SENSOR RESPONSE ANALYSIS, FEATURE EXTRACTION AND CLASSIFICATION OF VOLATILE ORGANIC COMPOUNDS

BY

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ABSTRACT

This project focuses on analyzing sensor response from different types of functionalized reduced Graphene Oxide (rGO) based VOC sensors on three selected VOC gases (acetone, toluene and isoprene), performing feature extraction and employing supervised learning for VOC classification. The rGO as sensing material was functionalized with nanoparticles (NPs); such as gold (Au), silver (Ag) and platinum (Pt) and plasma treatment; such as ammonia (NH_3), hydrogen (H_2) and Octafluorocyclobutane (C_4F_8). The sputtering duration and relative frequency power (W_{RF}) are varied for the functionalization of the nanoparticles while the temperature is varied for the plasma treatment functionalization. The sensor response then was measured from the change of resistance signal during the presence of the VOC gas at low concentration, from 1 to 6 parts per million (ppm). Sensors with thin-film from rGO/Au NPs, rGO/Ag NPs, rGO/Pt NPs, rGO/H_2 (RT) and rGO/ C_4F_8 had shown a good response toward the VOC gas while sensor with rGO/NH_3 and rGO/H_2 (except for the RT recipe) showed poor responses. Sensors that have a good response then proceed with the analysis, feature extraction and machine learning part. Average Resistance value at the presence of clean dry air (CDA) only, Rair and in the presence of the VOC gas, Rgas were extracted from the original sensor signal and manipulated into 10 new features. Then, five supervised learning models such as k-Nearest Neighbors (kNN), Random Forest (RF), Logistic Regression (LR), Support Vector Machine (SVM) and Artificial Neural Network (ANN) were benchmarked for the VOC classification task. The model performances were evaluated using k-Fold Cross-Validation and the prediction of the classification are visualized by using a Confusion Matrix. The results showed that RF and kNN have good performances with a mean of accuracy and standard deviation, 0.813 ± 0.035 and 0.803 ± 0.033, respectively. However, ANN, LR and SVM (Polynomial kernel) showed poor performance with 0.447 ± 0.035, 0.403 ± 0.041 and 0.419 ± 0.035 respectively. Based on the reported performance, it shows that 2 out of the 5 models could deal with the feature selected in the VOC dataset and it is feasible to analyze the classification of VOC gases based on single sensor arrays. It is therefore interesting to explore the analysis of combined sensor arrays for such tasks in future research.

ملخص البحث

تبحث هذه الدراسة في استجابة المستشعر من أنواع مختلفة من مستشعرات المركبات العضوية المتطايرة المختزلة الوظيفية، على ثلاث غازات مختلفة من المركبات العضوية المتطايرة (الأسيتون والتولوين والأيزوبرين)، بالإضافة (rGO) القائمة على أكسيد الجرافين كمادة استشعار مع الجسيمات النانوية rGO إلى استخراج الميزات والتعلم الخاضع للإشراف لتصنيف المركبات العضوية المتطايرة. تم توظيف والهيدروجين (NH_3)، بالإضافة إلى معالجة البلازما، والتي تضمنت الأمونيا (Pt)، والبلاتين (Ag)، والفضة (Au)، مثل الذهب (NPs)، في حين (W_{RF}) بالنسبة لوظيفة الجسيمات النانوية، يتم تغيير مدة الرشوقوة التردد النسبي. (C_4F_8) وثماني فلورو سيكلوبوتان (H_2)، تتنوع درجة الحرارة لوظيفة معالجة البلازما. ثم تم تحديد استجابة المستشعر عن طريق قياس التغيير في إشارة المقاومة عندما كان غاز المركبات العضوية المتطايرة موجودًا بتركيزات منخفضة، تتراوح من 1 إلى 6 أجزاء في المليون (جزء في المليون). مستشعرات الأغشية الرقيقة المصنوعة من rGO / Au NPs وrGO / Ag NPs وrGO / Pt NPs وrGO / H_2 (RT) وrGO / C_4F_8 لديها استجابات جيدة لغازات المركبات العضوية المتطايرة، ولكن المستشعرات المصنوعة من NH_3/rGO و rGO / H_2 (RT) باستثناء لوصفة استجابات ضعيفة. تم إجراء التحليل واستخراج الميزات والتعلم الآلي على أجهزة استشعار ذات استجابة جيدة. تم أخذ متوسط Rair، متوسط قيمة المقاومة في وجود غاز المركبات العضوية المتطايرة، من إشارة Rgas، و (CDA)، قيمة المقاومة في وجود هواء جاف نظيف المستشعر الأصلية وتم اختبارها في 10 خصائص جديدة. بالنسبة لتحدي تصنيف المركبات العضوية المتطايرة، تم اختبار خمسة نماذج تعليمية خاضعة للإشراف: k-Nearest Neighbors (kNN)، Random Forest (RF)، آلة المتجه الداعمة (SVM)، الانحدار اللوجستي، والشبكة العصبية الاصطناعية (ANN). تم تقييم أداء النموذج باستخدام k-Fold Cross-Validation (k = 10) وتم عرض تنبؤات التصنيف باستخدام مصفوفة الارتباك. بمتوسط دقة وانحراف معياري قدره 0.813 ± 0.035 و 0.803 ± 0.033 على التوالي، أظهرت النتائج أن RF و kNN يعملان بشكل جيد. من ناحية أخرى، أداء ANN وLR وSVM كان ضعيف، حيث بلغ 0.447 ± 0.035 و 0.403 ± 0.041 و 0.419 ± 0.035 على التوالي. بناءً على النتائج، كان اثنان من خمسة نماذج قادرين على التعامل مع الميزة المحددة في مجموعة بيانات المركبات العضوية المتطايرة، مما يشير إلى أنه من الممكن فحص تصنيف غازات المركبات العضوية المتطايرة باستخدام مصفوفات استشعار واحدة. في البحث المستقبلي، سيكون من الرائع النظر في تحليل مصفوفات المستشعرات المختلطة لمهام مماثلة.

