CHAPTER 2

OVERVIEW OF SMELL SENSOR SYTEMS

Here importance of smell in our life, process of smell sensing, basics of electronic nose systems, type of odor sensors, available sensor systems, applications etc are discussed.

2.1 The Sense of Smell and its Role in Our Life:-

The sense of smell has a fundamental influence in human development and social interactions among human as well as between human and animal kingdom. The importance of aroma qualities can be seen in wide range of applications like perfumes, wines, cuisines, colognes added to personal healthcare products, scents applied to product packaging. Throughout human history spices have been used to enhance the flavor of foods and air as we know that spices were among the most valued commodities for trade in ancient times,.

In order to improve product appeal, quality and consistency so that consumers quickly identify unique scents with individual brands, the olfactory sense has become a key element in the development of various commercial industries that manipulate the aroma properties of their products. Therefore, aroma characteristics have contributed a lot to the value of many commercial products. Since product consistency is essential for maintaining consumer brand recognition and satisfaction, the research and quality control of aroma characteristics during production has become of utmost importance in industrial production operations.

Although the olfactory sense is very important to the mankind but the sense of smell in human is often considered the least refined and far less sensitive than that of other animals. For example, the human nose has only about a million aroma receptors that work in tandem to process a particular smell whereas dogs have about 100 million receptors that obviously can distinguish smells at least 100 times more effectively than the average human [16].

2.2 Human Sensor System

Process of detection of smell in human nose is shown in Figure 2.1[17]. As we breathe in air through our nostrils, we inhale airborne chemical molecules, which are detected by the 10 million to 20 million receptor cells embedded in the olfactory membrane of the upper nasal passage.

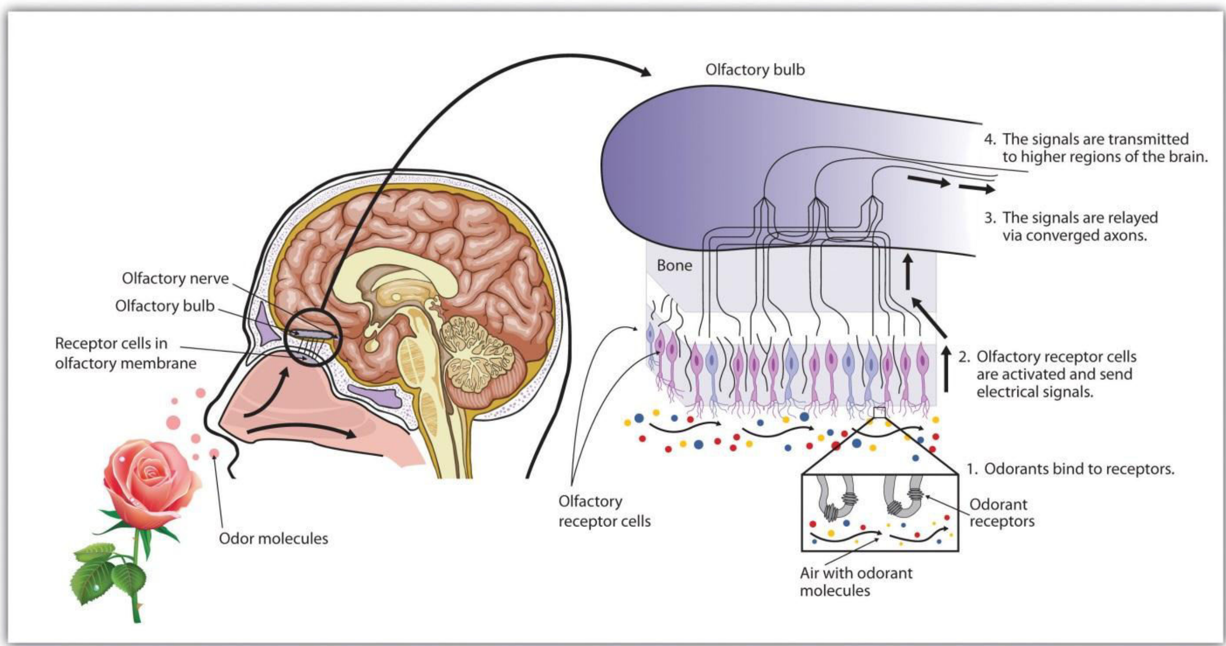

Figure 2.1 Human nose smell detection process

The olfactory receptor cells are topped with tentacle-like protrusions that contain receptor proteins. When an odor receptor is stimulated, the membrane sends neural messages up the olfactory nerve to the brain for distinguishing. Biological nose is very adaptive but unlike the electronic nose, saturation can happen and that is one of the reasons why it operate only for a short periods of time. In Figure 2.2 we can see the analogy between generic architecture of Electronic nose [18] and Mammalian olfactory system [19] in which array of sensors are working like receptor cells and signal processing/pattern recognition unit is like Mammal brain.

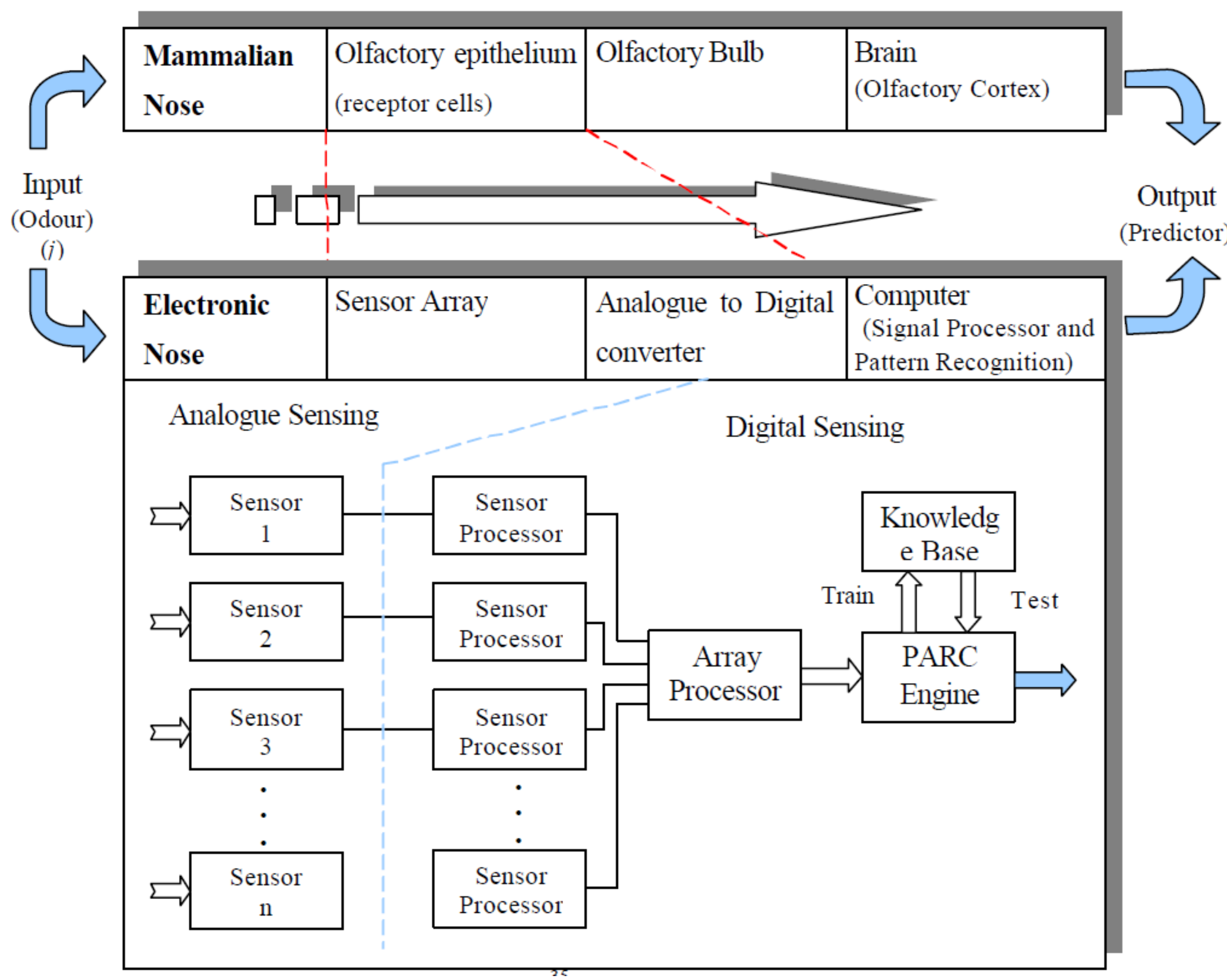

Figure 2.2 Generic architectures of Electronic Nose and Mammalian Olfactory System

It can identify variety of odors along with a detection of some specific molecules but it cannot detect some other simpler types of molecules. Another drawback is that in a biological system, infection can take place that can affect the ability of smelling. Also smelling can be associated with human experience and memory. In biological nose there are millions of self-generated receptors with different selectivity classes of range from 10 to 100 [20]. Let's take another Figure 2.3 [21] that shows the biological and artificial sensing systems.

