ELECTRONIC MUCOSA SYSTEM FOR COMPLEX ODOUR RECOGNITION

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Specially dedicated to

My beloved father and mother (Abah & Ma)

Azahar Bin Shaarani

&

Hasnah Binti Mat'il

My beloved father and mother-in-law (Ayah & Mak)

Abdul Jamil Bin Mohd Noor

&

Zaiton Binti Ahmad

My beloved husband

Abdul Azim Bin Abd Jamil

My beloved son

Abdullah 'Ammar Bin Abdul Azim

My Siblings

Mohd Safuwan, Nur Intan Azrina, Mohd Syafiq

For your unwavering love, sacrifice, patience, encouragement and inspiration

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ABSTRACT

Over the years, electronic nose technology has been researched extensively and used in a wide range of applications. Commercial electronic nose system has been in the market for quite some time and is being used in several applications such as environmental, food, medical and perfume industries. However, sensitivities and selectivities of current electronic nose have still not achieved the biological olfactory system. Motivated to improve the performance of current electronic nose, this research discusses the development of electronic mucosa system which is an extention of the electronic nose system. Electronic mucosa system refers to an electronic nose system combined with gas chromatography column to produce extra information, which is spatial and temporal information of odour profile. The main components in electronic mucosa system are four sets of sensor arrays. Each sets containing five metal oxide chemo resistive sensors also known as e2v sensors. Each e2v sensor in an array has different sensitivity. Three gas chromatography columns coated with polar, mid polar and non-polar compound are added in the system to aid the ability of the system to discriminate between simple and complex odour. This electronic mucosa system mimics nasal chromatography effect in human mucous. This system produces spatial and temporal information also known as spatio-temporal information. The spatio-temporal data aid pattern recognition technique and enhances the ability to distinguish more complex odour compared to conventional electronic nose. The electronic mucosa system has successfully discriminated complex odours: lavender, lemon and floral essential oils with small number of sensors with high accuracy. This electronic mucosa system may improve electronic nose technology in the future.

ABSTRAK

Setelah bertahun-tahun, teknologi hidung elektronik telah dikaji secara meluas dan digunakan dalam pelbagai jenis aplikasi. Sistem hidung elektonik komersial juga telah berada di pasaran untuk tempoh yang agak lama dan diguna dalam pelbagai industri seperti alam sekitar, makanan, perubatan dan minyak wangi. Walau bagaimanapun, kepekaan dan pemilihan sistem hidung elektronik masih belum mencapai sistem biologi hidung manusia. Bermotivasikan untuk meningkatkan prestasi hidung elektronik masa kini, kajian ini membincangkan pembangunan sebuah sistem mukosa elektronik sebagai lanjutan kepada sistem hidung elektronik. Sistem mukosa elektronik merujuk kepada sistem hidung elektronik bergabung dengan lajur kromatografi gas untuk menghasilkan maklumat tambahan dalam profil bau. Komponen utama dalam sistem mukosa elektronik ini adalah empat susunan set penderia. Setiap set mengandungi lima penderia kemo-rintangan logam oksida yang juga dikenali sebagai penderia e2v. Setiap penderia e2v dalam set penderia mempunyai perbezaan sensitiviti. Tiga lajur kromatografi gas yang bersalut dengan sebatian kutub, sebatian pertengahan kutub dan sebatian bukan kutub ditambah dalam sistem untuk membantu keupayaan sistem untuk membezakan antara bau yang mudah dan kompleks. Sistem mukosa elektronik ini meniru kesan kromatografi hidung dalam mukus hidung manusia. Sistem ini menghasilkan maklumat ruang dan masa yang juga dikenali sebagai maklumat ruang-masa. Dengan bantuan teknik pengiktirafan corak, maklumat ruang-masa yang dihasilkan dapat meningkatkan keupayaan mukosa elektronik untuk membezakan bau yang lebih kompleks berbanding dengan hidung elektronik konvensional. Sistem mukosa elektronik telah berjaya membezakan bau-bau kompleks: minyak-minyak pati lavender, lemon dan floral dengan menggunakan bilangan sensor yang sedikit tetapi berkepekaan tinggi. Sistem mukosa elektronik ini mungkin boleh meningkatkan teknologi hidung elektronik pada masa akan datang.

