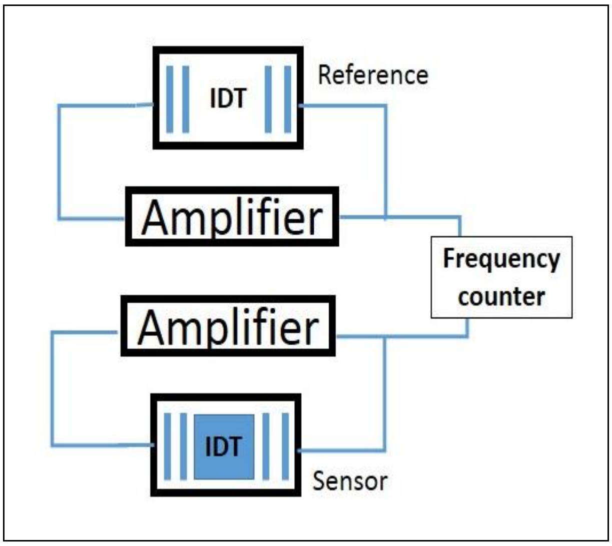

Fig. 2: Schematic of Differential SAW oscillator

The reflectors are shorted or groove-shaped metal stripes to eliminate regeneration of SAW (Namdeo *et al.* 2013; Manenti *et al.* 2016). The Colpitt oscillator was formed by the SAW sensor while connected as feedback to an amplifier, as shown in Fig. 2. Two oscillators were used; one SAW device with a sensing layer and another one with a bare SAW device. The bare SAW device acts as a reference oscillator. These two oscillators were connected in a differential mode to minimize the error due to fluctuations and other ambient conditions. The difference in frequency between the reference oscillator (f_0) and the sensing oscillator (f_s), F = (f_0-f_s), was measured by the frequency counter (Raj *et al.* 2013a; Singh *et al.* 2014; Fahim *et al.* 2017; 2021).

3. CHARACTERIZATION OF SAW E-NOSE DEVICE

In this work, the characterization of the SAW e-Nose device was performed by exposing it to chemical vapors of volatile organic compound - xylene, simulants of chemical warfare agents - triethyl phosphate, nitrobenzene and toxic industrial chemicals (TICs) of low (Dimethyl sulfate), medium (1,2 dibromoethane) and high toxic (phosphorous trichloride) chemical compounds. The sensing capabilities of this device against these chemical compounds were analyzed by utilizing a vapor generation and delivery system, which included a multi-vapor generator system for the generation of vapors and a zero air generator for delivery of vapors at desired concentrations.

Zero air is synthetic air, normally used as a carrier gas. The carrier gas generated at a zero-air generator is completely free from hydrocarbons, CO_2, NO_x and H_2O from the air. It filters out the hydrocarbons less than 0.1 ppm. This eludes the necessity of incompatible air cylinders, which are hazardous to laboratory operations.

The schematic of a multi-vapor generator (MVG) system is shown in Fig. 3. It consists of valves V_1, V_2, V_3, V_4, respective to the flasks F_1, F_2, F_3, F_4 which are used to allow the carrier gas into the flasks. Valve V_5 was used for the direct dilution of chemical vapors. This custom-designed semi-automated MVG system could generate a mixture of multiple vapors simultaneously by loading different samples in the thermal flasks. The flow rate of carrier gas was controlled by flow controllers (M_1 to M_4), M_5 and M_6, exclusively meant for controlling the test vapors of specific concentration to the sensor device precisely. A hydrocarbon-free air was used as the carrier gas to carry the vapors from the sample (analyte) to the sensor.

A small quantity of analyte (VOCs, simulants, TICs), either in liquid or solid form, to be tested, was placed at the thermal flasks (F_1, F_2, F_3 and F_4) for 15 minutes under different temperatures (30 °C, 35 °C and 40 °C). The temperature of the thermal flasks can be altered with the help of a thermocouple attached to the PID (Proportional Integral Derivative) controller. The generated target chemical vapors at different concentrations were exposed to the sensor device for testing and calibration.

