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Electronic Nose Evolution for Food Adulteration: A Review

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Abstract: An electronic nose (e-nose) is a device that identifies the specific components of an odor and analyzes its chemical makeup to identify it. An electronic nose consists of a mechanism for chemical detection, such as an array of electronic sensors, and a mechanism for pattern recognition, such as a neural network. The invention of many new e-nose sensor types and arrays, based on different detection principles and mechanisms, is closely correlated with the expansion of new applications. The present paper illustrates the function of electronic nose, its application and investigates the effective use of e-nose in detecting gases that have some smell developed by the volatile organic compounds (VOC) like ethanol, acetone and benzene at different concentrations. The focused food products for adulteration study are milk, wine and some fruits.

Keywords: E-Nose, sensors array, food adulteration, volatile organic compound (VOC), *Machine olfaction, milk, wine.*

I INTRODUCTION

In almost all living being there is an **olfactory system**, or sense of smell, is the part of the sensory **system** used for smelling (**olfaction**). Artificial olfactory system (**Machine olfaction)** is the automated simulation of the sense of smell. It is an emerging application of modern engineering where robots or other automated systems are needed to measure the existence of a particular chemical concentration in air. Such an apparatus is often called an electronic nose or e-nose. 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. As current knowledge does not allow the replacement of the human nose, constructors tend to compensate by integrating several sensor technologies into one instrument. However, one single instrument to be used in every possible application would be over-complicated due to the large number of sensors and time consuming statistical analysis. The trend is to create a system for one specific application. This means that a compact and portable instrument would be desirable. Electronic noses have been around for several years but have typically been large and expensive. Current research is focused on making the devices smaller, less expensive, and more sensitive. The smallest version, a nose-on-a-chip is a single computer chip containing both the sensors and the processing components.

II ELECTRONIC NOSE (E-NOSE)

An electronic nose (e-nose) is a device that identifies the specific components of an odor and analyzes its chemical makeup to identify it. An electronic nose consists of a mechanism for chemical detection, such as an array of electronic sensors, and a mechanism for pattern recognition, such as a neural network. Electronic noses have been around for several years but have typically been large and expensive. Current research is focused on making the devices smaller, less expensive, and more sensitive. The electronic Nose is an artificial olfactory system that consists of an array of chemical sensors, a sample handling system and a pattern recognition system [4]. The smallest version, a nose-on-a-chip is a single computer chip containing both the sensors and the processing components. Artificial olfactory system (**Machine olfaction)** is the automated simulation of the sense of smell. It is an emerging application of modern engineering where robots or other automated systems are needed to measure the existence of a particular chemical concentration in air. Such an apparatus is often called an electronic nose or e-nose. The figure below shows the E-nose techniques [7].

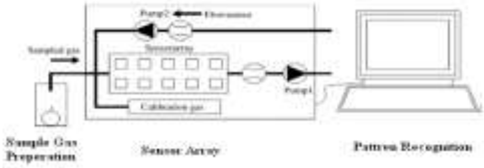

Figure 1: Basic E-nose Techniques

A variety of different sensor types have been developed, to which three types of materials are commonly used: metal oxides, conducting polymers composites and intrinsically conducting polymers. Apart from conductive sensors, gas detection has also been done using optical sensors, surface acoustic wave sensors, gas sensitive field effect transistors and quartz microbalance (QMB) sensors. Micro-electro-mechanical systems (MEMS) plus nanotechnologies are the most promising emerging technologies in the area. Once the data from the individual sensors from the array is collected, the electronic nose systems require a suitable post processing procedure to analyze and classify the data. Pre-processing of multivariate signals in sensor arrays represents an essential part of the measuring system. Data processing techniques used in post processing of pattern recognition routines include principal component analysis (PCA), linear discriminate analysis (LDA), partial least squares (PLS), functional discriminate analysis (FDA), cluster analysis (CA), fuzzy logic or artificial neural network (ANN) such as probabilistic neural network (PNN). Among these techniques, PCA, PLS, LDA, FDA and CA are based on a linear approach while fuzzy logic, ANN and PNN are regarded as nonlinear methods (Amy et al [12]). This technology is still in the early stages of development, but it promises many applications, such as

- Quality Assessment of beverage products
- Quality control in food processing like taints, adulteration, bacterial spoilage etc.
- Detection, diagnosis and analysis in medicine
- Disaster Response
- Environment Monitoring

III APPLICATION AREA IN FOOD INDUSTRY

The main application areas related to the food industry have been: fish, meat, milk & milk products, wine, coffee, fruits, tea and constitute approximately 5000 publications since 1993. This demonstrates that food applications are central to electronic olfaction and nearly half of the publications are in this area [12]. Within each application in the food industry, research contributions have focused on the detection of a variety of aspects such as freshness, adulteration, off-flavors and bacteria detection. The E-nose evolution for few food products such as milk adulteration, wine and some fruits has been considered in this article.