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LIST OF ABBREVIATIONS

ABS	-	Acrylonitrile Butadiene Styrene
Carbomax	-	Poly (ethylene glycol)
CO_2	-	Carbon Dioxide Gas
E-mucosa / EM	-	Electronic Mucosa
E-Nose / EN	-	Electronic Nose
GC	-	Gas Chromatography
MICS	-	Metal Oxide Sensor
MOS	-	Metal Oxide Semiconductor
NO_2	-	Nitrogen Dioxide
O_3	-	Ozone Gas
OV-1	-	Poly (dimethylmethylphenyl-siloxane)
PCA	-	Principal Component Analysis
PCB	-	Printed Circuit Board
PDMS	-	Poly (dimethylsiloxane)
PEG	-	Poly (ethylene glycol)
QCM	-	Quartz Crystal Microbalance
SAW	-	Surface Acoustic Wave
STL	-	StereoLithography
VOC	-	Volatile Organic Compound

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CHAPTER 1

INTRODUCTION

1.1 Introduction

This chapter introduces the Electronic Mucosa (E-Mucosa) through extensive literature review of electronic nose system. E-Mucosa will be compared to conventional electronic nose instruments based on their advantages and disadvantages. The objectives and scope of the research are then expressed. Finally, an outline of the thesis is presented.

1.2 Mammalian Olfactory System

There are five basic senses that are common in human and animal sensory system. These senses are sight, touch, taste, hear and smell. Each sense plays an important role in the survival of living things. Odour sensor, to be precise, has a very unique and important role itself. As for animals, odour sensing is created as warning mechanism, for finding food, avoiding predator or choosing a mate (Gardner et al., 1999; Goldstein, 2002).

Meanwhile, in human daily life, smell sense help us to perform many functions such as to detect a gas leak in our car or house, burning food while cooking, check the quality of food and drinks. 75% - 80% of everything we taste involves the sense of smell (Bear et al., 1996). The importance of the smelling sense is also seen in food and beverages industry where it was used to analyse and check the food quality in term of odour and flavour. In the chemical industry, odour sensing is used to detect hazardous chemical in the environment.

The sense of olfaction is complex and works through chemical reaction. It is chemical in nature because it is detecting the chemical in environment. It is called chemical sense because it detects the chemicals in the environment. Like external environment, the odour perception also influent by many factors. Basically, the odour perception is related to chemical molecules in gases state. The chemical molecules can be detected by olfactory receptor and olfactory epithelium. The process of smelling will undergo a few steps systematically which is known as biological olfactory system. The process in biological olfactory system is clearly shown in Figure 1.1.

Nasal cavity of olfactory system is an area near the septum reserved for nasal mucous membrane. Nasal mucous is the membrane where the olfactory receptors and olfactory epithelium are located. This area is called olfactory mucosa. Firstly, when the odorant molecules in the air are smelled, the odorant will bind with the olfactory receptors. Once the receptor received the odorant molecules known as odorant impulse, the olfactory receptor cell become activated and send electrical signals. The signals pass through olfactory epithelium and relayed in glomerulli. Then, the signal flow and transmitted through olfactory bulb to the higher regions of olfactory cortex also known as brain. Lastly, the brain will process the odorant impulse and described the types the odorant smelled to human. There were several features of biological nose that undergo trials over researches under artificial intelligent development in order to get to most nature-like results. This topic will be discussed in Chapter 2 thoroughly.

