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Advances In Electronic - Nose Technologies

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ABSTRACT:

Food quality analysis is one among the most important, complex and challenging discipline in the food sector. Due to its strict interaction with the quality of life it is extremely important to improve the performances of the methods in the field of food quality analysis. Now a days electronic noses have provided a plethora of benefits to a variety of commercial industries, including the agricultural, biomedical, cosmetics, environmental, food, manufacturing, military, pharmaceutical, regulatory, and various scientific research fields using mainly metal oxide(MOS), metal oxide field effect transistor(MOSFET), acoustic (bulk (BAW) and surface (SAW) wave) and polymer sensor. These sensors has unique advantage of providing fast results by identifying, characterizing and quantifying the target analytes of interests present in the flavor emitted by foods without destructing them. All these sensors are highly sensitive and inexpensive. However, these sensors face a numerous problems during operations in different aspects. Among the four sensors, polymer based on nano material may overcome the problems encountered by the environment including the temperature and humidity.

Key Words*: E-nose, metal oxide, acoustic and polymer sensors, gas sensor, aroma detection.*

1. INTRODUCTION

Now, many of the food industries are looking forward for the non destructive food quality analysis techniques. The emerging non destructive food quality analysis techniques are capable of evaluating the finished products quality by analyzing their sensory outputs which may be in the form of flavor, odor, color, texture and taste. Electronic Nose (E - nose) seems to be a new electronic device which can report on the finished products quality by analyzing the head space gas which is generally composed of the volatile organic chemicals that contributes to the typical flavor for the different food materials.

The term "electronic nose" was coined in 1988 by Gardner and Bartlett, (1994) who later defined it as "an instrument which comprises an array of electronic chemical sensors with partial specificity and appropriate pattern recognition system, capable of recognizing simple or complex odors".

2. HISTORICAL MILESTONES IN DEVELOPMENT OF E- nose

The first real tool for measuring aromas was developed by Hartman in 1954. The sensing element was a microelectrode, a simple platinum wire of 0.8 mm in diameter, which measured the flow of current by a sensitive milli voltmeter. Metal oxide semiconductor gas sensors (MOS) were first used commercially. In 1960's MOS were used as household gas alarms in Japan under the names of Taguchi or Figaro. Moncrieff (1961) worked on the concept that different coatings materials, such as polyvinyl chloride, gelatin, and vegetable fats could be capable of providing different and complementary data for the discrimination of simple and complex aromas. His studies were limited to the use of a single temperature-sensitive resistor, but postulated that an array with six thermistors, provided with six different coatings, could discriminate large numbers of different aromas. The principle of BAW sensors was introduced by King in *1964* with his Piezoelectric Sorption Detector. Buck *et al.* (1965) studied the modulation of conductivity as an answer to differentiating aromas bouquets, while Dravnieks and Trotter (1965) used the modulation of contact potential to monitor aromas. These studies have been considered as a first approach to aromas evaluation because of the lack of analytical instruments. The hydrogen sensitive PdMOS (palladium metal oxide semiconductor) device was developed in 1973 by a group of Swedish researchers (Lundström *et al.,* 1975, 1990 and 1993). The first gas sensor based on a SAW oscillator was introduced by Wohltjen and Dessy (1979). Conducting organic polymer (CP) sensors have been under development for approximately 10 years *(Hodgins, 1997)* since 1990's. CP sensors rely on changes of resistance by the adsorption of gas.