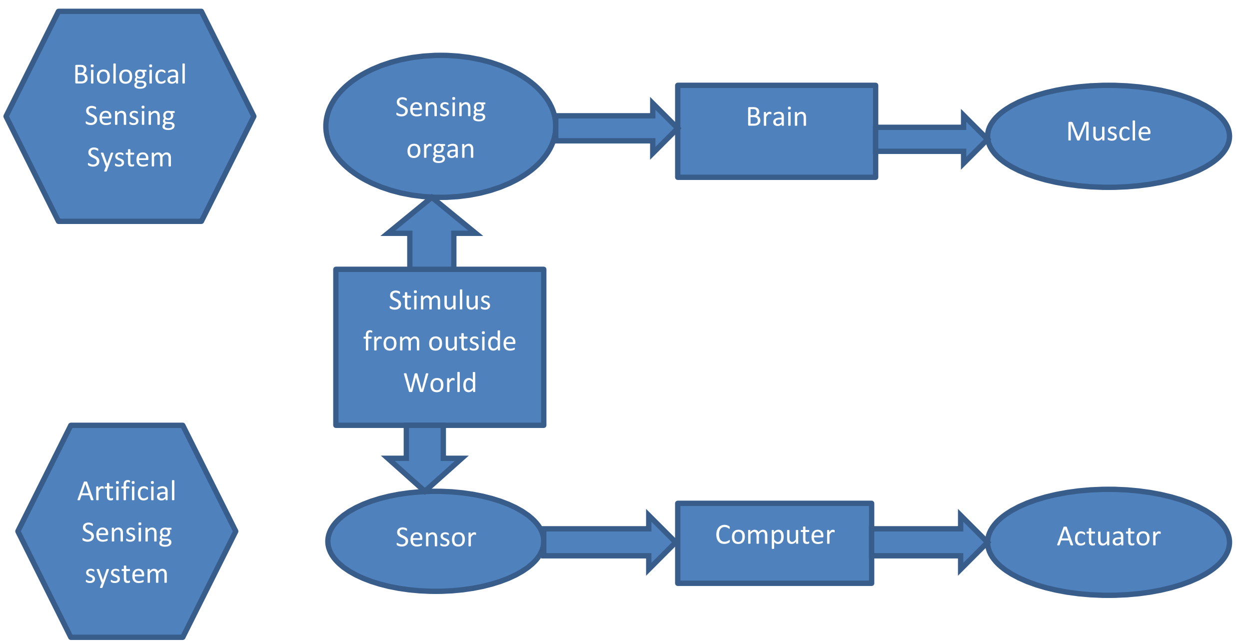

Figure 2.3 Processes of Reception and Action in Biological and Artificial Sensor Systems

As can be seen that there are basically three blocks in each sensing system- first is sensor, second- processor (brain or computer) and the third- action taking device. For example if a food has good smell it will be eaten otherwise thrown.

2.2.1 Sensitivity of Human and Dog Sensing Systems

Now compare the sensitivity of human and dogs smell sensing system for different types of acids exist in different types of food and non-food items [22].

In the Table 2.1 the threshold in terms of molecules/ml water is given for odor substances of human and dogs. It can be seen that for dog nose the threshold is almost million times lower than human nose for each of the acids.

Table 2.1.Threshold for odor substances in dogs and humans

S.N.	Odor substances	Dogs($\times10^3$molecules/ml water)	Humans($\times10^{10}$molecules/ml water)
1.	Acetic acid	500	5000
2.	Propionic acid	250	42
3.	Valeric acid	35	6
4.	Butyric acid	9	0.7
5.	Caproic acid	40	20
6.	Caprylic acid	45	20

2.3 Artificial Nose System

In smell sensor or electronic nose due to absorbed gas with material in gas sensor chemical reactions take place due to which the electric resistance or other properties like voltage, frequency etc changed which generates an electrical signal that is processed to recognize the odor. For example enzyme sensors (ion selective electrode) are very powerful and useful for detecting a specific chemical substance with high sensitivity and selectivity [23]. There is a diversity in sensitivity and selectivity in different biological sensing systems e.g. dogs have 10^6 to 10^8 times higher sensitivity than humans for many chemicals and insects have very high selectivity and sensitivity to a particular molecule pheromone.

The sense of smell has two opposite properties [24] first one is recognition of smell without separating the chemical components of the gas, and detection of one or a few molecules by one olfactory cell on average with extremely high selectivity and sensitivity.

At present nobody has the answer of the question: whether the reception mechanism is same for these two properties or not [15]. Response to odorants are obtained not only in smell systems but also in non-smell systems such as frog taste cells and neuro-blastoma cells which contain no specific receptor proteins for odorants. Depending upon material used, developed odor sensors are of two types. First are the Biomimetic in which the odor molecules are absorbed in hydrophobic portion of a bilayer lipid membrane and second ones are the non-biomimetic in which lipid membrane is not used. Because of relatively low sensitivity and discrimination capabilities of the human nose, coupled with common occurrence of olfactory fatigue, there is a need of electronic instrument with sensors capable of performing repeated detections with precision without fatigueness.

2.3.1 How Odor Reception Takes Place

Generally in a sensor system receptors for different molecules (nonspecific to an odorant) are used. Figure 2.4[25] shows different molecules having different

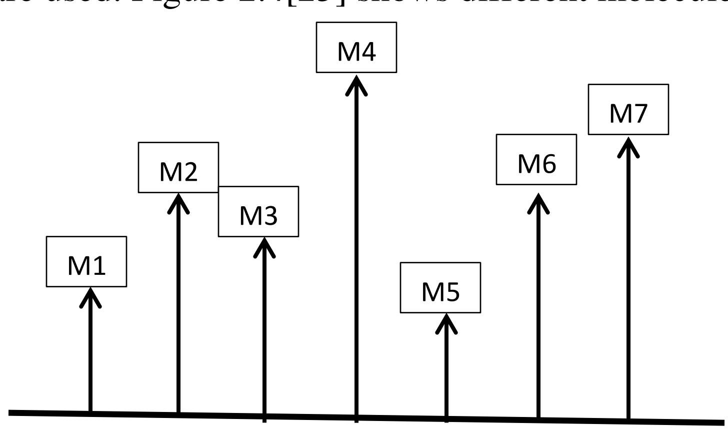

Figure 2.4 Detected Molecules with Different Concentrations

molecular weight, retention time etc., that can be detected by a sensor; but still we cannot get name of particular odor because each odor has different molecules with different concentrations as shown in the Figure 2.5[25].

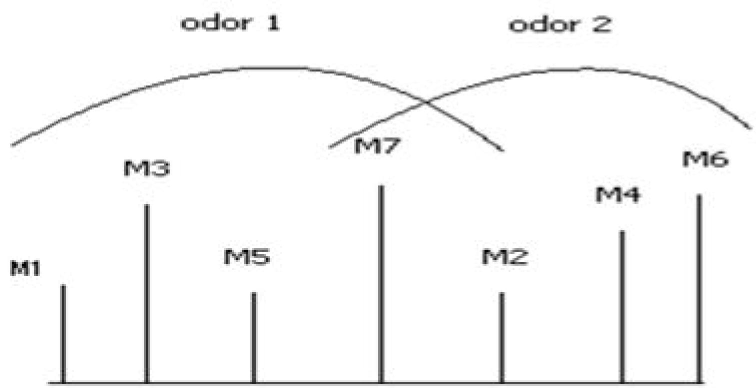

Figure 2.5 Odors consisting of Different Molecules

Since sensitivity to each molecule is different so in practice we use number of sensors. For example a sensor S1, it detects M1 but M2 is also detected by it so to subtract the effect of M2, S2 is used that may detect M3, M4 too so a third sensor S3 is required as shown in Figure 2.6[26].

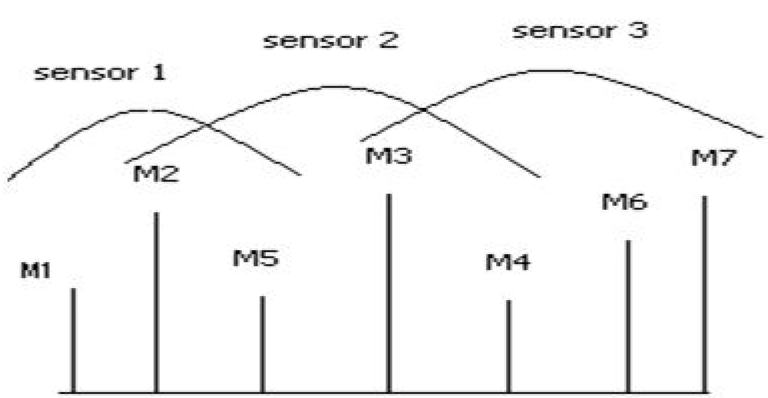

Figure 2.6 Sensing Characteristics of Sensors

Because of distinct pattern of response across the sensors for different odorant an unknown odor from the pattern of sensor responses can be distinguished and identified. Each sensor in the array has a unique response profile for the different odorants under test and the unique pattern of response across all sensors in the array is used to identify and/or characterize the odor.

2.3.2 Smell Sensing

In a typical electronic nose, an air sample is pulled by a vacuum pump through a tube made of plastic or stainless steel into a small chamber housing the electronic sensor array where exposure of the sensors to the odorant take place, producing a transient response as the VOCs interact with the surface and bulk of the sensor's active material.

Initially each sensor should be driven to a known state by having clean, dry air or some other reference gas passed over its active elements. Depending on the type of sensor a steady-state condition is reached in a few seconds to a few minutes. During this interval, the responses of sensors are recorded and send to the processing unit. A washing gas such as an alcohol vapor is applied to the array of sensors for a few seconds to a few minutes to remove the odorant mixture from the surface and bulk of the sensor's active material. This washing step can be skipped in some designs. To prepare the sensor array for a new measurement cycle, the reference gas is applied again. The duration of odorant application to the sensor array is called the response time and the duration of washing and application of reference gases is called as recovery time [20].