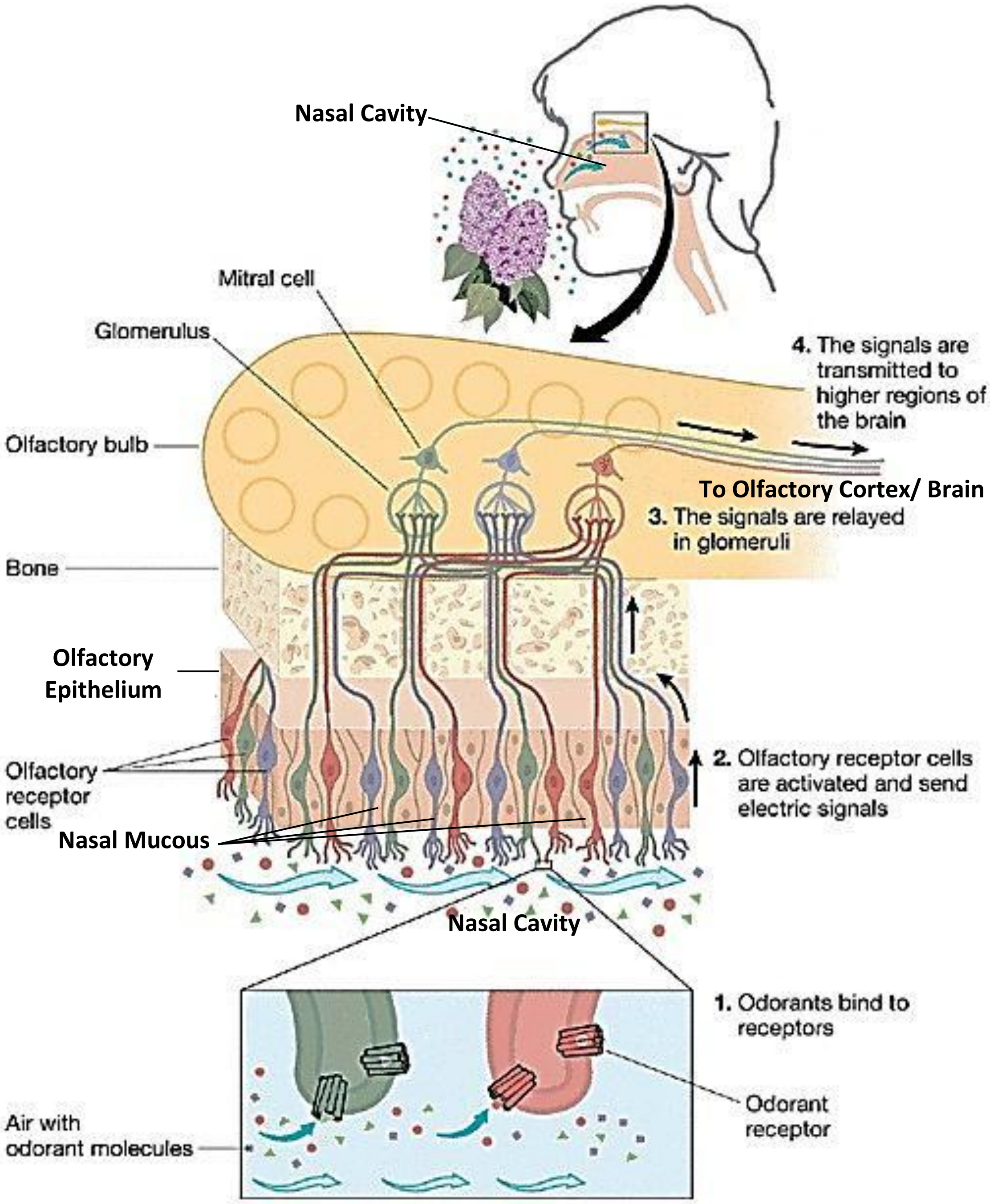

Figure 1.1: Process in Human Olfactory System

Human and animal olfactory systems are different in terms of sensibility. Through the understanding of biological olfactory system research among the animal including insects (Hebb et al. 2002; Vosshall 2000; Keller & Vosshall 2003; Sato et al. 2008), fish (Shoji et al. 1996; Hara 2001; Wilson 2004), dog (Rex 2004; Willis et al. 2004) and mice (Buck 2004; Zhang & Firestein 2002), it can be said that many animal have more sensitive sense of smell compared to human especially dog and rat.

Dogs are 300 to 10,000 times more sensitive depending on the odorant (Heidelberg et al. 1991). While, rats are 8 to 50 times more sensitive to odours than human (Rouquier et al. 2000). Dogs have 1 billion receptor cells while human only has about 12 million olfactory receptor cells and 1000 different receptor protein. Nowadays, because of the high sensitivity and selectivity in their sense of smell, dogs have been used to detect drugs, helping in forensic area and trace the element in airport and customs.

Based on Quignon et al. (2003), the sensitivity of mammalian olfactory system depends on several aspect such as the number of olfactory receptor, size of olfactory bulb, size of olfactory epithelium and the density of neuronal cells. From the research above, it shows that the number of olfactory receptor cell is the most significant factor to the sensitivity of olfactory system.