Table: 1 Sensor Types, sensitivity and their detection principle

S.No	Sensor Type	Sensitive Material	Detection Principle
1	Metal Oxides Semi-conducting (MOS, Taguchi)	Doped semi-conducting metal oxides (SnO_2, GaO)	Resistance change
2	Metal Oxide Semiconductor field-effect sensors (MOSFET)	Catalytic metals	Electric field change
3	Conducting polymer sensors	Modified Conducting Polymers	Resistance change
4	Acoustic sensors: Surface and Bulk acoustic wave (SAW and, BAW)	Organic or inorganic film layers	Mass change (frequency shift)

3. BIOLOGICAL AND ARTIFICIAL OLFACTION

The human olfactory system is more complex and contains thousands of receptors that bind odor molecules and can detect some odors at parts per trillion levels *(Breer,1997)* and include between 10 and 100 million receptors *(Deisingh et al., 2007).* Apparently some of the receptors in the olfactory mucus can bind more than one odor molecule and in some cases one odor molecule can bind more than one receptor. This results in a mind-boggling amount of combinations that send unique signal patterns to the human brain. The brain then interprets these signals and makes a judgment and/or classification to identify the substance consumed, based in part, on previous experiences or neural network pattern recognition.

The E - nose often consists of non-selective sensors that interact with volatile molecules that result in a physical or chemical change that sends a signal to a computer which makes a classification based on a calibration and training process leading to pattern recognition (Fig. 1). The non-selectivity of the sensors results in many possibilities for unique signal combinations, patterns or fingerprints (Baldwin *et al.,* 2011).The greatest advantage of using E - nose is that it can be calibrated to be reliably consistent and can give objective data for important functions like quality and safety control. These instruments can also test samples that are unfit for human consumption. The disadvantage of the E - nose is that they are affected by the environment including temperature and humidity, which can cause sensor drift.

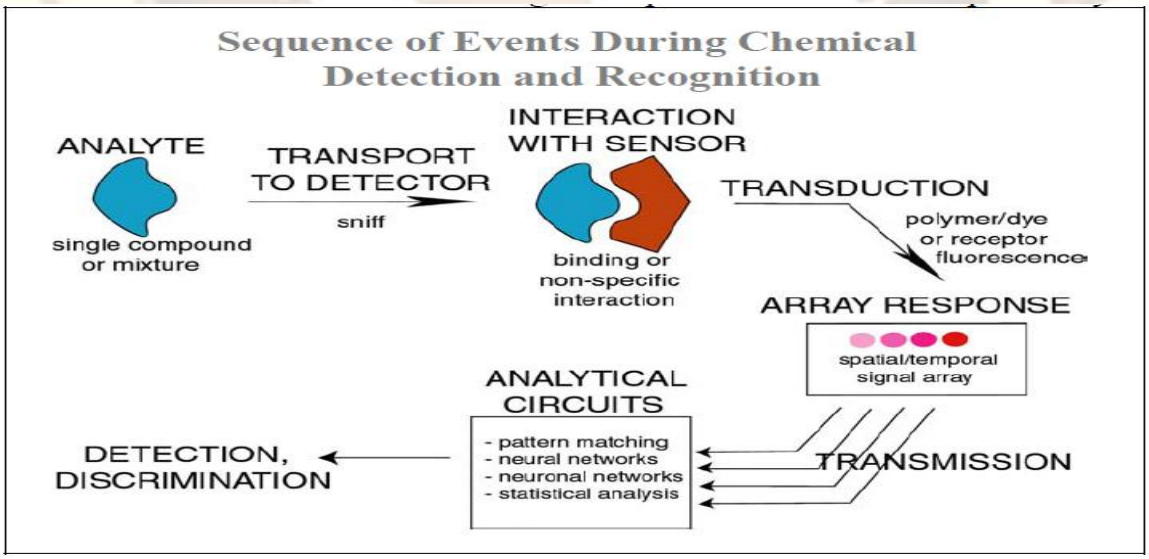

Fig: 1 Sequence of events that occurs in order for chemical recognition in air phase compounds to be detected in both biological and artificial olfactory system *(Kauer et al., 2003)*