APPROVAL PAGE

I certify that I have supervised and read this study and that in my opinion, it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a thesis for the degree of Master of Science (Computational and Theoretical Sciences)

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DECLARATION

I hereby declare that this dissertation is the result of my own investigations, except where otherwise stated. I also declare that it has not been previously or concurrently submitted as a whole for any other degrees at IIUM or other institutions.

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In the name of Allah, the Most Gracious and the Most Merciful.

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CHAPTER ONE

INTRODUCTION

OVERVIEW

Due to the development of nanotechnology, the implementation of a sensing system has been beneficial to the medical fields where it is being used in medical devices to diagnose pulmonary diseases by detecting volatile organic compounds in human exhaled breath. The sensing systems such as gas sensor array or electronic nose is used as a gaseous compound detector to identify numerous respiratory-related diseases noninvasively, such as like asthma and lung cancer (Feng et al., 2019). It is more portable and has low power consumption compared with conventional methods such as gas chromatography-mass spectroscopy (GC-MS). However, the gas sensor also meets a few challenges in areas such as a lack of sensitivity, sensor drift and stability (Yi & Li, 2019) (Vergara et al., 2012). The generated data response from the sensor may contain high noise and irrelevant information. Therefore this project focuses on analyzing sensor responses from different types of functionalized nanomaterial reduced Graphene Oxide (rGO) sensors on three selected VOC gases (acetone, toluene and isoprene), performing feature extraction and employing machine learning for gas classification.

1.1 RESEARCH BACKGROUND

Volatile organic compound (VOC) is a wide class of chemical compounds containing carbon with a high vapour pressure at room temperature (Jalal et al., 2018). High concentration VOCs present in the air could be dangerous and become toxic especially for human health (Khalaf et al., 2008). VOCs have been used as preclinical biomarkers in breath analysis to monitor health and diagnose various pulmonary diseases such as asthma and lung cancer (Krisher et al., 2014) (Dragonieri et al., 2017) (Thriumani et al., 2018)(C. Chen et al., 2020).

An electronic nose (e-nose) is an artificial olfactory sensor system designed to detect and differentiate a wide variety of gas compounds. The device which is inspired by the olfactory system of humans or mammals (sense of smell) is composed of a collection of an array of gas sensors with a pattern recognition system (Hu, Wan, Jian, Ren, Jin, Su, & Bai, 2019). The gas sensor is a main component in the sensing system and e-nose application.

Chemoreceptive or resistive type gas sensors based on metal oxides are widely used and have been developed for the electrical detection of targeted gas molecules (Mirzaei et al., 2019). The detection of the gas compound occurs when the gas interacts with the surface of the sensor sensing element by absorption and adsorption, thus generating a change of the electrical conductivity as an output signal (Kim & Lee, 2012). Oxidizing or reducing gases will change the oxygen amount on the sensing surface and change the resistance of the sensor as a signal output (Bastuck, 2019).

The combination of the chemical sensing element of the sensor and machine learning principles makes an e-nose a powerful tool for the detection and recognition of gas samples with concentration estimation. Machine learning algorithm processes the joint input and output of the sensor array to classify the target and/or quantify the samples (Gongora et al., 2018).