In analysis and industry, the ability of human and animal olfactory systems was limited at certain purpose. Some analysis and sector in industry are not suitable and also not capable to involve human or animal in the process. For example, it is impossible to use human or animal olfactory to categorize toxic and hazardous odorant in explosive detection and medical industry. Moreover, human and animal olfactory ability are too subjective, unreliable, not accurate, highly cost and prone to fatigue (Burian 2010). For example, to differentiate the maturity of the fruit, human olfactory are not accurate to give the measurement. Different person will give their own opinion based on their experiences but it is too subjective. Because of these, the human and animal olfactory systems are not practical to be used in analysis and industrial area.

Hence, in order to overcome the drawback of human and animal olfactory in analysis and industry, the gas sensing technologies introduced the artificial olfaction. The ideas to mimic the biological nose system drive the technology of gas sensing to invent the artificial nose. It is agreed that the sensitivity of olfactory system depends on the numbers of the olfactory receptor cell (Heidelberg et al. 1991; Rouquier et al. 2000), thus number of sensor will be considered in order to increase the diversity of detection of artificial nose system.

1.3 Electronic Nose

The electronic nose is a bio-mimetic device which described to be able to mimic the human sense of smell. The idea of this system and device was started late 1980s and first created in early 1990s. Basically, human nose have around 100 million olfactory receptor cells which is glomeruli nodes, mitral cells and tufted cells (Gardner 2003) and 300 distinct genes. The genes will encode olfactory receptor proteins and improve the specificity of olfaction. Electronic Nose is an instrument that requires a sensor array combined with a pattern recognition system to be functioning. Since the human olfactory system have 100 million of receptor and each of the receptors are very sensitive to great number of compound, each individual sensor in the array of electronic nose system needs to be differently coated so that it can provide partially different information between each other.

1.3.1 Commercial Version of Electronic Nose

In advance technology of gas sensing, the electronic nose have been used to discriminate a diversity of odours in wide range of application (Dodd et al. 1982; Gardner 1999; Pearce et al. 2003). However, the solid state sensor in electronic nose have serious limitation such as low sensitivity and selectivity, drift problem, difficult calibration and the sensor have short life span. Therefore, to overcome the problem of solid state sensors and improve the ability of electronic nose, a new electronic nose is being introduced by using different sensors as a new approach.

In mid-1990s, the first commercial version of electronic nose, namely the Fox 2000, from Alpha MOS reached the market (J.W. Gardner 2001). The Fox 2000 have been mainly used in the food industries area. In advanced technology of electronic nose, there are more than 10 companies who designed and established their own version of electronic nose. The passing years recorded the evolution of commercial electronic nose starting from desktop size to portable handheld size instrument (Che Harun, Covington, et al. 2009). Table 1.1 shows some commercial electronic nose instrument.

Table 1.1: Commercial Electronic Nose

Device	Manufacturer	Sensor Type	Sensor Number	Size
Fox 2000,3000, 4000	Alpha MOS	Metal Oxide Sensor/Surface Acoustic Wave	6,12,18	Desktop
z-Nose 4500	EST	GC/Surface Acoustic Wave	2	Laptop
BloodHound ST214	University of Leeds Innovation Ltd	CP	14	Laptop
Heracles	Alpha MOS	GC-MS	-	Desktop
Smart Nose	Smart Nose Inc.	MS	-	Desktop
Cyranose 320	Smith Detection	Carbon Black	32	Portable
PEN-2 WMA	AIRSENSE ANALYTICS	Metal Oxide Sensor	10	Portable
HAZMATCAD, CW Sentry 3G	Microsensors System	Surface Acoustic Wave	-	Portable
SensorFreshQ	FQSI	-	-	Portable
LibraNose	TECHNOBIOCHIP	Quartz Crystal Microbalance	8	Portable
e-Nose 5000	Marconi Applied	Conducting Polymer	12	-
AromaScanner	AromaScan	Conducting	32	Portable

Model A32S		Polymer		
The Nose	Neotronics	Conducting Polymer	12	Portable

Source: (Che Harun, et.al 2009)

Each of these commercial electronic noses has its own advantages and disadvantages. In order to overcome the drawback from the latest electronic nose version, new version of electronic nose was invented. Throughout the years, the first invention of commercial handheld electronic nose was developed with 32 carbon-black polymer composite sensor array used in the system.

Cyrano Science Company (USA) introduced the first portable electronic nose, Cyranose 320. Cyranose 320 system is the conventional electronic nose in market currently. This electronic nose system utilize up to 32 chemo resistive sensor in array which built up from carbon and polymer element. It is rugged, handheld and portable electronic nose tool.