A. Milk and milk products

Milk is one of the most vulnerable food which can be adulterated very easily with low value ingredients like water and whey into natural milk is known as '**economic adulteration'.** Low priced whey and rennet whey solid are often mixed with liquid milk and milk powder. Sometimes urea is added to milk to increase the shelf life. Milna et al [7] gives an overview of various types of food samples like liquids and solids in dairy industry for adulteration analysis. And summarized the data sets taken from various sensors and combined those data sets with multivariate statistics along with efficient tools for classification, discrimination, recognition and identification of samples. The applications of E-nose sensor in diary industry are

1. **Determination of Milk quality and shelf life:** After detailed study it is concluded that semiconductor thin film based e-nose is most suitable to well distinguish UHT and pasteurized milk during days in which the milk has different rancidity values to well distinguish UHT and pasteurized milk during days in which the milk has different rancidity values.
2. **Differentiation of cheeses** - classification based on the cheese variety, its geographical origin, ripening stage and manufacturing procedure: An electronic nose based on an array of six MOS sensors in combination with artificial neural network (ANN) can be very effective in classification of Pecorino cheeses (typical Italian Pecorino and "fossa" Pecorino cheeses) based on their ripening time and manufacturing techniques and e-nose successfully discriminated between various classes of cheese.
3. **Screening of aroma-producing lactic acid bacteria:** MS-based electronic nose system can be very efficient in discriminating closely related strains and different bacterial species based on their production of volatile compounds.

Huichun et al [8] proposed the method to identify adulterated milk. The objective of author to study this is to monitor the adulteration milk with water or reconstituted milk powder using an E-nose.[8] conducted various experiment using E-Nose and taking milk sample with different level of water, reconstituted milk and concluded that E-Nose could discriminate between skim milk and reconstituted milk by their aroma. E-Nose could discriminate between skim milk adulterated with different volumes of water for milk sample stored for 1-4 days by sensing odor changes during four days duration. Preparation of synthetic milk from liquid-whey is simple and can camouflage the natural milk easily [9]. There are several methods available in the literature to detect different types of adulterants in milk. Few of the methods are

a) Electrical conductivity (EC) method to determine additional water present in the milk.
b) Electrical admittance spectroscopy to detect fat and water content present in milk
c) Freezing point osmometry to determine the additional water content in milk.

A new method named "Capillary Electrophoresis" for determination of whey as an adulterant in milk is proposed by Das et al [9]. This method accurately detects the fraudulent addition of rennet (enzymes for curdling of milk) whey solid in milk kept at ultra high temperature (UHT). For Urea Detection method proposed in literature are [9]:

a) Potentiometric biosensor method
b) Measure of change of pH:
c) Enzyme based piezoelectric sensor
d) Infrared spectroscopy method

Gupta P et al [10] presents the theory, design, fabrication, and test results of a new system, which is used for the detection and measurement of adulteration of clarified butterfat, a classification of anhydrous milk fat.

B. Wine

Currently employed methods for evaluating the aroma and flavor characteristics of wine grapes, such as tasting by winemakers or sensory experts or gas chromatography-mass spectrometry [4], are subjective and/or costly and time consuming. Different classes of reducing gases have different reaction rates at the metal oxide surface. *Amala et al* ***[5]*** proposed metal oxide semiconductor gas sensor based E-Nose technology for grapevines Aroma analysis that can easily detect volatile organic compounds (VOC) like ethanol, acetone and benzene at different concentrations produced at appropriate working temperature. The author has proposed two approaches for studying the sensor response to change in temperature: i) physicochemical modeling. ii) Black box Modeling. *Amalia et al [5]* employed thermally cycled metal oxide sensors to analyze grape juices with differing known qualitative traits. *Matteo et al* [6] proposed constructing an artificial system able to identify different beverages depending on the VOCs that are given off. Samples of **grape juice, wine and vinegar** have been used because they represent the three most important states of wine fermentation. The factors that limit the remote sensing range are also analyzed, estimating the furthest distance where the array could be placed.