4. E - nose SENSORS

All types of sensors exhibit interactions with the gas to be measured so that a series of physical and/or chemical interactions occurs when volatile compounds flow over the sensor. A dynamic equilibrium develops as volatile compounds are constantly being adsorbed and desorbed at the sensor surface (*Shiers,P., 1995*).The ideal sensors to be

integrated in an electronic nose should fulfill the following criteria (*Demarne, et al*. 1992, *Mari and Barbi* 1992, *Bartlett, et al*. 1993, *Hodgins,*1995 and 1997): high sensitivity towards chemical compounds; low sensitivity towards humidity and temperature; selectivity to respond to different compounds present in the headspace of the sample; high stability; high reproducibility and reliability; short reaction and recovery time; durable; easy calibration; easy to process data output. Various kinds of gas sensors are available, but only four technologies are currently used in commercialized electronic noses: metal oxide semiconductors (MOS); metal oxide semiconductor field effect transistors (MOSFET); conducting organic polymers (CP); piezoelectric crystals (bulk acoustic wave = BAW). Others such as fibre optics *(Dickinson, et al.1996, Nakagawa, et al.1997, Eguchi, 1992 and Sutter and Jurs 1997),* electrochemical *(Mari, and Barbi 1992 and Baltruschat, et al.1997)* are still in the developmental stage and may be integrated in the next generation of E - Noses. Such sensors can be divided into two main classes: hot (MOS, MOSFET) and cold (CP, SAW, BAW). The former operate at high temperatures and are considered to be less sensitive to moisture with less carryover from one measurement to another. Therefore, they should offer the best ratio of drift and lifetime to sensitivity (*Shiers, 1995*).

4.1Metal oxide semiconductor sensors (MOS)

The metal oxide coating may be either of the n-type (mainly zinc oxide, tin dioxide, titanium dioxide or iron (III) oxide) which responds to oxidizing compounds, or of the p-type (mainly nickel oxide or cobalt oxide) which responds to reducing compounds (*Mielle, 1996, Huheey, 1983 and Greenwood and Earnshaw 1988*). N-type semiconductors increase the reactivity with oxidizing molecules whereas excited p-type promotes reactions with reducing compounds (*Huheey, 1983 and Greenwood and Earnshaw 1988*).

The film deposition technique further divides each sensor type into thin (6–1000 nm) or thick (10–300 μm) film MOS sensors. Film deposition includes physical or chemical vapour deposition, evaporation, or spraying for thin films, and screen printing or painting for thick films (*Demarne and Sanjines* 1992). The thin film devices offer a faster response and significantly higher sensitivities, but are much more difficult to manufacture in terms of reproducibility. Therefore, commercially available MOS sensors are often based on thick film technologies. Due to the high operating temperature (typically 200–650°C), the organic volatiles transferred to the sensors are totally combusted to carbon dioxide and water on the surface of the metal oxide, leading to a change in the resistance (*Wünsche et al., 1995*).

The mechanism is based on an oxygen exchange between the volatile molecules and the metal film. To shift the selectivity of a metal oxide film towards different chemical compounds the film is doped with noble catalytic metals (e.g. platinum or palladium), or the working temperature is changed within the range of 50–400°C. Although the selectivity is also greatly influenced by the particle size of the polycrystalline semiconductor, the MOS sensors are usually less selective than other technologies such as CP, BAW, SAW or MOSFET. However MOS sensors are extremely sensitive to ethanol (*Mielle*, 1996 and *Wünsche et al, 1995*).

4.2Metal oxide semiconductor field-effect transistor sensors (MOSFET)

A MOSFET sensor comprises three layers, a silicon semiconductor, a silicon oxide insulator and a catalytic metal (usually palladium, platinum, iridium or rhodium), also called the gate. A normal transistor operates by means of three contacts, two allow the current in (source) and out (drain), and the third acts as the gate contact that regulates the current through the transistor. In the MOSFET transistor, the gate and drain contacts are shortcut, giving a diode mode transistor with convenient electronics for operation, characterized by an IV-curve. The applied voltage on the gate and drain contact creates an electric field, which influences the conductivity of the transistor. When polar compounds interact with this metal gate, the electric field, and thus the current flowing through the sensor, is modified. The recorded response corresponds to the change of voltage necessary to keep a constant preset drain current (*Lundström et al., 1992 and 1993*). The metal oxide semi- conductor field-effect transistor (MOSFET) sensors rely on a change of electrostatic potential.