The concentration of target gas was calculated via weight loss measurement. The weight of the liquid was noted for each loading and unloading of the sample from the flask. The weight loss after the vaporization was estimated by the least-square fitting to decrease the error. The data generated by the SAW e-Nose device on exposure of target gas was collected using a data acquisition system and further evaluated using specially designed data processing software. The analysis and classification of test data were performed using Principal Component Analysis (PCA) - a pattern recognition method, which helps to classify and identify the target gas vapors.

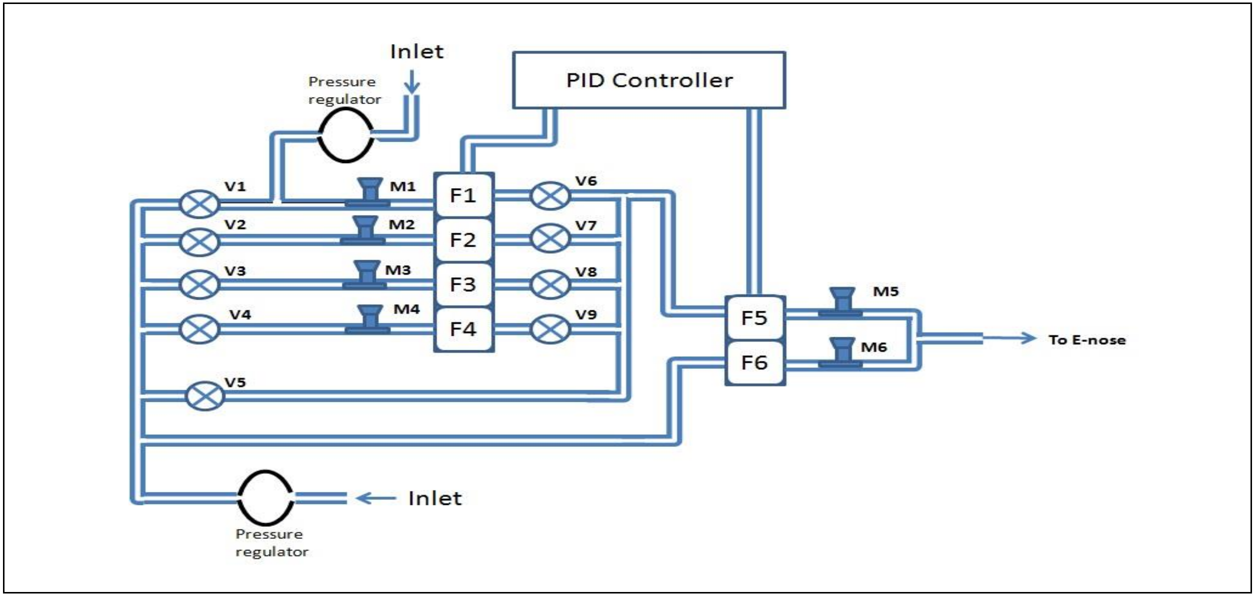

Fig. 3: Schematic of multi-vapor generation system

4. RESULTS AND DISCUSSION

The frequency response of custom-designed SAW e-Nose device was studied on exposure of chemical vapors of xylene, 1, 2 – dibromoethane, dimethyl sulfate, triethyl phosphate, nitrobenzene and phosphorous trichloride. Several frequency responses were recorded in terms of time versus frequency. The corresponding shift in frequency was noted as a reference for individual target gas vapors. It was interesting to note that the time domain remained unchanged for an individual chemical compound, whereas frequency shift was associated with the temperature. As the temperature increased, the concentration increased and these changes were represented in terms of frequency shift which shows the sensing efficacy of the e-Nose device (Fig. 4). The device was exposed to target gas for a repeated number of cycles, out of which the sensing response for one test cycle is shown Fig. 4. The calculated frequency shift for three different temperatures of the sample vapors xylene, 1, 2 - dibromoethane, dimethyl sulfate, triethyl phosphate is 2499, 2544, 2147 and 1290 Hz at 30 ºC, 2222, 2133, 1696 and 1208 Hz at 35 ºC and 1870, 1816, 1313 and 1150 Hz at 40 ºC, respectively. These frequency shifts correspond to the second raising peaks and the first one was not taken into account as it was caused by the humidity in the atmosphere.