However, to improve the application of sensing systems in medical devices, there is an urgency to design VOC sensors with high sensitivity. Reduced Graphene Oxide (rGO) is reported as one of the promising gas sensor materials that have good sensitivity towards a VOC gas and can detect the gas at room temperature (Lu, Ocola, & Chen, 2009). Functionalization rGO with other metal nanoparticle and plasma treatment has been used as an effective approach to enhance the sensitivity of sensing material to a wide range of chemical compounds (Liu et al., 2019).

In this research, VOC gas such as acetone, toluene and isoprene with a low concentration, 1 to 6 ppm were tested individually on a VOC sensor from different functionalization of rGO as the sensing material. The VOC test was conducted at low operation conditions with room temperature 30°C and 40% of relative humidity (RH). The sensing material of the VOC sensor comes from functionalized rGO with nanoparticles such as gold (Au), silver (Pt), platinum (Pt) and plasma treatment such as hydrogen (H_2) and Octafluorocyclobutane (C_4F_8) with different recipes of functionalization. The sputtering duration and power are varied for the nanoparticle functionalization and temperature are varied for plasma treatment functionalization. Then, selected sensors that have good responses are proceeded with data analysis, feature extraction and machine learning to perform the gas classification task.

Hence, this project reports a preliminary result to test the Proof of Concept (PoC) of applying machine learning to individual sensors for VOC classification. Five Supervised learning algorithms such as k-Nearest Neighbors (kNN), Random Forest (RF), Logistic Regression (LR), Support Vector Machine (SVM) and Artificial Neural Network (ANN) were used for the classification task. The classifier models are then put into the comparison to determine which model provides the best evaluation and high accuracy in performing the classification from the processed sensor data.

1.2 PROBLEM STATEMENT

In this era of the Internet of Things, there is an increase in demand for the high accuracy of e-nose in gas detection. The VOC sensor as a sensing unit faced a few limitations such as lack of sensitivity (Yi & Li, 2019) (Vergara et al., 2012). Besides, generated sensor responses from gas sensor arrays have problems in the redundancy of data and noise-contained signals. Many feature extraction methods have been reported (Yan et al., 2015) but no optimal feature has been concluded/ found due to various types of sensors with different types of sensing material used in e-nose application. Meanwhile, feature selection has a definite relationship to optimizing the performance of pattern recognition algorithms that uses classifier techniques for gas recognition (Yan et al., 2015).

Data collection from a gas sensor array can also be cumbersome and time-consuming which poses a nuisance in employing data-hungry machine learning algorithms. Hence, applying supervised learning as a classifier technique remains a challenge (Francesco et al., 2017). The focus of this research is on the preliminary development of sensor array using machine learning which involves analyzing and classifying responses from individual sensors to provide a Proof of Concept of classification of VOC gas from rGO based VOC sensor.

1.3 RESEARCH OBJECTIVES

This research is conducted to achieve some objectives, which are:

1. To analyze the response of targeted VOC gas on the different types of functionalized reduced graphene oxide (rGO) based VOC sensor.
2. To perform feature extraction of low concentration VOC gas from the resistance signal of the sensor.
3. To implement supervised learning algorithms as a classification tool in classifying the type of VOC gas.
4. To evaluate and compare the performance of the proposed machine learning model for the VOC classification.

1.4 RESEARCH QUESTIONS AND HYPOTHESIS

1. Which functionalized sensing element showed a good response on the selected VOC gas?
2. What is the best feature that can provide pertinent information for the gas system analysis?
3. What is the suitable algorithm used in the development of machine learning for VOC classification?
4. Which model will perform the best evaluation and accuracy in classifying the VOC gases?

It is hypothesized that the types of VOCs can be differentiated using raw signatures of the sensor array value as well as its secondary features from the original response curve as input data to machine learning algorithms. However, it is important to account for the significant amount of noise in the sensor data that can be proven severe to classification algorithms.

1.5 RESEARCH SCOPE AND LIMITATIONS

This project focuses on the study of sensor response from different functionalization of rGO based VOC sensors, performing feature extraction from the raw signal and employing supervised learning for VOC classification. The detection of the VOC gas was performed on VOC sensors of various functionalized rGO. Reduced Graphene Oxide as a sensing layer was integrated on a Ti/Pt interdigitated electrode (IDE) and deposited on a SiO_2/Si substrate. The VOC test was conducted at room temperature 30℃ and in the presence of relative humidity below 40% RH. The targeted VOC gas used are acetone, toluene and isoprene which have been suggested as pulmonary disease-related biomarkers (Liu et al., 2019) with the concentration level ranging from 1 to 6 ppm.