Cyranose 320 is being used in variety of industry such as food and beverage (Casalinuovo et al. 2006), medical diagnosis (Boilot et al. 2002; Pavlou et al. 2002; M.E.Shykhon et al. 2004; Dutta et al. 2005), chemical (Henderson et al. 2010; Dutta et al. 2004; Rains et al. 2004), food industry (Schaller et al. 1998, Erika Kress-Rogers 2001), packaging material (Werlein 2001; Rajamäki et al. 2006), agriculture (Gardner et al. 1992; Magan & Evans 2000; Eklo et al. 1998) and many more.

In more recent years, the idea of mimicking the biological olfactory system keeps evolving. Many researches are moving forwards trying to imitate the biological olfactory system into commercial olfactory instrument (M M Mozell 1970; Maxwell M Mozell et al. 1987; M. a. M. J. M. M 1973).

It is known that the number of the sensor gives the vital factor to the sensitivity of the electronic nose system. However, there are also several researches done and proved that biological mucus odour retention effect will give extra information and improve the odour discrimination in electronic nose system (C.J.Lu 2003; Bhushan et al. 2006; Ragazzo-Sanchez et al. 2005; F.K. Che Harun, J. a. Covington, et al. 2009). Thus, in striving to improve the electronic nose instrument, the new technology of gas sensing has come out which is electronic mucosa system.

1.4 Electronic Mucosa

Conventional electronic nose system requires sensor array to be combined with pattern recognition system. While for electronic mucosa, it is the combination of array of sensors and gas chromatography column with pattern recognition system. This additional retentive column in the electronic nose system is actually mimicking the biological nasal mucous in human olfactory. The function of chromatography column in the system can give extra information from the odour. This information is an important feature for advance odour distinguishing task.

The electronic nose system produced spatial information as output while electronic mucosa system produced spatial and temporal information as the result. The spatial and temporal information (spatio-temporal information) gain from nasal chromatography effects by adding the array of sensor and retentive column.

'Spatio-temporal' data term used here refer to the output response from the electronic mucosa system. The data consist both information, magnitude of the response and time for response delay. In Chapter 3, the gas chromatography column principle and spatio-temporal information will be described in detail.

1.5 Problem Statement

Nowadays, development of electronic nose has already led to significant advances in the field and being used in various industry such as food, medical and agriculture area. However, sensor based electronic nose today generally suffer from significant weakness. Table 1.2 shows the problem of electronic nose system found by previous researchers.

Table 1.2: Problem of Electronic Nose System

Problem of System	Ways to Overcome	Weakness
- Widespread applications of electronic nose are limited. - Difficult for electronic nose to detect the odour in very low concentration (below ppm) of odour in air.	Developed expensive auto samplers and supply clean air to the electronic nose.	The electronic nose becomes bulky and expensive.
- To overcome bulky weakness, the electronic nose system comes out with the small number of sensors. - However, is not easy to distinguish odours with similar properties.	In order to mimic million receptors in human olfactory system, electronic nose system used large number of sensor.	- However, electronic nose sensing tends to heavily affected by environmental factor which is general drift due to temperature, humidity and background nose. - Design of electronic nose become complex with large number of sensor and component.
To overcome complex system problem, the	The system developed with the variety of chemo	However, for the time being, chemo resistive

number of sensor and component have to reduce.	resistive sensor coated in the system, it can provide partially different information between each other.	sensor elements are limited. There are only several types of metal oxide semiconductor (MOS) material that suitable and being used.

The most vital problem is that the widespread applications of electronic nose are limited. It is difficult for the electronic nose to detect the odour in very low concentration (below ppm) of odour in air. In order to overcome the problem, previous researcher developed expensive auto samplers and clean air supply to the electronic nose. Unfortunately, the electronic nose becomes bulky and expensive (J.Covington et al. 2012).

Hence, to overcome the problem, the artificial olfaction comes out with the small number of sensors. However, it is not easy to distinguish odours with similar properties. Since human olfactory system has 100 million of receptors, the researchers mimic the idea onto advance electronic nose. In advance, the artificial nose system develops 100 – 1000 chemo resistive sensors in sequence to mimic and to get similar response as human nose. Due to the large number of sensor, its sensing tend to heavily affected by environmental factor such as general drift due to temperature, humidity and background odour. Besides, the design of electronic nose becomes complex with a large number of sensors and components.

