Electronic Nose- An epitome of perfect sensor systems

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An old saying, “United we stand, divided we fall”. Synonymously, if an array of sensors is used instead of a single one, then a high degree of specificity can be achieved. Thus, there is a great advantage of using multiple sensors, training these and performing artificial intelligence. These are very closely related and together the system is called as ‘Electronic Nose’ or ‘E-nose’. An E-nose is an artificial nose which mimics the human olfaction system, uses the intelligence to make decisions and identify specific analytes from a mix of analytes [1]. Traditionally, an E-nose system has an array of electronic sensors, pattern recognition ability and decision making capability. In simple words, it is a machine, intelligent enough to recognize flavours and odors. It started around 1980s when scientists were making efforts to manufacture an intelligent device for recognizing odors. This field is still being explored and continuously expanding. Immense attempts are evidenced in the literature which talk about e-nose [2-6]. Various application areas of E-nose are agriculture, cosmetics, biomedical, food, environmental, pharmaceutical and various other scientific research fields.

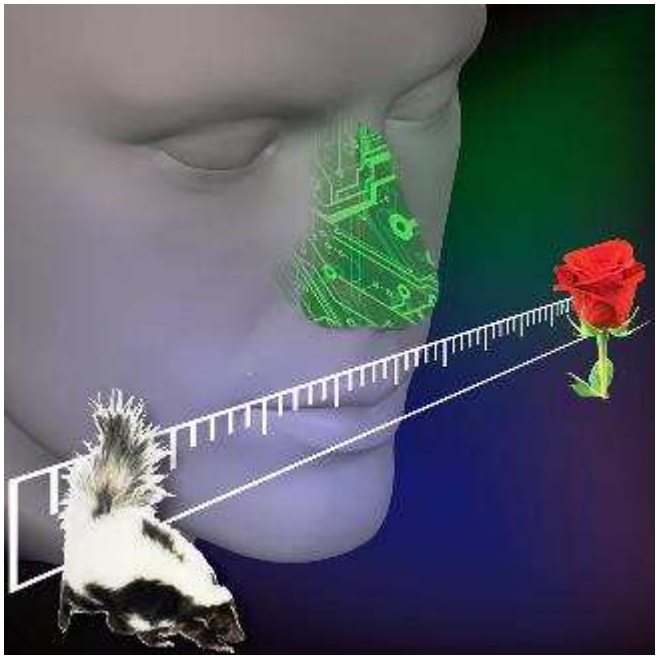

Fig. 1. Electronic nose schematic [1]

The most primary section of the E-nose is a sensor array. The array comprises of individual sensors which give different response to analytes. The parameters which set the benchmark of the quality of a sensor are: specificity, sensitivity, response- and recovery-time, ageing, stability and reproducibility. Many reports have shown the performance of sensors based on these parameters [7-9]. And of course, the parameters are highly dependent on the make of these sensors which includes the material, mechanism, interconnects and packaging. Several materials and their types have been explored till date, examples include porous, metal oxides, semiconductors, nanowires, nanorods, thin films, nanotubes etc. Also, many mechanisms like electrical, optical, chemical, electrochemical and physical are continuously being researched. These building blocks of a sensor effect not just the performance but also the viability of the sensor. The biggest challenge with the individual sensors is specificity especially when it is exposed to a combination of analytes of similar type. Here, the sensor needs to be intelligent to decide which analyte it is exposed to. Also, then it is required for it to apply learning patterns, think, decide, quantify and convey. Thus the sensor array passes on the information to a signal conditioning circuit which extracts the information from the signal and sends it to the decision making (neural network) system. This uses graphical analysis, multivariate data analysis and network analysis to make decisions. In the end, the quantified information is converted to a user friendly interface for display.

Artificial intelligence (AI) is an area of computer science and electronics where the intelligent machines/systems are endowed with the intellectual processes of human characteristics or machines just behave like humans. This similarity with human brain makes the operation of such systems extremely challenging. These not have to just be fast like humans but also accurate and should have a good memory capacity to save the necessary information. Efforts are being made to have

AI in applications like medical diagnostics, space and communications and voice or handwriting recognitions. There are three main aspects of AI, (1) main AI (a machine doing work like human would do), (2) Machine Learning (a computer learning how to perform a function) and (3) Deep Learning (technique or algorithm for learning a pattern) [10].