2.4 Development of the Electronic Nose: A Historical View

In 1920s Zwaardemaker and Hogewind [27] have done a study involving aroma measurements for the first time in the history that focused on measuring the electricity of a fine spray of water. They have stated that spray-electricity can be increased by the addition of volatile substances to the water that could be used to detect the presence of aromatic compounds by means other than through the sense of smell. In 1954 Hartman [28] has developed the first real tool for measuring aromas. He used microelectrode (a simple platinum wire of 0.8 mm in diameter)as sensing element, to measure the flow of current by a sensitive mille-voltmeter. He also proposed the idea of several different coated sensitive elements on electrode to get differential responses to different chemical compounds [29].

Moncrieff [30] worked on the discrimination of simple and complex aromas and found that different coatings materials like polyvinyl chloride, gelatin, and vegetable fats are capable of providing different and complementary data. He had postulated that an array with six thermistors having six different coatings, could be able to discriminate the large numbers of different aromas. In 1965 Buck et al. [31] have studied the modulation of conductivity of sensors for different aromas bouquets, in the same year Dravnieks and Trotter [32] used the modulation of contact potential of sensors to monitor the odors. Because of the lack of analytical instruments the above two studies have been considered only a first step to odor evaluation. However, about 20 years later (1982), studies of Persaud and Dodd [33] and Ikegami and Kaneyasu [34] have given the idea of an electronic-nose an intelligent instrument having chemical sensor array for aroma classification. By that time, the development of computers and different types of electronic sensors made it conceptually possible to get an electronic sensor device capable of imitating the mammalian olfactory system.

2.4.1 Origin of Electronic Nose:-

Gardner and Bartlett were the first persons who coined the term “electronic nose” in 1988, who later defined it as an electronic instrument consists of an array of chemical sensors with partial specificity and an appropriate pattern recognition system, capable of discriminating the simple or complex odors [35]. In 1991, during a session of the North Atlantic Treaty Organization (NATO) that was entirely dedicated to the topic of artificial olfaction, scientific interest in the use of electronic noses was sanctioned by the first advanced workshop on chemosensory information processing. From 1991, interest in biological sensors technology has increased dramatically because of various research articles on the subject and industrial efforts to develop and improve sensor technologies and equipment's of better performance capabilities, diverse sensitivities with ever-expanding applications.

2.4.2 Electronic Nose Terminology

The electronic nose was born from the real need in the food industry for objective, automated quality-monitoring sampling systems that can characterize the odor of a product and determine if the production is running according to standards without the need of a human sensory panelist, or time consuming analytical methods and data interpretation. An electronic nose does not exactly replicate the mammalian nose. Anything that smells cannot be a good electronic-nose application and anything that can be measured by an electronic nose has a smell.

An electronic nose has no problem in detecting gases like carbon monoxide and hydrogen cyanide that a mammalian nose cannot, as long as they are in sufficient quantity. Also, the electronic noses not always can succeed in detecting all of them. Mammals have capability of detecting certain gases with low limit of detection with high receptor specificity, while electronic noses do not show the same performance. Thus an odor description from an electronic nose is not always possible. A mammalian nose focuses only on the odorant volatiles and an electronic nose analyses the whole volatile species whether odor and odorless. Because of these differences the term electronic nose should not be stressed too much. Some authors [36] point out the fact that the two noses do not work in the same way and do not detect the same chemicals so suggest the use of the term electronic nose between quotation marks. They have suggested the use of terms like: aroma sensor, gas sensor, flavor sensor and odor sensor, which does not suggest such strong links with the human olfaction.

2.4.3 Short Description of the Electronic Nose Concepts and Modules

An electronic nose is a machine that is designed to detect and discriminate among complex odors using a sensor array. The sensor array consists of broadly tuned (non-specific) sensors that are treated with a variety of odor sensitive biological or chemical materials. An odor stimulus generates a characteristic fingerprint (or smell print) from the sensor array. Patterns or fingerprints from known odors are used to construct a database and train a pattern recognition system so that unknown odors can subsequently be classified and identified. This is the classical concept of an e-nose; however, in recent years, the classical sensor types used for e-noses have been enhanced and complemented by other technologies introduced in this field. Nevertheless, in a broader sense, electronic nose instruments are composed of three elements: a sample handling system, a detection system, and a data processing system.

2.5 Components of e-Nose Systems

Gardner and Bartlett [35] have given the basic structure of an electronic-nose device and provided a list of necessary components, given in Figure 2.7 as follows:-

1. An aroma delivery system that transfers the volatile aromatic molecules from the source material to the system of sensor array

2. A chamber where sensors are fixed in which usually has fixed temperature and humidity to avoid unwanted effect on the aroma molecules adsorption
3. An electronic circuit that converts the chemical reaction effect into an electrical signal, amplifies and conditions it
4. An analog to digital converter that converts the sensor signal (analog) to equivalent digital signal, and
5. A microprocessor that reads the digital signal and after statistical analysis for sample classification, recognition is done and output is displayed

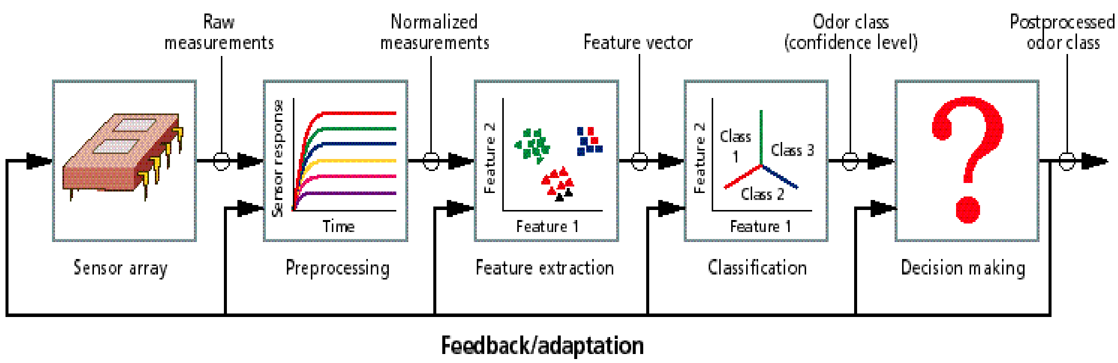

Figure 2.7 Olfactory Signal Processing and Pattern Recognition

2.6 Different type of Sensors

2.6.1 Metal Oxide Sensors

Metal oxide sensors (MOS) have semi conducting sensing elements made from a metal oxide film. Generally tin, zinc, titanium, tungsten, iridium and mixed metal oxide sensors are available. They are commercially available at low cost and have been used in e-nose systems. The basic structure of a MOS sensor is shown in Figure 2.8.

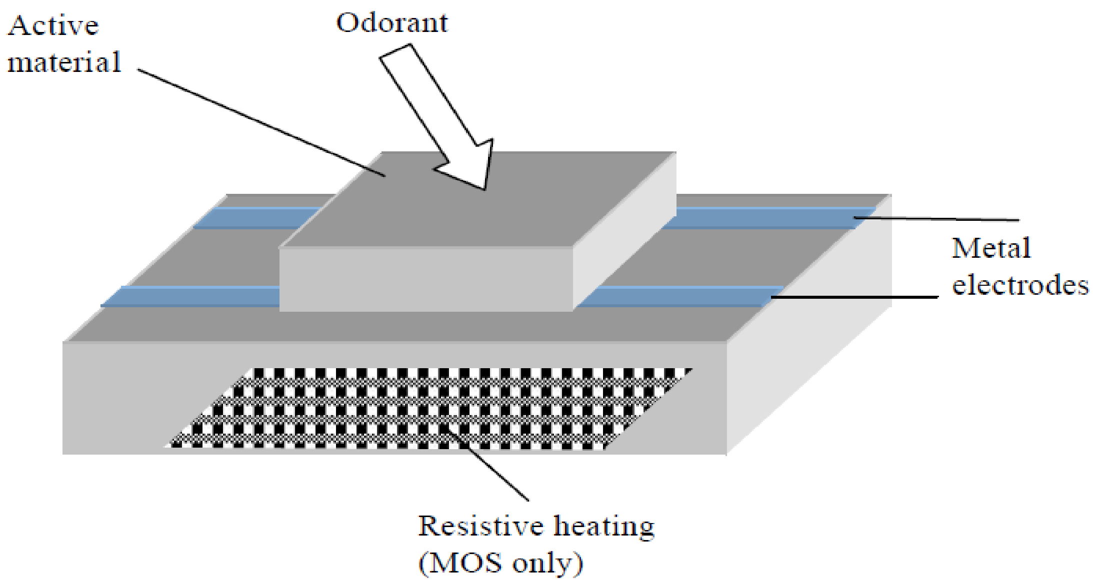

Figure 2.8 Conductive MOS sensor

The semi conducting material is placed in the base of the sensor between two metal electrodes and a resistive heating element (usually platinum). It has diameter of 15mm and length of 15mm and fairly robust. On the surface of the semi-conducting element, a redox reaction triggered due to presence of VOCs that change the conductivity of the sensor. This change of conductivity depends on concentration of the VOCs and measured to recognize the organic compounds. So, oxygen is required for the sensors to operate.

By the doping of metal oxide with a material such as palladium or platinum the selectivity of the sensors can be modified. A sensor responds to a range of gases that create problems in analyzing their output [37]. The sensitivity of the sensors depends largely on the operating temperature in the range of 300°C to 550°C [38], because of this power consumption of the sensors are quite high usually 800mW. The sensitivity range is between 5 and 500ppm of gas.

2.6.2 Conducting Polymer Resistive Sensors

Typically it is constructed by bridging two electrodes, generally 10 – 20 μm apart [39], with a semi conducting polymer film that forms a chemo-resistor. The commonly applied polymers are based on pyrrole, tiophene or aniline. In Figure 2.9 the structure of a conducting polymer sensor is shown. When the polymers are exposed to different chemical compounds or molecules it get bonded with the polymer backbone that affects the transfer of electrons in the polymer chain finally change the conductivity of sensor.