As evident from Fig. 4 (d) and Fig. 4 (e), nitrobenzene and phosphorous trichloride sample vapors gave rise to two peaks other than the humidity peak. The frequency shift of nitrobenzene with reference to the first peak (1347, 1255 and 1158 Hz) and the second peak (1144, 1136 and 1066 Hz) at 30 ºC, 35 ºC and 40 ºC were obtained. Similarly, for phosphorous trichloride, the frequency shifts for the first peak (1887, 1644 and 1485 Hz) and the second peak (1308, 1233 and 1178 Hz) at 30 ºC, 35 ºC and 40 ºC have occurred. These responses were correlated with the reference data of the e-Nose device and unique responses were obtained against individual chemical compounds. As the device underwent a repeated number of trials for each target gas, the data sets obtained were large; therefore, a pattern recognition tool called principal component analysis (PCA) was used to analyze the raw data.

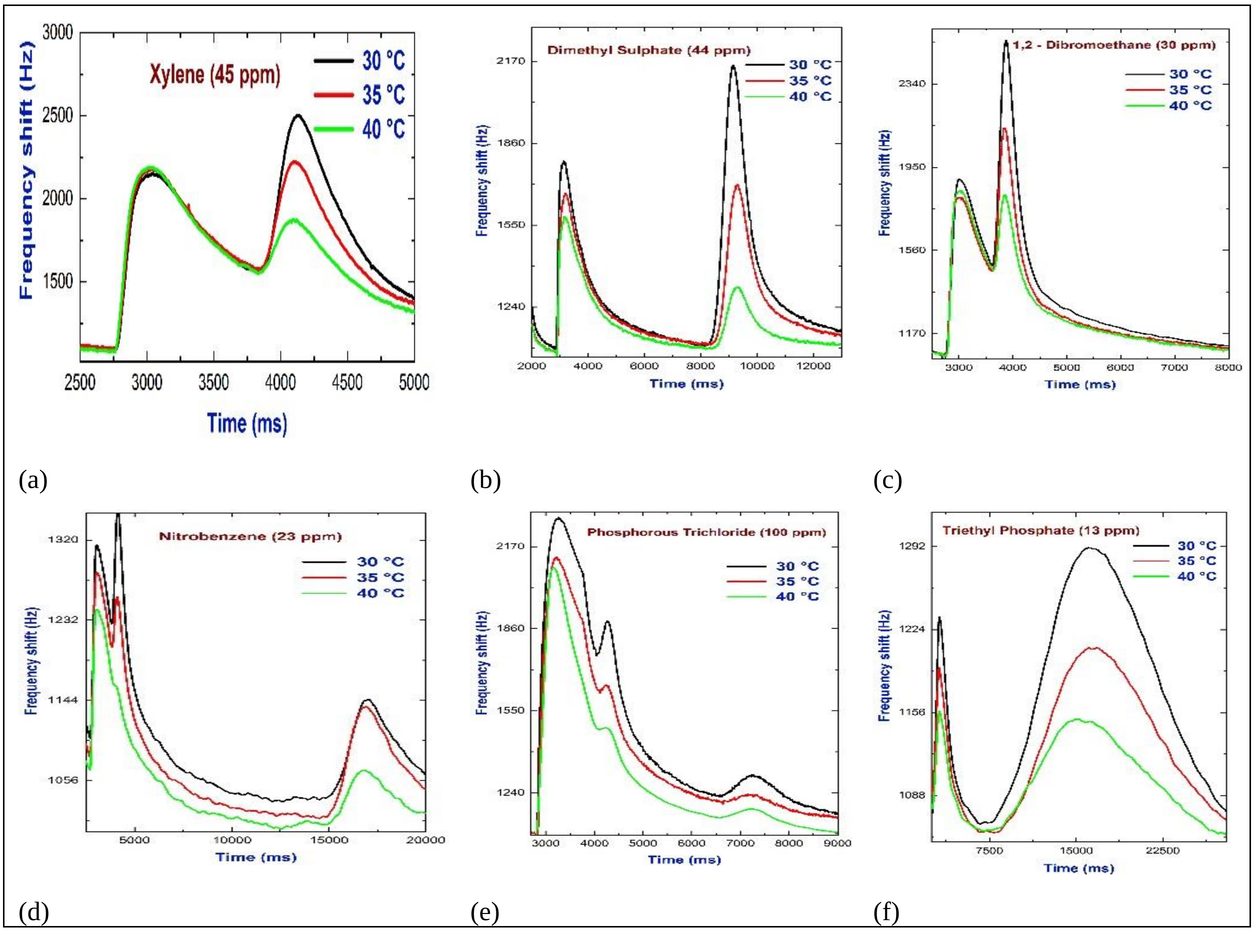

Fig. 4: Frequency responses of GC-based SAW e-Nose device at different temperatures of target chemical vapors: (a) Xylene (b) Dimethyl sulphate (c) 1, 2 – dibromoethane (d) Nitrobenzene (e) Phosphorous trichloride (f) Triethyl phosphate

4.1 Principal Component Analysis

The principal component analysis (PCA) is a widely used pattern recognition technique for multiple data sets. It reduces the dimensionality of multiple data sets while conserving most of the information (EL Gowini *et al.* 2010; Jha *et al.* 2010). PCA constructs the new set of variables as a linear combination of primary variables. The eigenvectors of new variables are the axes that contain the most information (most variance) and they are called principal components (PC). The eigenvalues are the coefficients of eigenvectors. The first principal component (PC1) contains the maximum variance, which means the highest eigenvalue, and the rest is in the second and so on (Singh *et al.* 2014; Singh *et al.* 2016) (Refer Table 1).