As in the coating of MOS sensors, the gate structure of a MOSFET sensor is either a thick, dense metal film (100–200 nm) or a thin, porous metal film (6–20 nm). The thick, continuous metal gate responds almost exclusively to molecules that dissociate hydrogen on the catalytic metal surface. It is implicitly assumed that the insulator is not exposed to the ambient molecules. The dissociated hydrogen atoms diffuse within microseconds through the metal causing a dipole layer at the metal–insulator interface, leading to a potential change in the transistor. Detection of molecules such as ammonia or carbon monoxide is not possible with such a layer since no hydrogen atoms are released (*Spetz et al., 1992*). However, it transpired that the latter compounds respond well when the metal gate is thinned. The selectivity and sensitivity of MOSFET sensors may be influenced by the operating temperature (50–200°C), the composition of the metal gate, and the microstructure of the catalytic metal (*Lundström et al, 1975 and 1993*). MOSFET sensors, like MOS sensors, have a relatively low sensitivity to moisture and are thought to be very robust. However, high levels of manufacturing

expertise are necessary to achieve good quality and reproducibility. (*Schaller et al.,1998*).

4.3 Conducting organic polymer sensors (CP Sensors)

Conducting organic polymer (CP) sensors are like MOS sensors, rely on changes of resistance by the adsorption of gas. These sensors comprise a substrate (e.g. fibre-glass or silicon), a pair of gold-plated electrodes, and a conducting organic polymer such as polypyrrole, polyaniline or polythiophene as a sensing element. The polymer film is deposed by electrochemical deposition between both electrodes previously fixed to the substrate (*Mielle, 1996 and Amrani et al., 1995*).

As the conducting polymer is grown out of a solution, the deposited film contains cation sites balanced by anions from the electrolyte and the solvent residue (*Wünsche et al.*, 1995 and *Hodgins et al., 1995 and 1997).*The cation sites probably consist of polar ions or bipolar ions which are small regions of positive charge in the polymer chain providing mobile holes for electron transport.

When a voltage is passed across the electrodes, a current passes through the conducting polymer. The addition of volatile compounds to the surface alters the electron flow in the system and therefore the resistance of the sensor (*Shiers, 1995).* The volatiles may interact at least with (i) the polymer itself, (ii) the counter ion, or (iii) the solvent (*Hodgins, 1997).* Therefore, good selectivity in the CP sensors may be achieved by altering one of these parameters or the electrical growth of the polymer coating.

In general, these sensors show good sensitivities, especially for polar compounds. However, their low operating temperature (< 50°C) makes them extremely sensitive to moisture (*Shiers, 1995*). Although such sensors are resistant to poisoning (*Zannoni, 1995*), they have a lifetime of only about 9–18 months. This short life may be due to the oxidation of the polymer, or to exposure of the sensor to different chemicals that may develop contact resistances between the polymer and the electrodes. Unlike MOS sensors, the CP sensors are not yet widely marketed, and laboratory-scale manufacturing renders them expensive. The difficulty of producing good batch- to-batch reproducibility and a pronounced drift of the response are their main disadvantages (*Mielle, 1996*)

4.4 Acoustic sensors

Piezoelectric sensors are based on a change of mass, which may be measured as a change in resonance frequency. These sensors are made of tiny discs, usually quartz, lithium niobate ($LiNbO_3$) or lithium tantalite ($LiTaO_3$), coated with materials such as chromatographic stationary phases, lipids or any non-volatile compounds that are chemically and thermally stable (*Guilbault and Jordanel*, 1988, *Nieuwenhuizen and Nederlof*, 1992, *Holmberg* , 1997).