Supervised learning algorithms such as K-Nearest Neighbors, Random Forest, Logistic Regression, Support Vector Machine and Artificial Neural Network were used to demonstrate the PoC of the gas classification. The classifier models were performed by using Python 3.6.1 programming language and grid search for parameter setting. The models were then evaluated in terms of accuracy to study the model performance.

The limitations faced in this research are the time consumption for data collection, the minimum number of experiments and noise that keeps coming from the gas sensing instrument and mixed with the measured value. Besides, the VOC gas that was used in the experiment is designated. Thus, it took a few months to order and receive a new stock of gas which lead to the postponement of the experiment. Other than that, this research was performed during the Covid-19 (2020) pandemic which resulted in only a handful of experiments conducted due to the instruction of the government to work from home (March 2020 until June 2020).

CHAPTER TWO

LITERATURE REVIEW

2.1 INTRODUCTION

This chapter discusses the related aspects in the research which are the definition of volatile organic compound (VOC), VOC gas detection device and also machine learning as an analyzer of gas sensor data.

2.2 VOLATILE ORGANIC COMPOUND

Volatile organic compound (VOC) is a wide class of chemicals compound containing carbon with relatively low molecular weight and high vapour pressure at room temperature (Jalal et al., 2018)(Sun et al., 2016). A high concentration of VOCs present in the air could be dangerous and become toxic especially for human health (Khalaf et al., 2008). VOC gaseous molecules originate from two sources which are endogenous and exogenous.

Exogenous VOCs are from external sources related to environmental exposures such as from the emission of fuel and also consumer products including paints, cleaning agents and many more (Ghimenti et al., 2013). The consumption of many exogenous VOC-related products has led to VOC exposure around affected vicinity that can harm the environment as well as bring negative impact to human health (Nurmatov et al., 2015). For instance, exogenous VOC are benzene and naphthalene, trichloroethylene and tetra- chloroethylene, chloroform and haloketones, toluene, ethylbenzene, and m-xylene (Gaude et al., 2019).

On the other hand, endogenous VOCs gaseous are from internal metabolic products produced in the human body which can be detected from exhaled breath, blood, urine, faeces, skin or sweat (Jalal et al., 2018)(Hu, Wan, Jian, Ren, Jin, Su, Bai, et al., 2019). The production of endogenous VOCs in human breath is linked with metabolic activity in the body and provides information about an individual's health condition. Therefore, VOCs have been used as preclinical biomarkers in breath analysis to monitor health and diagnose various pulmonary diseases (Krisher et al., 2014) such as asthma, lung cancer and chronic obstructive pulmonary disease (COPD) (Thriumani et al., 2018)(Dragonieri et al., 2017)(C. Chen et al., 2020). For example, endogenous VOC are acetone, isoprene, acetaldehyde, ethanol, acetic acid, phenol and 2-propanol (Ghimenti et al., 2013)(Gaude et al., 2019).

2.2.1 VOC for Pulmonary Disease

Based on National Cancer Institute Dictionary, pulmonary disease or also called respiratory disease is a condition affecting a human's respiratory system, specifically the vital organ called the lung. VOCs can be found not only in exhaled breath but also in other body fluid samples such as urine and faeces (Wilson, 2018) caused by normal and disease-related pathways. The production of endogenous VOCs is linked with metabolic activity in the human body where they can be formed by bacteria or by the biological process in the liver, kidneys and pancreas (van de Kant et al., 2012). Significantly, VOCs have been used as preclinical biomarkers in breath analysis to monitor health and diagnose various pulmonary diseases noninvasively (Krisher et al., 2014)(Vergara et al., 2007) (Raquel Cumeras, R. and Correig, X., 2019).

VOC metabolites are identified as possible biomarkers and chemical indicators of specific diseases through many kinds of research that study the features of VOC profiles (disease-specific aroma signatures) of the diseases. For example, in one study, the VOC profile of an ill patient was compared to the normal VOC profile of a healthy individual (Wilson, 2018). Due to its volatile properties that can be obtained noninvasively which bring useful for early diagnosis, VOC has become the centre of interest among researchers from various fields. Based on Electronic Supplementary Material (ESI) for Chemical Society Reviews (2018), some of the VOCs that are related to asthma and COPD disease is ethene, nitric oxide and hydrogen peroxide which can be found in a breath sample. Table 2.1 showed a list of VOCs found in the breath as biomarkers for other corresponding diseases in which shows Nitric oxide as one of the VOC biomarkers for Asthma disease (Krisher et al., 2014).