In order to mimic 100 million of human olfactory system, each sensor in the array needs to be coated individually. With the variety of sensor coated in the system, it can provide partially different information. However, currently, chemo resistive sensor elements are limited. There are only several types of metal oxide semiconductor (MOS) material that suitable and being used. For instance, carbon black polymer, zinc oxide and tin oxide (Wilson & Baietto 2009; Wilson & Baietto 2011; Eklo et al. 1998; Casalinuovo et al. 2006).

This challenge provides chances for more innovative invention for electronic nose. A novel mechanism is being designed to deal with this issue and improve the sensitivity and selectivity of bio-mimetic devices. The novel device developed known as electronic mucosa, where the electronic nose system combines with gas chromatography columns. More recently, it has been suggested that chromatography column plays a role in odour discrimination within the biological olfactory system as mention before, nasal chromatography effects.

1.6 Objective of the Research

There are two ultimate target of the study. First objective is to develop e-mucosa prototype with retentive columns. The retentive columns used are coated separately from each other. There are coated with polar, mid polar and non-polar compound. By using different coat of gas chromatography column, this study will identify the suitable column depending on the sample of odour used.

Second objective of this project is to evaluate the performance of multi-dimensional e-mucosa system on essential oil. The system will be applied onto essential oil sample under controlled condition to monitor its effectiveness. Principal Component Analysis (PCA) technique is used to represent the data in classification of essential oil.

1.7 Scope of Study

There are three main parts of this study which are the development of the system hardware, the development of system software, and testing of sample. The electronic mucosa system circuit consists of an array of sensors circuit and retentive column which will be designed and developed. There are a lot of odours in the environment, carried by gases around us. These environmental gases may affect the response of the sensor. Thus, a chamber has been designed to put on each of sensor array.

Second part of this study is development of the system software. Data acquisition board, National Instrument M-Series PCI-6229 Multifunctional Card will be connected between the sensor circuit and computer system. LabVIEW software programming will be used to collect and display the data and signal responses from each sensor. In order to analyze the data, MATLAB programming and Principal Component Analysis (PCA) will be used.

Third part of this study is testing on samples. Several experiments will be conducted using several sample of fragrance as test subjects to measure the response of the gas sensor. Data will be recorded into a data acquisition system for further processing.

1.8 Concept of Project

The sniffing process happens in nasal cavity which is area near the nasal mucous. Olfactory receptor and olfactory epithelium locate at nasal mucous. When the odour is sniffed by human nose, the odour will flow through nasal cavity and go to olfactory receptor. Olfactory receptor neurons will react and generate the signal. The signal will pass through olfactory epithelium. Then, the signal will pass over olfactory bulb to the olfactory cortex/brain. Signal produce will be sent to brain for the signal recognition.

The same process goes to the concept in electronic mucosa system as shown in Figure 1.2. The test odour will collect and passing by using pre-concentrator. The test odour will pass to receptor of electronic nose system which is replaced by an array of chemo-sensor. The tested odour also passes over chromatography column which play a role as nasal mucous in the nature. When an array of chemo-sensor in large sensor arrays 'smells' or detect the odour, these chemo-sensor element will also react and produce the signal. The signal produced will be processed and identified by pattern recognition system in the computer. The pattern recognition system worked as a brain for electronic nose system.