C. *Fruits*

E-nose system can detect if the fruit is spoiled or fresh and it can classify different type of fruit products by sensing the aroma. In some applications E-noses can be used to estimate the ripeness and harvest date of the fruits. Jose et al [1] proposed few methods to analyze peer, orange, apple drinking water and truffle classification. The proposed e-nose uses the truffle smell and calculates the exact degree of maturity to determine the ripeness. Once the e-nose has *learned* the characteristics of an optimally ripe truffle, a specialized smell operator is not necessary; rather, the e-nose will provide results in seconds. Jose et al [1] employed the e-nose with 32 sensors to identify the presence of common bacteria of water that increases sulfur and selenium content which results in off-odors usually described by associations to wet cloth, butane, rubber, or rotten eggs. To test the sensibility of the e-nose, molecules at two different concentrations: parts per billion (ppb) and parts per trillion (ppt), were employed. This work showed a high rate of sensitivity and selectivity of the e-nose that is able to distinguish among the different molecules and between the different concentrations. The accuracy of the results is closely related to the statistic test: for low concentration, the probability model showed the best classification.

The method proposed for orange aroma analysis is suitable for predicting the harvest period, shelf life and quality of orange ranging from harvest to till consumer eats.The hand squeezed juice sample, total 10 samples at taken at different time kept at different storage conditions were used for the analysis. The e-nose system using Figaro metal oxide semiconductor, was able to reveal that orange juice flavor changes because of off-odors even when fruits are stored under refrigerated conditions. The e-nose was also tested with two types of fruit: pear and apple. The output of the sensors consisted of 32 independent analog voltages. Each one varied with time and odor. Then pear and apple samples were analyses with WEKA classification algorithms as proposed by [1]. The e-nose can easily distinguish between pears and apples and selected samples were actual pieces of the fresh fruits.

A novel non destructive artificial olfactory system for measuring fruit maturity is proposed by Jesús et al [2]. The fruit samples selected are: Apple (Classification as per shelf life Period), Peer (ripeness state irrespective of harvest date, total 8 samples selected), Peach and Nectarine (for harvest date prediction). A relationship between harvest date and ripeness state based on the type of fruit, harvest period, shelf life associated with an accumulation of aromatic volatiles during ripening for both climacteric and non-climacteric fruit is developed. For fruit ripeness evaluation parameters selected: Firmness (for all fruits), Clorimetery (peaches, pears and nectarines), Soluble solids content (SSC) (for peaches, pears and nectarines) and titratable acidity (TA) (for all fruits), Starch Index (for apples) and Ethylene (for all fruits). Compared to classical and other novel analytical methods E-nose method offers cheap and nondestructive instrument that can be operated by a layman. E-noses proposed for determining fruit quality, shelf life, harvesting period and ripeness are

- Figaro semiconductor type Gas sensors (TGS2610-C00, TGS2611-C00, TGS2600-B00)
- MQ gas sensor (MQ2, MQ3, MQ-303A,MQ4,MQ5)

IV ADOPTED METHODOLOGIES

The detection process of an Electronic Nose system is shown in figure.2. This process is divided into five groups namely, raw measurement, pre-processing, feature extraction, pattern recognition, classification and decision making. The initial block in the figure represents the electronic nose hardware, which typically consists of a gas sensor array. Preprocessing compensate for sensor drift, compress the transient response of the sensor array, and reduces sample to sample variations. Computer processing controls the artificial olfactory system and then Process raw data into useful information for pattern recognition algorithm. With the help of commercial DAS computer can control the complete process. The computer records resistance changes that sensor experience and acquired data is stored as text file. Matlab tools are then used to extract relevant feature of each measurement file and to test different pattern recognition methods [4].

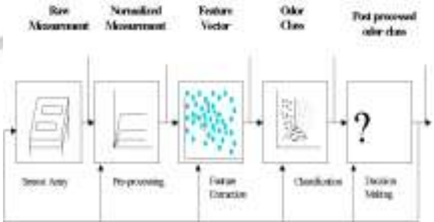

Figure 2: Gas Detection Process [4]

Fiber optic sensor and PCA classification: [5] prepared Four fiber optic sensors based on reflective configuration using standard single mode fiber (SMF). Thus the response signals from all the sensors were transmitted through the same fiber by using an optical wavelength demultiplexer/multiplexer (C31, C35, C42, C47 ITU grid channels, with a 2 nm band pass): together with the sensors, they form the Sensing Unit (sensing array). Amalia et al [5] With grape juice, the signal gets stabilized 0.3 dB over the base line; in case of wine, the power attenuated at 0.7 dB and with vinegar, 0.25 dB. *T*he response from the sensors has been compressed and clustered by applying FFT and PCA. Individually, each sensor is not capable to distinguish the samples, but jointly it is possible to do by training an ANN. This processing eliminates the effect of power fluctuations, making the system more robust and able to identify the beverages. These results encourage the development of arrays with more than 4 sensors with SMF to tasks related with VOCs or gases identification, taking advantage of all the features that optical fiber offers [5].