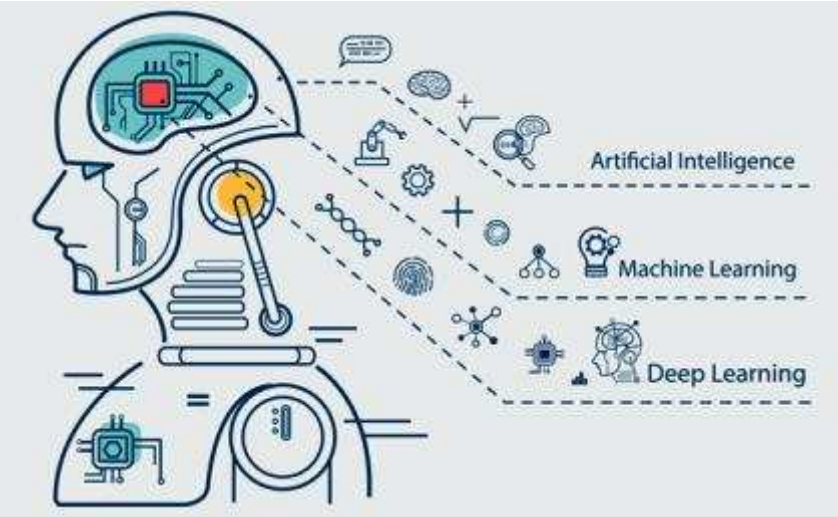

Fig. 2. Artificial Intelligence and its aspects [10]

One of our work on an E-nose system was published in IEEE Transactions on Electron Devices in 2018 where the concept of E-nose with 3 sensors was discussed [11]. Three types of devices viz. porous silicon (PS), TiO_2 on PS and TiO_2 on crystalline silicon (c-Si) were fabricated, characterized and tested for sensing applications. These were fabricated using scalable processes and wire bonded to the header for packaging. Sensing tests were performed in the presence of VOCs (volatile organic compounds) and the data from each sensor was combined for signal processing applications. Ethanol (alcohol) was distinctively separated from the other VOCs using Principal Component analysis (PCA).

The fabrication process involved a 2" silicon wafer which was turned into porous silicon (PS) using electrochemical etching. A very thin layer of TiO_2 was then deposited on this PS. Cr/Au electrodes were formed using metal mask and RF sputtering technique (Fig. 3(a)-(e)). The model and the schematic of the device is shown in Fig, 3(f) and 3(g) respectively.

The characterization of the device shows an underlying porous layer and a top porous TiO_2 layer. Further XRD and Raman spectroscopy results confirmed the structural formation of the materials. Sensing tests were performed in presence of VOCs and the concentration of these were controlled by tuning their vapor pressures. The real time data was acquired using Keithley electrometer and labview program. The sensing response is shown in fig, 4. The optimized response of the TiO_2/PS sensor was obtained at around 100 deg C which was quite close to the one obtained at the room temperature (Fig. 4(a)). As the temperature was increased, a drop in the response was noticed. This sensor had shown maximum response to ethanol and lesser to other VOCs. In comparison to this, its single layer counterparts have either shown more response to acetone or less to ethanol. PS alone had shown higher affinity to acetone in comparison to other VOCs tested (Fig. 4(b)). The sensor response was measured as a change in resistance of the device upon exposure to different analytes. It can be formulated as in the following equation:

$$S(\%) = (R_a - R_0)/R_0 \, x \, 100\% \quad (1)$$

where R_a and R_0 are the resistance change in presence and absence of analyte, respectively.

Sensitivity is calculated as slope obtained from sensor response % versus ppm graph. For TiO2/PS, it was calculated as 8.5 unit/ppm (Fig. 4(c)). Dynamic response for TiO_2/PS sensor measured at 100 deg C is shown in Fig. 4(d)). Response time is defined as the interval between the time when response to analyte starts from 10% (t_s) to 90% (t_{90}) of the response value and is given by $t_{90} - t_s$. Recovery time is defined as the interval between