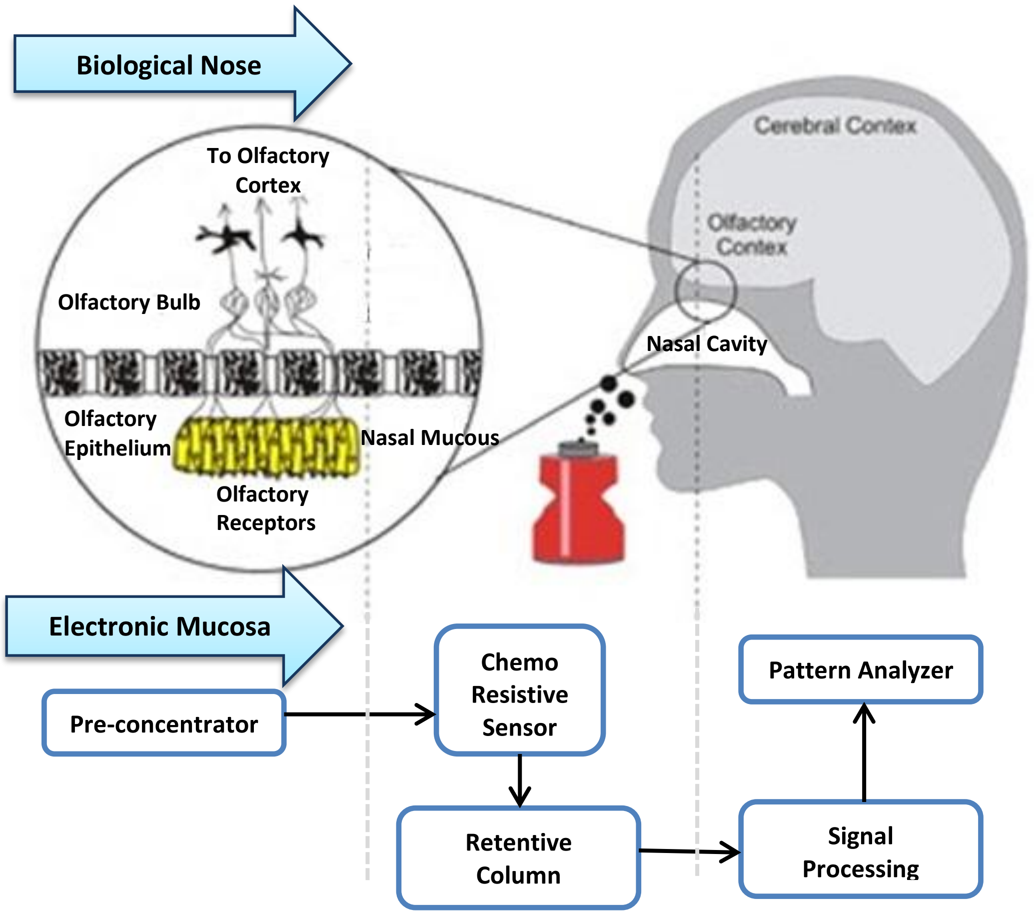

Figure 1.2: Concept of Electronic Mucosa

Figure 1.3 illustrates a block diagram of electronic mucosa system. The system develop comprising an arrays of sensor covered with the chamber sensor, gas chromatography columns, an interface of printed sensor circuit board (PCB) combine with pattern recognition system. Pattern recognition system involve data acquisition card (DAQ) and a laptop with self-developed of LabVIEW program to collect the data response from the sensors.

This electronic mucosa system consist four arrays of sensor. Each array of sensors has five different sensors which are made from e2v sensor. Initially, the odour sample will pass over the first array of sensor. In this stage, the sensor response data will give magnitude sensor response as the output. It is also known as spatial information.