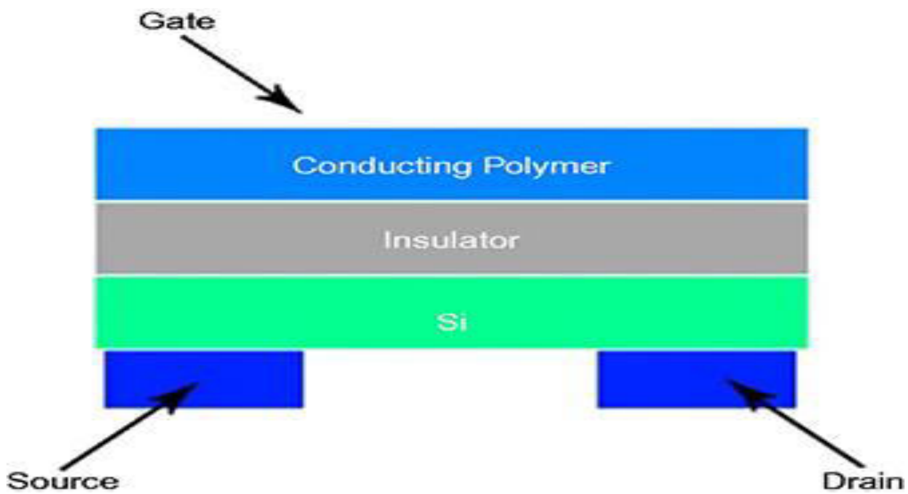

Figure 2.9 Conductive Polymers Resistive Sensor

This change can be measured, generally by applying a current and recording the resistance, and used to identify the species present. The resistance of the final device depends on the resistivity of the polymer used, the geometry and the film thickness. The selectivity of the devices is controlled by the polymer used, and thus with a variety of polymer coatings, the range of VOCs that can be detected is wide.

The devices are physically small with low power consumption since they operate at, or close to, room temperature. The response time is rapid at about 2–20 seconds, and this is coupled with rapid recovery of the baseline when the sample is removed [39]. The sensors can detect odors at sensitivities of 0.1 ppm, but 10 to 100 ppm is more usual.

2.6.3 Quartz Crystal Microbalance (QCM)/ Bulk Acoustic Wave (BAW)

The QCM or BAW sensor is a piezoelectric device based on a quartz crystal oscillator, developed by King in 1964 with his piezoelectric absorption detector. The sensors can be used to detect changes in temperature, pressure, force and acceleration, but in e-noses they are used to measure mass change [40]. The structure of a QCM sensor is shown in Figure 2.10.

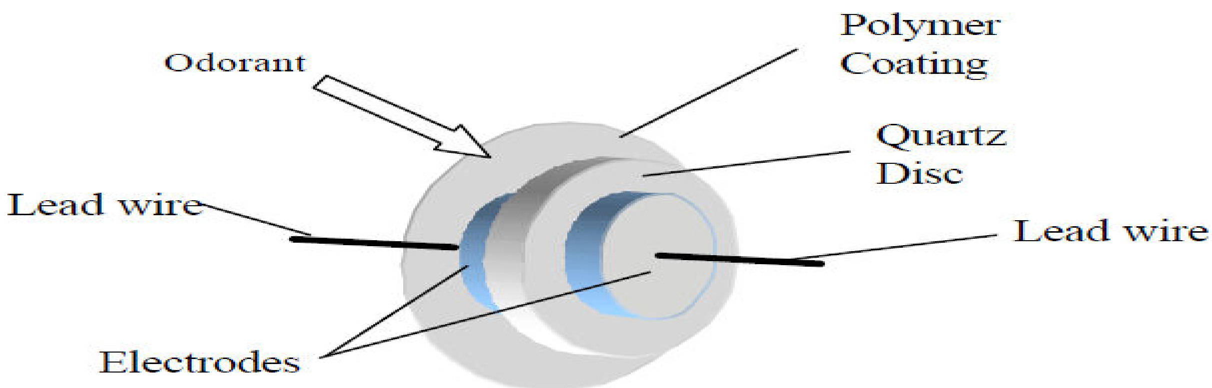

Figure 2.10 QCM/BAW Sensor

The sensor consists of a polymer coated disc of resonating quartz crystal. It is a few millimeters in diameter with metal electrodes on either side that are connected to a lead wire. When a voltage is applied across the disc it can be made to oscillate at its resonant frequency. Gas molecules are adsorbed onto the polymer coating and increase the mass of the disc. This increase in mass causes a measurable reduction in the resonant frequency of the disc that can be used to recognize the odors present. The sensitivity and selectivity of QCM devices can be altered by the use of different polymer coatings.

2.6.4 Surface Acoustic-Wave Sensors (SAW)

Like QCMs, SAWs are piezoelectric sensors. They rely on the transmission of a surface wave, (Raleigh wave) across the surface of quartz or silicon substrate onto which a thin coating has been applied. The waves are stimulated by an A.C. voltage, applied to the fingers of an inter-digitated electrodes structure. The structure of a SAW sensor is shown in Figure 2.11.

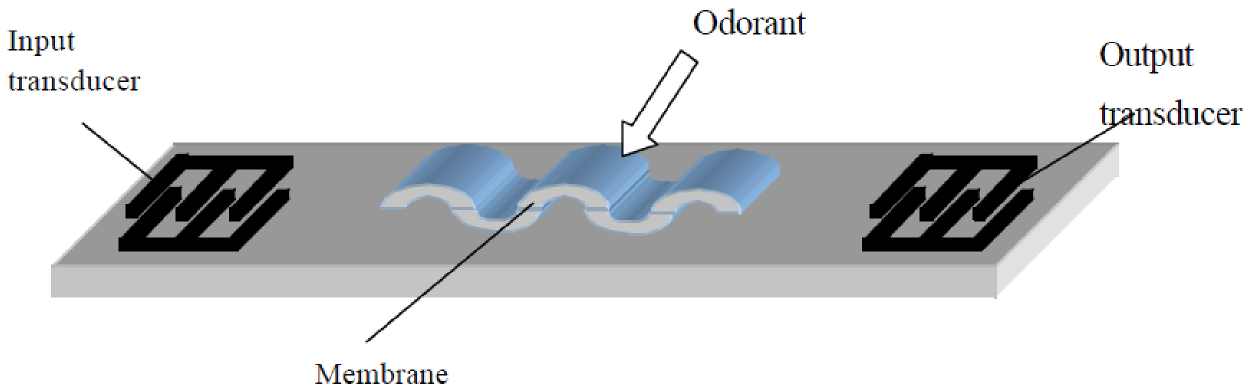

Figure 2.11 A Surface Acoustic-wave Sensor

The most common structure has two devices coupled in parallel. One plate is coated with an absorbent film, and other one is left uncoated as a reference to remove common mode effects. They operate by detecting the effect of the absorbed mass of volatile molecules on the propagation of the Raleigh waves on the coated plate. The absorption causes a change in wave velocity, therefore changing the frequency, and amplitude of oscillation. The differential frequency is used to recognize the odor. They are not widely available but have successfully been used for electronic nose applications [41].

2.6.5 Field Effect Gas Sensors

They are based on metal-insulator-semiconductor structures and are mainly two types, metal insulator-semiconductor field-effect transistor (MISFET), called

MOSFET and metal insulator-semiconductor capacitor (MISCAP). MOSFET sensors are the most commonly used in many e-nose applications. The basic structure of a MOSFET sensor is shown in Figure 2.12.

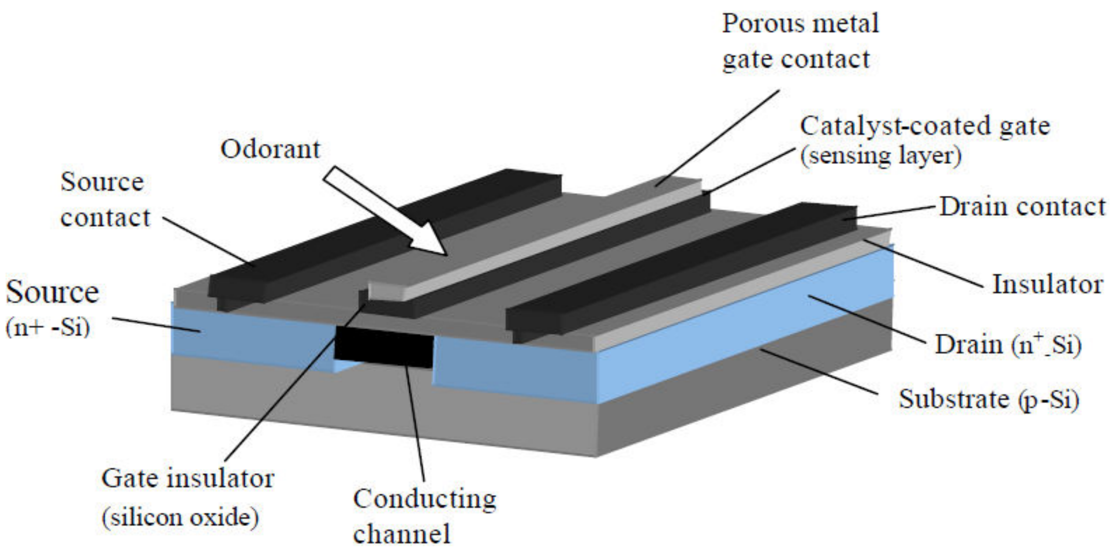

Figure 2.12 Structure of Conductive MOSFET sensors

The MOSFET sensor was invented in Sweden by Prof. Ingemar Lundstrom et al [42]. MOSFET gas sensors are metal oxide silicon field effect transistors coated with a thin catalytic metal layer. When the sensors come into contact with Volatile compounds a reaction takes place in the catalytic metal, that diffuses through the gate and change the electrical properties of the device. The sensitivity and selectivity of MOSFET devices can be changed by using different types, structures and thicknesses of metal catalyst and also by changing the operating temperature of the sensor between 100°C and 180°C.