PEN 2 E-Nose and PCA technique for milk Adulteration: [8] proposed a method in which Skim milk was adulterated with different volumes of water. 5 samples are proposed in this research study by the author. All E-nose experiments were conducted at 20^0c and 50-60% relative humidity. When sensors were exposed to volatiles a computer records the sensor resistance change. This data is recorded for later use. Set of signal from all sensors for a given sample constitute a pattern and pattern of multiple measurement dealing with same sample are stored in pattern file and are known as training set. Further PCA is applied to these data sets; PCA is used for analyzing, classifying and reducing the dimensionality of numerical dataset and allow extraction of useful dataset. Instead of PCA, LDA can also be used. LDA improves resolution of classes as it is able to collect information from all sensors.

Constant Phase Element (CPE) Sensor with Automatic Detection System (for milk) Siuli et al [9] proposed designing of a low cost automated sensing system in which the sensor detects the adulterated milk and a micro-controller based circuit closes the valve installed at the outlet of the milk supply line to prevent mixing. The pictorial view of the scheme is shown in figure below. The sensor, shown in figure is a copper clad printed circuit board, cut into suitable dimensions and coated with a thin film of Polymethyl-methacrylate (PMMA) by using spin coating technique. The sensor with coating thickness 13 µm, named as FOE13, is selected for designing the instrumentation system for detection of tap water and urea as an adulterant in pure milk in the frequency range 5–12kHz.

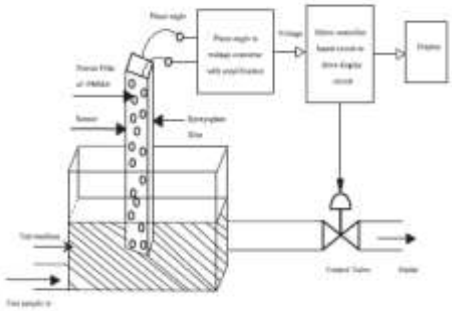

Figure 3: Automatic detection system for milk adulteration

The FOE sensor dipped inside the test milk sample. The output of the sensor is used as input to the phase detector which converts the phase angle into its corresponding voltage output. The output voltage from phase detector circuit is fed into microcontroller. The voltage corresponding to the pure milk is stored in the micro-controller and whenever a deviation occurs with respect to this stored voltage an error signal is generated and the driver circuit drives the control valve (solenoid valve). So, when ever adulteration is detected, the control valve at the outlet of the milk line shuts off the flow of milk to prevent mixing.

Pallavi et al [10] proposed an oscillating system, where motion of pendulum (Uniform metallic rod) is affected by the medium in which it is oscillating. The change in time period was measured and analyzed using Digital storage oscilloscope. experiments were conducted to observe time period of oscillation with no sample, and then different adulterated samples of clarified butterfat, a classification of anhydrous milk fat were used.

V DISCUSSION AND FUTURE PROSPECTIVE

In this article we have tried to summarize the contributions of E-Nose technologies relevant to food processing and adulteration area. Each paper summarized highlight one application to the food sample selected due to the requirement of specific hardware and/or software for each application. On the materials side, major focus must be given to the design and development of drift free sensors that can be used reliably over long temporal horizons. External factors like humidity, ambient temperature and atmosphere, presence of other gases/aromas during standardization procedure, accuracy in analog to digital data conversion process, etc. must be given special attention for enhancing the performance of the sensor system as a whole. It is also concluded that if the temperature and sensor combination could be optimized it would allow the use of only one or two sensors instead of large array saving cost and time. On the software side, researchers apply many of the available linear and nonlinear algorithms (either separately or combined). Clearly, selectivity is also a key issue. In addition, the top five needs in this area of research are

1. Drift free sensors that can be used reliably
2. Investigation of new material for achieving better selectivity
3. Better modeling and correlation between presence of chemical markers and the sensor response in the E-Nose array
4. Application of specific instruments with carefully selected sensor arrays and sampling system
5. Better understanding of the industrial need related to quality control and monitoring of food processing.

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