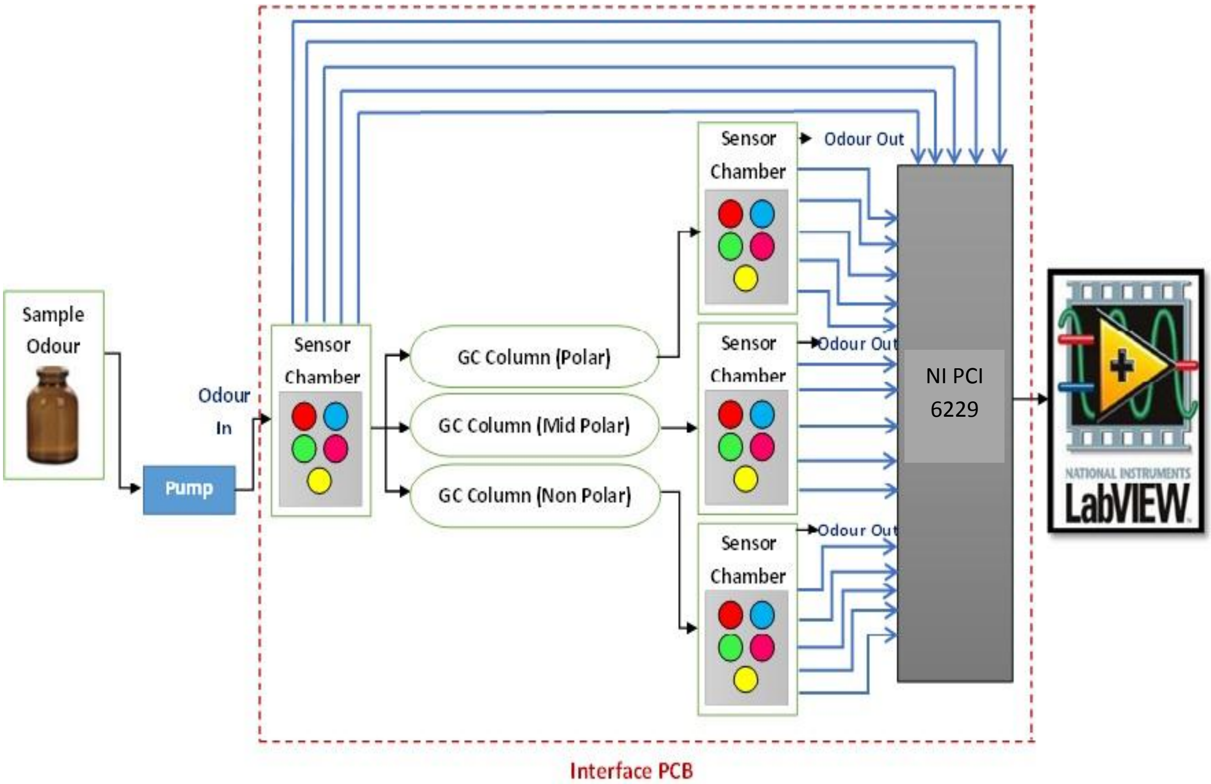

Figure 1.3: Block Diagram of Electronic Mucosa System

Then, the odour sample will be divided and pass along over the three retentive columns. The three retentive columns are the CARBOMAX, OV-1 and PDMS coated respectively. CARBOMAX, OV-1 and PDMS coated are columns that are coated with polar, mid polar and non-polar compound. Due to column principle, the odour which contain non-polar molecule will react with non-polar column and produce time delay effect. While for the odour which contain polar molecule will react with polar column and produce the same effect. Time delay effect will produce the signal response have retention time. It is will be clear if we see from the graph, the signal response before the odour pass over the column and after the odour pass over the column. This effect will discuss details in Chapter 4.

Lastly, the odour sample passes through the column will pass over the three sets of additional sensor array which is second, third and fourth sensor array at the end of sensor circuit system. All of them are a set of e2v sensors same like set of sensor in the first array. However, in the last three sets of sensor array, it will give spatio-temporal information as the output of the system. Spatio-temporal information of the system will give the magnitude and retention time of each gases flow through gas chromatography column.

The signal response data (spatial and temporal information) from the sensor will be sending to the laptop through National Instrument data acquisition card, M-Series PCI-6229 Multifunctional Card (NI PCI-6229). It is combining with self-develop of LabVIEW program to characterize sensor and odour data. The odour data collected which is spatial and temporal data are in Microsoft Excel format. The data collected is used for data processing using MATLAB software. In MATLAB, the data signal response from the sensor will be plotted in graph. Then, the temporal data will be measure manually based on the graph produce from the data. The spatial and temporal data produce from data processing part will be arranged properly in Microsoft Excel format before used for pattern recognition part. Principle Component Analysis (PCA) method is used to play roles likes a brain for pattern recognition part. PCA will process the spatial and temporal data form data processing part and represent the data in classification of odour sample. PCA will illustrate the odour classification in two-dimensional axis.

1.9 Outline of Thesis

The thesis is classified into five chapters which are the introduction, literature review, methodology, result and discussions, and conclusions. The outline of the thesis can be composed as follows;

Chapter 1 reviews the electronic nose and the weakness of the system. Then, introducing of electronic mucosa system and compared with electronic nose instruments. Then, the objectives and scopes of research are the presented.

Chapter 2 reviews the previous studies about electronic nose instruments involved with an array of sensor. The functionality and details on chromatography column was discussed in this chapter.

Chapter 3 describes the design and fabrication of the chemo sensor arrays that consists of sensors coated with different sensitive respectively. The sensor circuit developed will combine with different stationary phase of retentive column. This chapter also discusses about the types of retentive column used in this system.

Chapter 4 presents the result of sensor array characterization. The central focus here has been geared towards getting spatio-temporal information signal from the E-Mucosa system.

Chapter 5 presents the concluding remarks on the research work as well as recommendation for future studies.

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