MOSFET sensors respond to a wide range of complex odors, consist of amines, aldehydes, esters, ketones, aromates and alcohols [43]. The sensors respond to ppm concentrations of compounds. They are available commercially by special order only and till now used in some commercial e-nose systems.

2.6.6 Optical sensors (Fiber optics)

Fiber optics technologies are used for a wide range of applications and these have included gas and ammonia [44] sensing. They contain a thin chemically active material photo-deposited on their sides or ends [45] as shown in Figure 2.13.

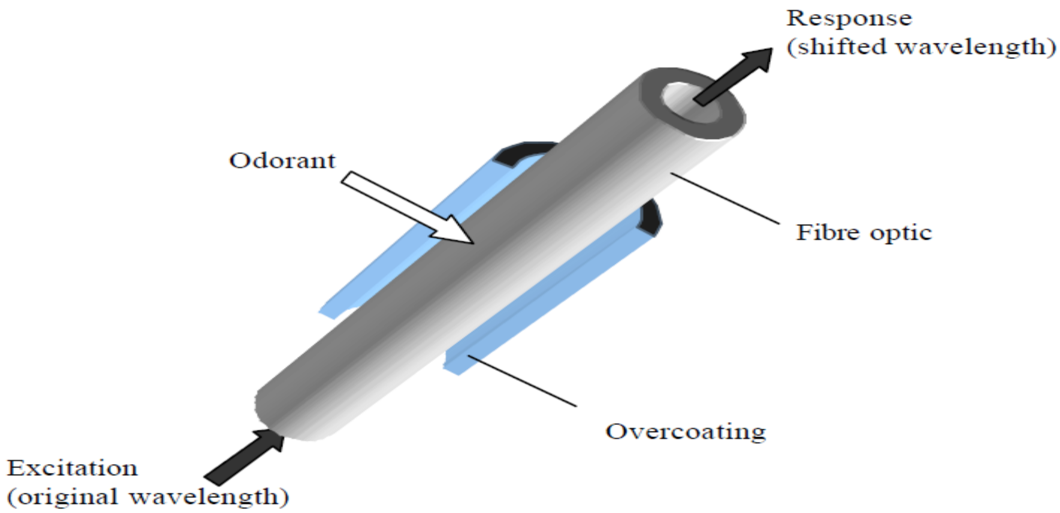

Figure 2.13 Odorant fiber-optic sensor

A light source at a single or narrow band of frequencies is used to activate the material, which responds in the presence of gas vapor. Change in path length, fluorescence, luminescence, absorption and reflectivity are the optical properties that can be measured. Different coatings interact with gases differently, such that upon exposure to a given sample the sensors provide unique information. It can be very small (2µm in diameter,) and big bundles are available, allowing fabrication of miniature arrays and remote measurements, and they are immune to electrical noise.

The sensitivity of the devices can be changed by altering the coating and to reduce common mode effects differential measurements are possible [46]. The complexity of the control system makes integration complicated and costly so these sensors are not commercially available.

2.6.7 Electrochemical Gas Sensors

They utilize oxidation or reduction of an analyte on the surface of a catalytic electrode. The presence of a specific gas triggers an electrochemical reaction, generating a current that can be measured. The main advantages of these sensors are their low power consumption at room temperature, long life and robust structure. However, they respond only to a specific gas and are large in size (up to 3cm diameter) [47].

2.6.8 Pellistors

They sense combustible gases by detecting heat produced from the gas reacting with oxygen on the surface of a catalytic bead [48]. They respond rapidly to a variety of combustible gases. They have advantages of robustness and lifetimes of several years. Their main disadvantages for use in e-noses systems are high power consumption (350mW) and poisoning by certain VOCs [49].

2.6.9 Chemoresistors

They have thin film of phthalocyanin molecules on interdigitated electrodes that used to detect a few number of electrophilic gases. They respond slowly, but are very sensitive. Their key advantages are the variety of polymers that can be used to change the selectivity of the devices; small in size and rapid respond (100 to 1000s). Some of them utilize electrodes coated with carbon-black polymer films that respond to VOCs. Their main disadvantage is very high selectivity, means they respond only to electrophillic gases [50].

2.6.10 Properties of a Good Sensor

The good sensors must fulfill a number of criteria. They should have the highest sensitivity to the target group of chemical compound(s) intended for identification with a threshold of detection similar to mammalian nose, down to about 10-12 g mL^{-1} [51]. They should have relatively low selectivity in order to be sensitive to a wide number of different chemical compounds. They must have low sensitivity to variable environmental parameters, particularly to temperature and humidity.

They should be capable of operating at relatively low temperatures if necessary, fast recovery time and less maintenance procedures to maintain low operating costs and have short calibration and training requirements,. They must also have high sensor array stability and short recording and analysis times, particularly when used as on-line systems. Finally they must be very portable and small for convenient diverse operations and should have built-in recording and analysis capabilities. Table 2.2 summaries of the types and mechanisms involved with some common gas sensor technologies.

Table 2.2: A summary of the types and mechanisms involved with some common gas sensor technologies.

S. N.	Sensor types	Sensitive Material	Detection Principle
1.	Electrochemical Sensors	Solid or Liquid Electrolytes	Current or Voltage Change
2.	Conducting Polymer Sensors	Modifying Conducting Polymers	Resistance Change
3.	Acoustic Sensors: Quartz	Organic or Inorganic Film	Mass Change

	Crystal Microbalance (QMB); Surface & Bulk Acoustic Wave(SAW, BAW)	Layers	(Frequency Shift)
4.	Calorimetric; Catalytic Bead(CB)	Pellistor	Temperature or Heat Change (Chemical Reaction)
5.	Catalytic field effect sensors(MOSFET)	Catalytic Metals	Electric Field Change
6.	Colorimetric Sensors	Organic Dyes	Color Changes, Absorbance
7.	Fluorescence Sensors	Fluorescence Sensitive Detector	Fluorescent-light Emissions
8.	Infrared Sensors	IR Sensitive Detectors	Infrared Radiation Absorption
9.	Optical Sensors	Photodiode, Light sensitive	Light Modulation, Optical changes
10.	Metal Oxide Semiconducting (MOS, Taguchi)	Doped Semiconducting Metal Oxides (SnO_2, GaO)	Resistance Change

2.6.11 Comparison among Sensor Technologies:-

Some of the major advantages and disadvantages associated with each e-nose sensor technologies are summarized in the Table 2.3

Table 2.3 A listing of some of the major advantages and disadvantages associated with each e-nose sensor

S. N.	Sensor Types	Advantages	Disadvantages	Sensitivity
1.	Electrochemical Sensors	Ambient temperature operation, Low Power Consumption, Very sensitive to diverse VOCs	Bulky size, Limited Sensitivity to simple or low molecular weight Gases	1-2000 ppm
2.	Conducting Polymer Sensors	Inexpensive, Room temperature operation, Sensitive to wide range of VOCs, Short response time, Diverse sensor coating, Resistance to sensor poisoning	Sensitive to humidity and temperature, Sensor can be overloaded by certain analytes, Sensor life is limited	0.1-100 ppm
3.	Acoustic Sensors: Quartz Crystal Microbalance (QMB);	High Sensitivity, Good Precision, Diverse range of sensor coating, Good response time(100-1000s), Good reproducibility, measure both polar and non-polar species	Sensitive to humidity and temperature, Complex circuitry, Poor signal to noise ratio	100ppb (1 ng mass change)
4.	Acoustic Sensors: Surface & Bulk Acoustic	Inexpensive, Small, High sensitivity, Good response time (100-1000s), Diverse sensor coating, Sensitive to virtually all gases.	Sensitive to temperature, Complex Circuitry, Specificity to analyte groups is affected by polymeric film sensor	1ppb (1 pg mass change)

	Wave(SAW, BAW)		coating	
5.	Calorimetric; Catalytic Bead(CB)	Fast response and recovery time, High specificity for oxidized compounds	High temperature operation, Only sensitive to oxygen containing compounds	700 ppm
6.	Catalytic field effect sensors(MOSFET)	Small sensor size, Operation cost is low, Low power consumption, Fast response time(10s), Insensitive to humidity, Slow Baseline drift	Require environmental control, Low sensitivity to ammonia and carbon dioxide, odorant reaction must penetrate gate	1 ppm
7.	Optical Sensors (Fiber optic)	Detection of wide range of VOCs ,Very high sensitivity, Capable of identifications of individual compounds in the mixtures, Multi parameter detection capabilities, Immune to electrical noise, Remote measurement	Complex sensor array systems, More expensive to operate, Low portability due to delicate optical and electrical components	1 ppb
8.	Metal Oxide Semiconducting (MOS, Taguchi)	Very high sensitivity, Limited sensing range, Rapid response and recovery time for low molecular weight Gases, Robust, Lifetime more than 5 years	High temperature operation, High power consumption(800mW), Poor precision, Sulfur and weak acid poisoning, Limited sensor coatings, Sensitive to humidity	5-500 ppm

A range of sensor technologies has been reported for e-nose applications [52], although no single gas sensor technology fulfills all the requirements of good sensor.

Metal-oxide semiconductors (MOS) is the most widely used sensor in e-nose applications today, having a high level of sensitivity for a wide range of organic vapors and provide perhaps the best balance between sensitivity , drift and lifetime. But they have serious disadvantages like high temperature operation, sensitivity to humidity, poor precision and logarithmic dependence of the sensor response on the gas concentration that causes problems in the presence of high concentrations of detectable species (e.g., ethanol in alcoholic products). Other potential problems related to use in with food products (e.g., the baseline recovery is slow when they are exposed to high molecular weight compounds and they are prone to poisoning by sulphur-containing species or by weak acids) [53].