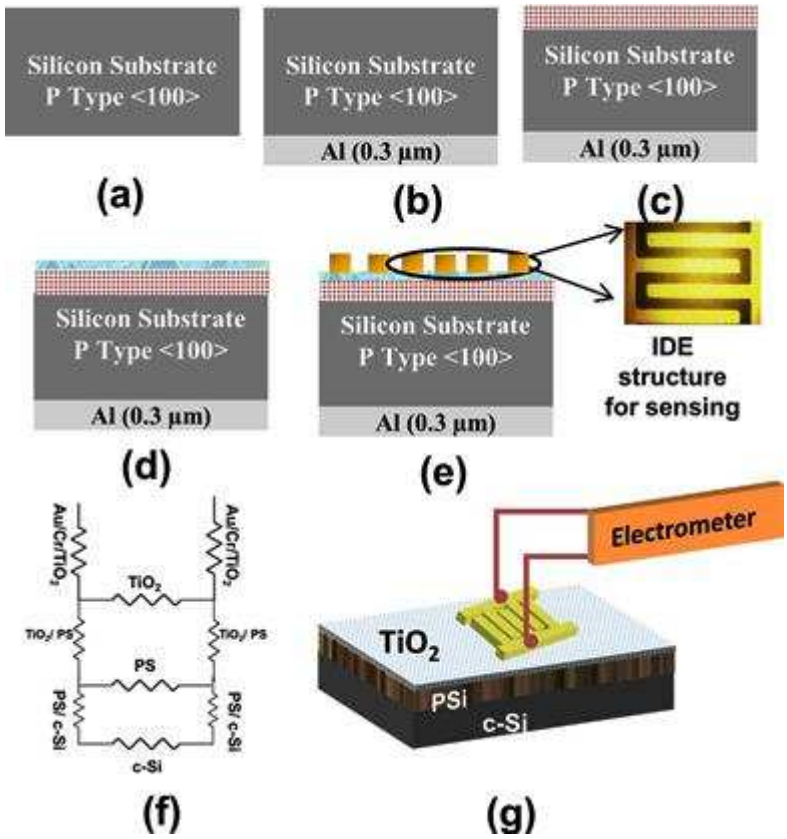

Fig. 3. (a)-(e) Fabrication process flow for making TiO_2/PS devices, (f) model and (g) schematic of the device

the time (t_{st}) when response to analyte stops or saturates to 10% (t_s) of its response value and is given by $t_{st} - t_s$ (Fig. 4(e)). It can be inferred from Fig. 4(f) that the values of response time and recovery time are very high for sensor tested at room temperature, however, as the temperature rose to 100 °C, the values were much lesser and almost constant at higher ppm. Repeatability and ageing studies were performed and the sensor was found to be quite stable. Also, sensors were picked up from various locations of the wafer and their similar response is a proof of scalability of the process and uniformity of the film.

The data from each of the sensor was combined to etimate the vapour concentration using regression model given in the following equation:

$$ppm_{gas} = a_1x + a_2x^2 + \ldots + a_nx^n. \quad (2)$$

Where a_i s are the coefficients and x is the sensor output. These coefficients are obtained using least-squares approximation [12].

The fourth-order and third-order polynomial regression models were found to be fit best to estimate ppm of ethanol and acetone vapors, respectively (Fig. 5(a)). PCA was used for overall view of the data by giving an appropriate visual representation with fewer dimensions in the form of clusters. Fig. 5(b) shows the clear separation obtained from the sensor outputs of all the sensors which directly discriminates between acetone and ethanol vapors. Further IPA, xylene, and benzene can also be separated with clear demarcation boundaries. Inset of Fig. 5(b) depicts the final fabricated sensor bonded on ceramic IC header for interfacing with other circuits. Likewise, a sensor array will have different sensors giving response to various vapour differently. Their data can be combined and estimation of their concentration and separation of a particular vapour from a mix of vapours can be done.

Thus, an E-nose system can easily and accurately identify an analyte from a group of analytes using AI, signal processing and machine learning techniques.