In the present work, the PCA technique was used for the analysis of GC-based SAW e-Nose device responses against six different target gas vapors at three different temperatures. Fig. 5 shows the PCA plot for xylene, 1, 2 - dibromoethane, dimethyl sulfate, triethyl phosphate, nitrobenzene and phosphorous trichloride at different concentrations. It can be seen from the plot (Fig. 5) that all the vapors are distinguished well from each other. The eigenvalue, percentage of variance and cumulative for the principal component of data sets are shown in Table 1.

From the table, it is evident that the first two principal components possess the most information and their percentage of variance is 71.25 % and 12.21 %, respectively. Thereby, the PCA technique is effectively utilized to discriminate six different target gas vapors used in this characterization of the SAW e-Nose device.

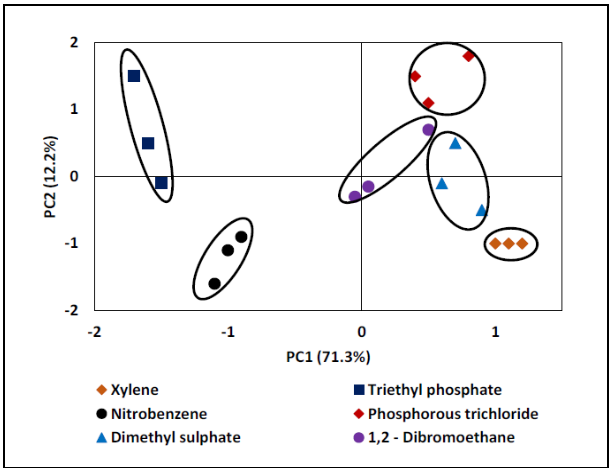

Fig. 5: PCA score plot of Xylene, 1, 2 - dibromoethane, dimethyl sulfate, triethyl phosphate, nitrobenzene and phosphorous trichloride

Table 1. Eigenvalue, percentage of variance and cumulative percentage estimated by PCA on the correlation matrix of the normalized frequency response of SAW e-Nose device

Principal Component Number	Eigen value	Percentage of Variance (%)	Cumulative (%)
1	2.42 x 10^7	71.25	71.25
2	0.42 x 10^7	12.21	83.46
3	0.30 x 10^7	08.88	92.34
4	0.18 x 10^7	05.25	97.59

5. CONCLUSION

Gas chromatography and the surface acoustic wave-based e-Nose device was successfully characterized using chemical vapors of xylene, 1, 2 - dibromoethane, dimethyl sulfate, triethyl phosphate, nitrobenzene and phosphorous trichloride at different concentrations. The unique signature obtained for each chemical compound in terms of frequency response shows the sensing ability and selectivity of the device. PCA is an effective pattern recognition tool for the classification of target gas vapors. The low detection limit (sub-ppm level), quick response and low recovery time was added advantage of this device. Hence, this device has futuristic scope for further development and characterization for on-field applications in the future.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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