The quartz crystal microbalances (QMBs) and the surface acoustic wave transducers (SAWs) have sensitivity around at the µg/l level more than sensitivities of MOS and Conductive Polymer sensors are at the mg/l level. But poor batch-to-batch reproducibility during manufacturing and response dependence on temperature are problems that still have to be rectified [54].

Despite the considerable number of applications that appear in food analysis literature [53-55], much development is still needed before e-noses based on gas sensors can attain their full potential in real, commercially ready applications.

2.7 Data analysis approaches for Electronic Nose systems

Pattern recognition methods are most widely classified by means of their learning approach (either supervised or unsupervised) even though they can also be classified in terms of linearity or parametric basis, and also by the dimensionality of data they deal with. Types of different data analysis are given in Figure 2.14.

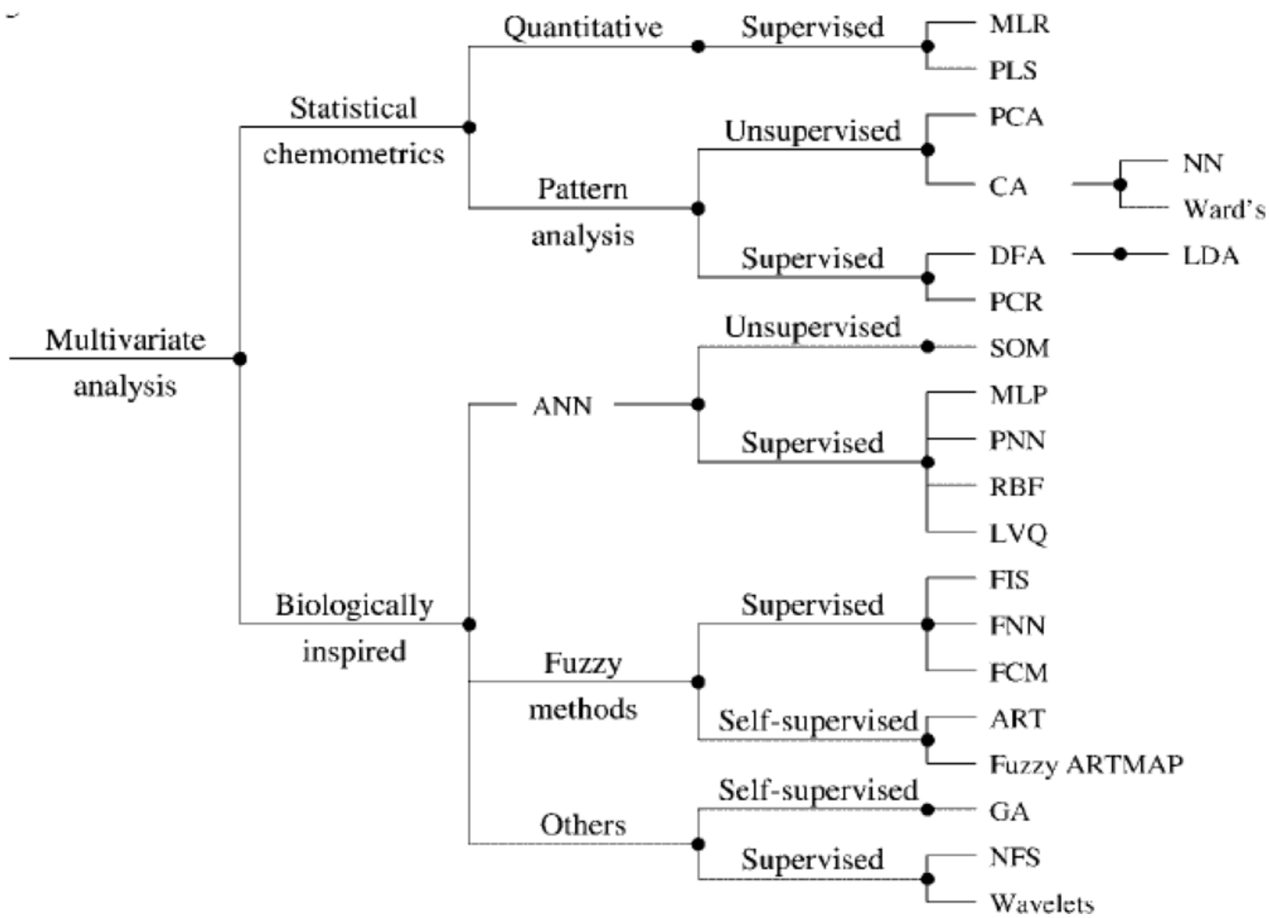

Figure 2.14 Types of data analysis

Commercially available analysis techniques fall into three main categories as follows:

1. Graphical approaches: bar charts, profiles, polar and offset polar plots
2. Multivariate data analysis (MDA): principal component analysis (PCA), canonical discriminate analysis (CDA), featured within (FW) and cluster analysis (CA)
3. Artificial intelligence approaches: artificial neural networks (ANN) and radial basis functions (RBF)

Artificial intelligence (AI) techniques are generally non-conventional, intuitive approaches for problem solving, often biologically inspired and can be divided into following sub-groups:

- Artificial neural networks (ANN), which include multi layer perceptrons (MLP), radial basis function networks (RBF), self organizing maps (SOM), learning vector quantization (LVQ) and self organizing competitive systems such as the adaptive resonance theory (ART) or the family growing cell of algorithms.
- Fuzzy logic and fuzzy rules based algorithms
- Genetic Algorithms (GA) used for feature selection based on the dimensionality of the multivariate data to be analyzed, we can classify the pattern recognition methods into two-way data analysis (PCA, PLS, etc.), three-way data analysis (PARAFAC, n-PLS, Tucker) and even multi-way data analysis, where the data exceeds three dimensions.

The choice of the method used depends on the type of available input data attained by the sensors and the type of information that is sought. The simplest form of data reduction is the graphical analysis approach, useful for comparing samples or comparing aroma identification elements of unknown analytes relative to those of known sources in reference libraries. Multivariate data analysis comprises a set of techniques for the analysis of data sets with more than one variable by reducing high dimensionality in a multivariate problem when variables are partly correlated, so they can be displayed in two or three dimensions. For electronic-nose data analysis, MDA is very useful when sensors have partial-coverage sensitivities to individual compounds present in the sample mixture. Multivariate analysis can be divided into untrained or trained techniques.

Untrained techniques are used when a database of known samples has not been previously built, therefore it is neither necessary nor intended for recognizing the sample itself, but for making comparisons between different unknown samples, to discriminate between them. The simplest and most widely used untrained MDA technique is principal component analysis. PCA is most useful when no known sample is available, or when hidden relationships between samples or variables are suspected. On the contrary, trained or supervised learning techniques classify unknown samples on the basis of characteristics of known samples or sets of samples with known properties that are usually maintained in a reference library that is accessed during analysis.

Artificial neural networks (ANN) are the newest analysis techniques incorporated to statistical software packages for commercially available electronic noses. Mimicking the cognitive processes of the human brain, neural network approaches are based on interconnected data processing algorithms that work in parallel. Various instrument-training methods are employed through pattern recognition algorithms that look for similarities and differences between identification elements of known aroma patterns found in an analyte-specific reference library. The training process requires a discrete amount of known sample data to train the system and is very efficient in comparing unknown samples to known references [56]. The result of ANN data analysis usually is in the form of a percentage match of identification elements in the sample with those of aroma patterns from known sources in the reference library.

Neural networks offer a chemo-metric technique of great potential for the treatment of signals generated by electronic noses based on sensors that can provide non-linear responses. Several types of classifiers based on different ANN-algorithms such as multi-layer perceptron (MLP), radial basis function (RBF), self-organizing map (SOM), PNN, FANN and SVM have been used in many electronic nose applications [57-61]. The nature of an ANN-algorithm is usually classified as a supervised or unsupervised method.

In a supervised learning ANN method, a set of known odors are systematically introduced to the electronic nose, which then classifies them according to known descriptors or classes held in a knowledge base. Then, in a second stage for identification, an unknown odor is tested against the knowledge base, now containing the learnt relationship, and then the class membership is predicted. For unsupervised learning, ANN methods learn to separate the different classes from the response vectors without any additional information. In other words, unsupervised methods discriminate between unknown odor vectors without being presented with the corresponding descriptors. These methods are closer to the way that the human olfactory system works. They both use intuitive associations with no, or little, prior knowledge.

2.8 Available Commercial E-Nose Systems

A summary of some of the most widely used electronic noses with manufacturers, models available and technological basis are listed in Table 2.4.