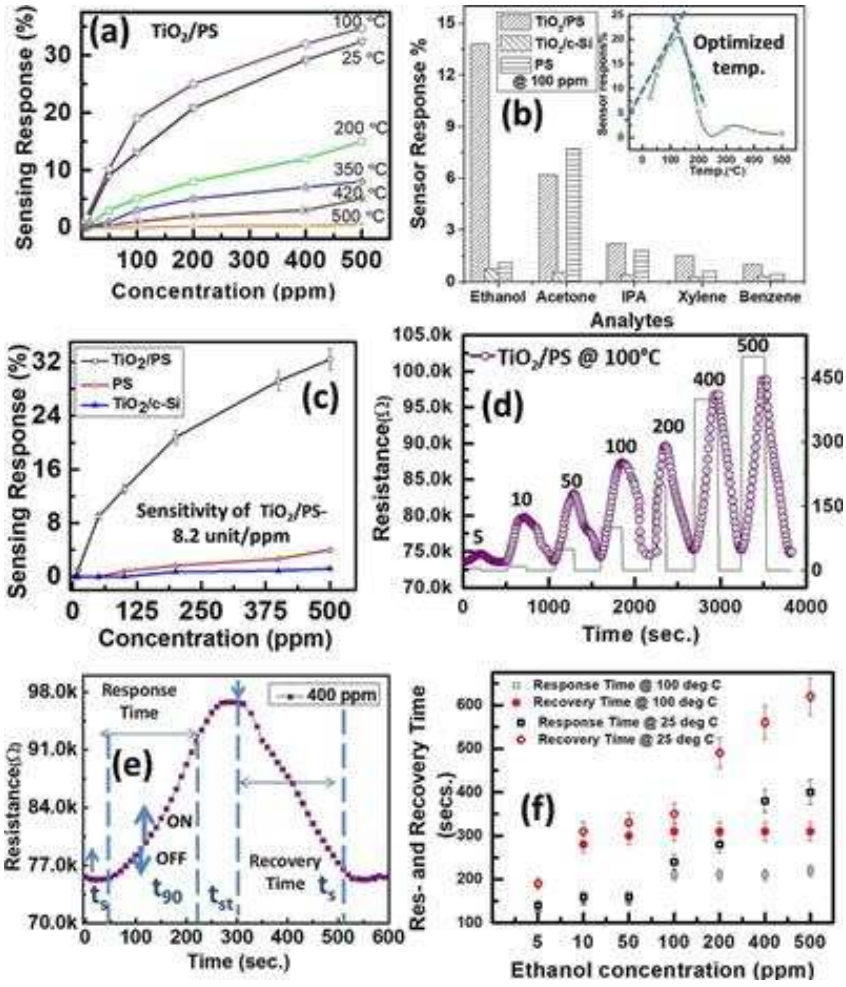

Fig. 4 Sensor response (%) (a) at various concentrations of ethanol and different temperatures, (b) given by all sensors for different analytes (inset shows optimized temperature range), (c) Comparative sensor testing of TiO_2/PS, PS, and TiO_2, error bars present but too small to be visible, (d) dynamic response of TiO_2/PS sensor at 100 °C; the grey dashed lines indicate exposure ON/OFF timings of the analyte, (e) response time and recovery time of sensors at 400 ppm of ethanol, and (f) response time and recovery time of TiO_2/PS at different temperatures.

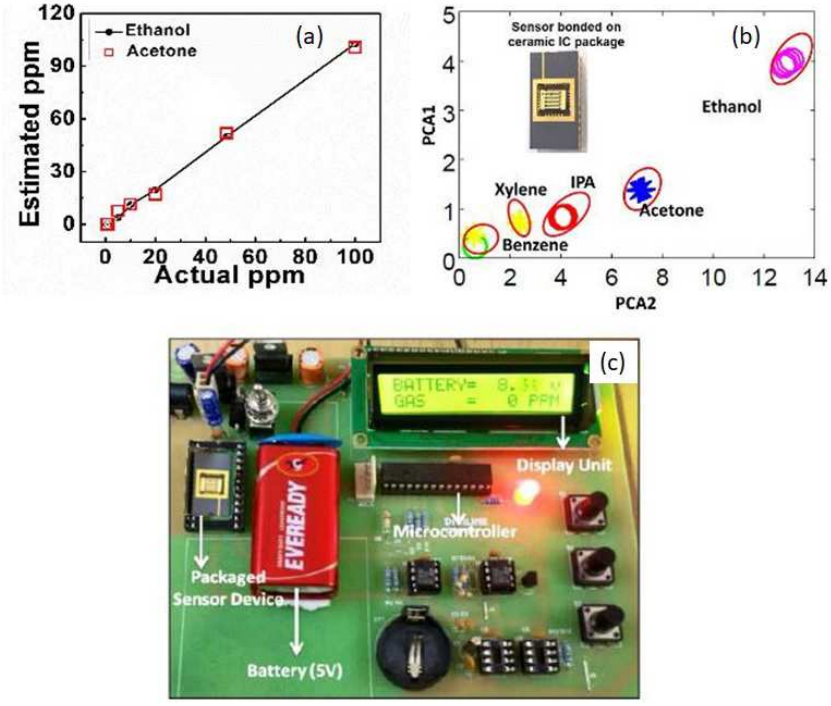

Fig. 5. (a) Estimated and actual ppm for ethanol and acetone. (b) 2-D PCA map showing ethanol vapors discrimination by the sensor, inset shows packaged sensor. (c) Lab prototype of TiO_2/PS sensor

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About the author:

Dr. Saakshi Dhanekar is working as SERB Research Scientist at Centre for Biomedical Engineering, IIT Delhi since February 2019. She was earlier working at Center for Applied Research in Electronics, IIT Delhi, as an INSPIRE Faculty from 2013-18. Prior to this, she has worked as Assistant Professor at Amity Institute of Nanotechnology, Amity University from 2011-12. She received the Ph.D. degree in Electronics from Faculty of Engineering and Technology, Jamia Millia Islamia (Central University), New Delhi, India, in 2012. She is passionately working in the area of gas- and bio-sensors using nanomaterials, MEMS, microfluidics and fabrication to prototype development of devices for social causes. She has filed 5 patents and has several publications in reputed journals to her credit. She is a member of IEEE, Execom member of IEEE Delhi Section 2019 and Young Professional Committee Chair of IEEE Sensors Council 2019. She has been actively involved in promoting Young Professional activities and Women in Sensors in and outside the IEEE community.