When an alternating electrical potential is applied at room temperature, the crystal vibrates at a very stable frequency, defined by its mechanical properties. Upon exposure to a vapour, the coating adsorbs certain molecules, which increases the mass of the sensing layer and hence decreases the resonance frequency of the crystal. This change may be monitored and related to the volatile present (*Hodgins*, 1997). The crystals may be made to vibrate in a bulk acoustic wave (BAW) or in a surface acoustic wave (SAW) mode by selecting the appropriate combination of crystal cut and type of electrode configuration (*Wünsche et al*, 1995).

BAW and SAW sensors differ in their structure. BAW are 3-dimensional waves travelling through the crystal, while SAW are 2-dimensional waves that propagate along the surface of the crystal at a depth of approximately one wavelength (*Nieuwenhuizen et al, 1992* and *Holmberg , 1997*). These devices are also called 'quartz crystal micro-balance' (QCM or QMB) because, similar to a balance, their responses change in proportion to the amount of mass adsorbed. BAW sensors vibrate with a frequency of 10–30 M Hz. Their thin coating (1 µm–10 nm) is deposited by spin coating, airbrushing or inkjet printing (*Wünsche et al.*, 1995, *Mielle*, 1996, and *Holmberg*, 1997).

The manufacturing technique includes photolithography and airbrushing, and is fully compatible with planar integrated circuits, fabrication, especially planar silicon technology. This enables SAW structures and conditioning circuits to be incorporated on the same silicon substrate, resulting in robust and inexpensive SAW sensors (*Caliendo et al.*, 1992).

Since piezoelectric sensors may be coated with an unlimited number of materials, they present the best selectivity (*Mielle*, 1996 and *Hodgins*, 1997). However, the coating technology is not yet well controlled, which induces poor batch- to-batch reproducibility (*Mielle*, 1996 and *Wünsche et al*, 1995). SAW sensors, though limited by the noise caused by their high operating frequency, are more sensitive than BAW sensors. However, both sensors require a higher concentration of volatiles to elicit response levels comparable to other sensor types (*Mielle, 1996* and *Hodgins, 1997*). The difficulty of integrating BAW and SAW sensors into an electronic nose resides in the more complex electronics and their high sensitivity to disturbances such as temperature and humidity fluctuations (*Mielle*, 1996 and *Wünsche et al.*, 1995).

Table: 2 Summary of advantages of Electronic Nose Sensor Type

S.No	Sensor Type	Advantages	Disadvantage
1	Metal oxides semi-conducting (MOS)	Very high sensitivity, limited sensing range and rapid response.	High temperature operation, high power consumption, sulfur and weak acid poisoning.
2	Metal oxide field-effect Sensors (MOSFET)	Small sensor size and inexpensive operating costs	Requires environmental control, baseline drift and low sensitivity to ammonia and carbon dioxide.
3	Conducting polymer Sensors(CP)	Ambient temperature operation, sensitive to many volatile organic compounds, short response time, inexpensive and resistive to sensor poisoning	Sensitive to humidity and temperature and sensor life is limited.
4	Acoustic sensors: surface and bulk acoustic wave (SAW and BAW)	High sensitivity, good response time, diverse sensor coatings, small, inexpensive, sensitive to virtually all gases	Complex circuitry, temperature sensitive, specificity to analyte groups affected by polymeric-film sensor coating

5. CONCLUSION

Although these sensors are sensitive and inexpensive, they face different kinds of problems, especially drift, noise, repeatability, environmental influence (temperature and humidity), poisoning and non - linearity in sensor response. Among the four categories discussed, the application of suitable polymer as a base material may help to overcome these problems. E - nose, with a nose – on – chip which is a single computer chip containing the polymer sensors and processing compounds for detection of flavors will suffice the requirements of various food industries in monitoring their products.

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