Table 2.4 Electronic noses with manufacturers, models and technological basis

S.N.	Instrument Type	Manufacturer	Models	Technology Basis
1.	Single Technology (e-nose sensors only)	Airsense analytes	i-Pen, PEN2, PEN3	MOS sensors
2.		Alpha MOS	FOX2000, 3000, 4000	MOS sensors
3.		Applied Sensor	Air quality module	MOS sensors
4.		Chemsensing	Chemsensing sensor array	Colorimetric optical
5.		Cogniscent Inc	Scen Trak	Dye Polymer

				sensors
6.	Single Technology (e-nose sensors only)	CSIRO	Cybernose	Receptor-based array
7.		Dr. Fodisch AG	OMD98, 1.10	MOS sensors
8.		Forschungszentrum Karlsruhe	SAGAS	SAW sensors
9.		Gerstel GmBH Co.	QSC	MOS sensors
10.		GSG Mess-und Analysengerate	MOSES II	Modular gas sensors
11.		Illumina Inc.	oNose	Flourescence Optical
12.		Microsensor Systems Inc.	Hazmatcad, Fuel sniffer, SAW mini CAD mk II	SAW sensors
13.		Osmetech Plc.	Aromascan A32S	Conducting polymers
14.		Sacmi	EOS 835, Ambiente	Gas sensor array
15.		Sensive Technol.	Bloodhound ST214	Conducting polymers
16.		Smiths Group plc	Cyranose 320	Carbon black polymers
17.		Sysca AG	Artinose	MOS sensors
18.		Technobiochip	LibraNose 2.1	QMB sensors
19.	Combined Technology	Airsense analytes	GDA 2	MOS, EC, IMS, PID

20.	(e-nose + other types)	Alpha MOS	RQ Box, Prometheus	MOS, EC, IMS, PID
21.		Electronic Sensor Technology	ZNose4200, 4300, 7100	SAW, GC
22.		Microsensor Systems Inc.	Hazmatcad Plus, CW Sentry 3G	SAW, EC
23.		Rae Systems	Area RAE monitor	CB, O_2, EC, PID
24.			IAQRAE	Thermistor, EC, PID, CO_2, Humudity
25.		RST Rostock	FF2, GFD1	MOS, QMB, SAW

The numbers of e-noses sold by various manufacturers has largely depended on the technology basis of individual instruments, costs per unit, and specific application needs. In 1997, there were about 500 total desk-top analytical instruments units sold worldwide with an approximate market value of $30 million Euros [62]. Within the past ten years, the Applied Sensor Company has sold the highest number of units (> 100,000) of their e-nose (the Air Quality Module electronic nose). Their system is primarily used to maintain ambient or environmental air quality by detection of odors, VOCs and carbon dioxide within living spaces [63].

The Alpha-MOS Fox electronic nose was designed in collaboration with the Universities of Warwick and employs either six (Fox 2000), 12 (Fox 3000) or 18 (Fox 4000) metal oxide gas sensors and can be used with external carrier gas bottles in a flow-injection system, or with an internal pump and mass-flow controller. Among the electronic noses that did not survive the economy we shortly mention: Aromascan A32S, Cyranose 320, Airsense PEN2. The Aromascan A32S was an organic matrix-coated polymer-type 32-detector e-nose based on an earlier design using technology arising from the University of Manchester, Institute of Science and Technology. Aromascan was acquired by the Osmetech Company, which currently produces the e-Sensor XT-8 systems, a diagnosis platform by means of electrochemical detection technology to detect nucleic acids on microarrays.

The Cyranose 320 was a portable electronic-nose system whose component technology consists of 32 individual polymer sensors blended with carbon black composite and configured as an array [64]. The company was acquired by Smiths Detection, whose products are oriented towards surveillance, explosive and narcotics detection. Airsense's PEN2 and PEN3 e-noses were unique on the electronic nose market in the sense that they were a very small and portable 10 metal-oxide semiconductor (MOS) gas sensor array with a small volume measuring chamber. It can be linked with an adsorbent trapping unit or a headspace auto sampler for laboratory analyses. Now the company does not market the product anymore, being oriented towards fire detection systems.

2.9 Electronic Nose Applications

Electronic-nose systems have been specifically designed to be used for various applications in many industrial production processes. There are variety of industries based on specific product types and categories, such as the packaging, cosmetic, automobile, food, drug and biomedical industries use e-noses for a broad and diverse range of applications like quality control of raw materials and manufactured products, process design, freshness and ripeness monitoring, shelf-life investigations, authenticity assessments of quality products, classification of perfumes and scents, microbial pathogen detection and environmental assessment studies. These all are summarized in Table 2.5.

Table 2.5 Examples of electronic noses in some industry-based applications

Industry sector	Application area	Specific use types and examples
Agriculture	a. crop protection b. harvest timing & storage c. meat, seafood & fish products d. plant production e. pre & post-harvest diseases	homeland security, safe food supply crop ripeness, preservation treatments freshness, contamination, spoilage cultivar selection, variety characteristics plant disease diagnoses, pest identification, detect non-indigenous pests of food crops
Airline Transportation	a. public safety & welfare b. passenger & personnel security	explosive & flammable materials detection
Cosmetics	a. personal application products b. fragrance additives	perfume & cologne development product enhancement, consumer appeal
Environmental	a. air & water quality monitoring b. indoor air quality control c. pollution abatement regulations	pollution detection, effluents, toxic spills malodor emissions, toxic/hazardous gases control of point-source pollution releases

Food & Beverage	a. consumer fraud prevention b. quality control assessments c. ripeness, food contamination d. taste, smell characteristics	ingredient confirmation, content standards brand recognition, product consistency marketable condition, spoilage, shelf life off-flavors, product variety assessments
Manufacturing	a. processing controls b. product uniformity c. safety, security, work conditions	product characteristics & consistency aroma and flavor characteristics fire alarms, toxic gas leak detection
Medical & Clinical	a. pathogen identification b. pathogen or disease detection c. physiological conditions	patient treatment selection, prognoses disease diagnoses, metabolic disorders nutritional status, organ failures
Military	a. personnel & population security b. civilian & military	biological & chemical weapons safety explosive materials detection
Pharmaceutical	a. contamination, product purity b. variations in product mixtures	quality control of drug purity formulation consistency & uniformity
Regulatory	a. consumer protection b. environmental protection	product safety, hazardous characteristics air, water, and soil contamination tests

Scientific Research	a. botany, ecological studies	chemotaxonomy, ecosystem functions
	b. engineering, material properties	machine design, chemical processes
	c. microbiology, pathology	microbe and metabolite identifications

2.10 Developments in Electronic Nose Systems

2.10.1 Hardware Developments

Gardner, J.W. et al [65] designed an electronic circuitry that measures the odors at the ppm and sub-ppm level for some analytes.

Baltes, H. Lange et al. [66] designed a miniature experimental system based on a CMOS chip to detect a range of gaseous compounds, to avoid use of handy electronic noses used in industrial cleaning or fabrication processes.

Depari, A. et al. [67] proposed a prototype based on a new approach to realize low-cost electronic noses. They have given a novel instrument to manage high-value resistive sensors varying over a wide range, from kilo Ohms to giga Ohms. It was a modular architecture which takes advantage from an improved resistance-to-period converter (RPC), where sensors were DC powered.

Subramanian, V. et al. [68] reported embedded sensors based on printed organic semiconductors that are attractive for use in product content monitoring due to their low cost. Arraying multiple sensor elements, in a bridge topology, yields signatures that achieve high specificity using non-specific elements. They got sensitivity up to 10ppm. They had demonstrated the wine-spoilage application.

De Girolamo Del Mauro et al. [69] developed a wireless electronic nose prototype, called Tiny Nose, having an array of four different polymeric-composite sensors that is for VOC compounds detection and discrimination purposes during a measurement campaign. Sensors are fabricated using a carbon black conducting phase dispersed in different polymeric matrices.

Morsi, I. [70] developed a prototype of multi-sensors monitoring system, which are TGS 822, TGS 2442, TGS 813, TGS 4160, TGS 2600, temperature sensor, humidity sensor and wind speed measurements. All sensors were connected to the microcontroller and PC to visualize and analyze data. Quadratic surface regression method is used to find possible correlations existence between some pollutants, elaborated by MATLAB software and analysis of variance (ANOVA) is used to detect the significant factors in the final quadrate equation and understanding the functional relationship between a set of independent factors.

Xiaojun Zhang et al. [71] have designed a bionic electronic nose for robot, which has a human-like nasal cavity structure to detect toxic gases, explosives, leakage, etc. It has compact structure, simple mechanical interface and communication interface so can work as a portable electronic nose. It can simultaneously detect five kinds of different toxic gases, namely, CO, SO_2, H_2S, Cl_2 and NH_3, by using of electrochemical gas sensor array in which each sensor responds to the specific gas, and has good selectivity and linear output. It greatly reduces the operation task of olfactory system and improves the detection accuracy and efficiency to meet the robotpsilas real-time requirement, and can be conveniently changed corresponding gas sensors based on the target gases.

Saad, Nor Hayati et al. [72] have used a new type chemical sensor named coupled micro resonator array (CMRA) that has been developed to improve the performance of the electronic nose by increasing the number of sensors that can be used to detect specific odours. They have modeled and evaluated the performance of an unsymmetrical yet balanced CMRA for individual resonator mass determination.

Kai Song et al. [73] had designed an electronic nose system based on a single chip to analyze methane, hydrogen and their mixtures, the main combustible gases in the environment that can detect the gases in real-time and quantify their concentrations accurately. They have employed four Fe_2O_3 gas sensors with little humidity effect in the gas sensor array and used principal component regression (PCR) method to process the electronic nose data. Software is developed in the LabVIEW environment.

Mamat, M. et al. [74] reported the design of an Electronic Nose prototype based on commercially available metal oxide gas sensors and a temperature sensor. It consists of a sampling chamber, sensing chamber, pumps, data acquisition system and controller unit and a computer for data analysis. By conducting many experiments on three beverages: blackcurrant juice, orange juice and soy milk they had verified that the developed system was reliable to produce correct measurement with high repeatability, reproducibility and discriminative ability.

Pogfay, T. et al. [75] have developed a wireless electronic nose by deploying commercial gas sensor arrays, microcontrollers and ZigBee wireless network for monitoring air quality of environment. The database station use PCA as linear explorative technique analysis to obtain odor dispersion and environment prediction. This system has potential for automatic environment monitoring with many industrial applications including livestock farm environmental control.

Kea-Tiong Tang et al. [76] reported the world's first E-Nose system-on-chip (SoC) based on TSMC 0.18μm 1P6M CMOS technology, which fully integrates the sensor array and signal processing circuit. The sensor array is coated with multiple-walled carbon nanotubes (MWNTs) conducting polymers. The signal processing circuit has interface circuitry, analog-to-digital converter, memory, and microprocessor embedded with a pattern recognition algorithm. The power consumption of the chip was measured at 1.05mW, making it very suitable for a wearable device. The E-Nose SoC has been integrated in a prototype, and successfully recognized eight odors emulating decayed fish, liver damage, careinogen and industrial solvent.

Kladsomboon, S. et al. [77] have reported an optical-based electronic nose system consisting of thin-film sensing materials, array of light emitting diode (LED), photo-detector and pattern recognition program to detect volatile organic compound (VOCs) vapors such as methanol, ethanol, and isopropanol. Principal component analysis (PCA) has been used as the pattern recognition system. The light intensity that is transmitted through the organic thin film during the experiment was detected by the color light to frequency converter device i.e. photo-detector.

Thepudom, T. et al. [78] have developed a portable optical-based e-nose that features several advantages over traditional e-nose systems, namely changeable dual sensor arrays, plug-in low-cost LED sources and switchable air/liquid sample handling. The principal component analysis (PCA) was used to analyze the data from both thin film gas sensors. It has been tested this e-nose with three types of alcohols such as ethanol, methanol and isopropanol.

Román Bataller et al. [79] designed a prototype of a humid electronic nose, consists of an array of four working electrodes (i.e. Au, Pt, Ir and Rh) housed inside a homemade stainless steel cylinder and a fabric mesh made of nylon damped with a NaCl aqueous solution as the supporting humid membrane. It is used to detect volatile samples (i.e. ammonia, acetone, acetic acid and 6-amino-1-hexanol) and different food samples (i.e. onion, coffee and Roquefort cheese). PCA used for analysis.

Mercedes, Crego-Calama et al. [80] presented a novel approach for fabricating electronic nose systems for adaptation into autonomous wireless sensor nodes. They used micromechanical systems consist of micromechanical beams with integrated piezoelectric transducers that are an ideal technology for fabricating miniaturized sensor arrays for low-power applications and fulfill requirements of sensitivity, arrayability and integratability.

Yong Kyoung Yoo et al. [81] reported a novel microcantilever array chip having four microreaction chambers, which consequently contains four different functionalized surfaces for multitarget detection. They have designed microcantilever chips and demonstrated the ability of binding of 2,4-dinitrotoluene (DNT) targets onto four different surfaces using high selective binding receptors, peptide.

Castro, M. et al [82] reported the original design of a new type of electronic nose having only five sensors made of conductive polymer nanocomposites (CPC) which is hierarchically structured. Each sensor has the properties of exceptional electrical properties of carbon nanotubes (CNT) used to make the conductive part and the assembly technique of spray layer by layer (sLbL) that provides the transducers of highly specific 3D surface structure. Nine VOCs are chosen from biomarkers for lung cancer detection in breath.

2.10.2 Software Developments

Gardner, J.W. [83] reported a multisensor array system using principal component and cluster analysis that allow the identification of mixtures of organic samples as a whole without having to identify individual chemical species within the sample mixture.

Gardner, J.W. et al [84] designed a electronic nose system consists of a multisensor array, an information-processing unit having software with digital pattern-recognition algorithms, and reference-library databases.

Gardner, J.W. et al [85] used an electronic nose to predict the class and growth phase of two potentially pathogenic microorganisms: *Escherichia coli* and *S. aureus*. Head spaces were examined by using an array of six different metal oxide semi-conducting gas sensors, classified by a multi-layer perception (MLP) with a back-propagation learning algorithm.

Pardo, M. et al. [86] reported feature selection (FS) on an electronic nose dataset composed of 30 features, obtained by extracting 5 diverse features that are the classical relative change in resistance, R/R_0; the curve integral over both the gas adsorption and desorption processes, from the response curves of 6 metal oxide sensors. Features (phase and integral) calculated on desorption seem consistently to give higher performance than the corresponding features calculated during adsorption. The standard R/R_0 stands in lower ranking.

Allen, J.N. et al. [87] have analyzed and implemented an olfaction detection spiking neural network that detects binary odor patterns. They have presented a new method for inhibiting spiking neural networks by modulating a detection threshold that reduces false-positive detection error in high noise scenarios. A digital implementation of the inhibition is done with fifteen odors active at a time.

Matthes, J. et al. [88] presented a new two-step approach based on an analytical diffusion-advection model on point wise concentration measurements from a network of stationary spatially distributed electronic noses, for locating an emission source. This approach overcomes the problem of poor convergence and multiple solutions of iterative algorithms, which minimize the output error of the model. In this approach first, for each Electronic Nose the set of points is determined, on which the source can lie. Then, an estimate for the source position is evaluated by intersecting these sets.

Daqi, G. et al. [89] proposed a method to simultaneously estimate the classes and concentrations of 4 kinds of fragrant materials, namely ethanol, ethyl acetate, ethyl caproate and ethyl lactate. They have presented an electronic nose having combinative and modular single-hidden-layer perceptrons. Every module is made up of multiple single-input single-output multilayer perceptrons.

Daqi, G. et al. [90] reported combinative and modular approximation models to simultaneously estimate odor classes and strengths, by first decomposing a many-to-many approximation task into multiple many-to-one tasks, then realize them using multiple many-to-one approximation models. A single model is regarded as an expert that is either a multivariate logarithmic regression model, or a multilayer perceptron (MLP), or a support vector machine (SVM).

Matthes, J. et al. [91] presented a new two-step approach for solving the source localization problem occurs in concentration measurements from spatially distributed electronic noses. This approach overcomes the problem of poor convergence of iterative algorithms, which try to minimize the least squares output error.

Depari, A. et al. [92] proposed a solution for the implementation of low-cost electronic noses suitable for food processing, pollution control and security system, as well as for laboratory works. They have used two innovative chemical sensor typologies (resonant and resistive) and related high-performance first conditioning circuits. The system has modular low-noise architecture, thus it can be tailored for the particular application, lowering the overall cost. To decrease cost further detection process can be performed directly by the instrument using a neural network approach.

Hong Men et al. [93] have applied Back-propagation neural network (BP), radial basis function neural network (RBF), and self-organization mapping networks (SOM) to identify three gases by electronic nose gas sensors (CO, SO_2, and NO_2) qualitatively. They have used three training algorithms, gradient descent (traingd), gradient descent with momentum of variable learning rate (traingdx) and Levenberg-Marquardt (trainlm) algorithm. They found traingd and traingdx algorithms too slow for practical problems. Training speed of trainlm is faster more. The RBF networks provide a simple and robust method and the sampling gases were clearly classified with few errors. The RBF networks trained faster than the BP networks do, while exhibiting none of back-propagation's training pathologies such as paralysis of local minima problems. The SOM networks can classify accurately and generalization capability is far superior, while recognized patterns are non-rectangular in shape and size, the performance is poor.

Fujinaka, T. et al. [94] designed an intelligent electronic nose system using cheap metal oxide gas sensors (MOGS) to detect fires at an early stage. The error back propagation (BP) method has been used for the classification of the tested smell and achieved 99.6% accuracy. By k-means algorithm accuracy achieved is

98.3%, so the Electronic Nose is able to detect the early stage of fire from various sources.

Zhou Tao et al. [95] proposed pattern recognition methods based on the probabilistic neural networks (PNN) used in electronic nose systems that can identify all the samples of beer, fruit juice and milk. They have discussed pattern recognition methods of the universal electronic nose that has much advantage over others such as simple methods of pattern recognition and classification, easy training approaches and wider application fields.

Yan Weiping et al. [96] have analyzed the neural network and the fuzzy logic of the pattern recognition technique, to realize signal preprocessing in electronic nose by combining PCA with ICA that effectively utilized both advantages. Takagi-Sugeno fuzzy logic system based on Neural Networks was used to recognize the alcoholpsilas quantitative of the multi-gas. They have shown that the signal preprocessing techniques and the fuzzy network algorithm could improve the identification of the electronics nose.

Bhattacharyya, N. et al. [97] developed an electronic nose anchored aroma characterization model based on PNN classification strategy having the incremental learning ability that can automatically include the newly presented patterns in the training dataset without affecting class integrity of the previously trained classifier. The incremental learning mechanism has been suitably grafted to the PNN model to have efficient co-relation of electronic nose signature with tea tasterspsila scores. The incremental PNN classifier promises to be a versatile pattern classification algorithm for black tea grade discrimination using electronic nose system.

Waphare, S. et al. [98] presented the implementation of 2 novel algorithms named Surge-spiralx and Surge-castx on sniffer robot for odor plume tracking in a laminar wind environment. The experimental set up had odor tunnel, Sniffer Robot with 3 odor sensors and one anemometer. Experiments are carried out in two environments to study behavior of an algorithm. The algorithms are implemented and they have shown very good performance in terms of success ratio, while Surge-Spiralx algorithm having less distance overhead.

E. Ongo et al. [99] presented a practical and promising approach to profile the headspace aroma attributes of Philippine civet coffee using electronic nose and gas chromatography mass spectrometry (GCMS). This method is applied to enhance the discrimination of civet coffee against its control coffee beans.

Hui Guohua et al. [100] proposed an electronic nose based quality predictive model (accuracy 87.5%) of grass carp stored at 277 K temperature having advantages of easy operation, quick response, high accuracy, good repeatability etc., to be used in aquatic food products quality evaluating applications. Principal component analysis method used to discriminate fresh, medium and aged grass carp samples. Stochastic resonance signal-to-noise ratio maximums distinguished fresh, medium, and aged grass carp samples successfully.

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