Table 2.1 List of VOCs Linked with Metabolic Disorder with Average Concentration That Found in Human Breath (Krisher et al., 2014)

Biomarker	Ailment (level of a biomarker)
Acetone	Diabetes (above 2 ppm*) [9]
Ammonia	Renal failure; ulcers related to bacterial infection (1500-2000 ppb* for both) [10]
Carbon disulphide	Schizophrenia; coronary disease (5.25 pmol l^{-1*}) [11]
Carbonyl sulphide	Liver diseases [12]
Hydrocarbons	Cancer (1-20 ppb) [13]; heart transplant failures (200-1000ppb) [14]
Hydrogen cyanide	Pseudomonas aeruginosa for Cystic Fibrosis (13.5ppb) [15]
Nitric oxide	Airway inflammation (30 ppb) [16]; Asthma (30 ppb) [17]

*ppb=part per billion, ppm=part per millions, pmol l^{-1}= picomoles per litre

2.3 Conventional Method for Gas Detection

Due to the development of nanotechnology, there has been a resurgence in the growth of biosensors to detect various odours or volatile compounds in areas involving industrial safety, environmental analysis and food industry and in medical diagnostic tools.

In the medical field, breath analysis has been used as a quick and non-invasive approach in medical diagnostic procedures in detecting VOCs. There were a few conventional methods used in analyzing VOC from a complex mixture of gas (exhaled breath)(Queralto et al., 2014) such as gas chromatography-mass spectroscopy (GC-MS), nuclear magnetic resonance (NMR) spectroscopy, selected ion flow tube-mass spectrometry (SIFT-MS), ion-molecule reaction-mass spectrometry (IMR-MS), secondary electrospray ionization-mass spectrometry (SESI-MS), proton transfer reaction mass spectrometry (PTR-MS) and field asymmetric ion mobility spectroscopy (FAIMS)(Wilson, 2018).

However, all of these conventional methods are quite cumbersome, non-portable, bulky, expensive, low-sensitivity, time-consuming and require highly skilled analysts to perform the qualitative and quantitative analysis of the sample (Krisher et al., 2014) especially with a more complex mixture of VOCs (Machado et al., 2005). To overcome the limitation of the spectroscopic and MS techniques, a handheld and portable device is known as the electronic nose (e-nose) has been developed as a gas compound detector to provide immediate results and identify numerous diseases non-invasively and in real-time (Feng et al., 2019).

2.4 Electronic Nose (Gas Sensor)

E-nose is an artificial sensor inspired by the olfactory system of humans or mammals (sense of smell), designed to detect and differentiate a wide variety of gas compounds. E-nose is composed of a collection of an array of gas sensors with a pattern recognition system (Bermak et al., 2006). The gas sensor is a main component in the electronic nose device. There are different types of sensors used for breath analysis including chemoresistive sensor, quartz crystal microbalance sensor and field-effect transistor sensor which apply different designs and working principles.

2.5 TYPE OF GAS SENSOR

2.5.1 Chemoresistor Based Gas Sensor

Chemoresistor sensor is a sensor with a sensing material deposited on the surface of two or interdigitated electrodes as a bridge gap between the electrode fingers. Chemiresistor sensor is the most used gas sensor and became the focus of recent researches because of its low-cost fabrication process, high sensitivity and probable miniaturization. The response of the chemiresistive sensor is measured by the changes of resistance on the sensor's surface upon gas exposure as its signal output. The detection of the gas compound occurs when the gas analytes interact with the surface of the sensing material that has been deposited as a thin/thick film on the semiconductor sensor by absorption and adsorption process. Oxidizing or reducing gases will change the oxygen amount on the surface and thus generating a change of electrical resistance of the sensor as the signal output (Dragonieri et al., 2017).

Chemiresistive sensors can be divided into two types depending on the density of the majority carrier in the metal oxide material; n-type (electron) and p-type (hole). The resistance from the n-type sensor will increase in the presence of oxidizing gas and will decrease in the presence of reducing gas. Meanwhile, for the p-type sensor, the resistance decreases in the presence of reducing gas and increases in the presence of oxidizing gas instead. Metal oxide chemiresistive sensors can perform the detection of the analytes at room and low working temperatures (Liu et al., 2019).

2.5.2 Quartz Crystal Microbalance Based Gas Sensor

Quartz crystal microbalance (QCM) is a type of sensor with a piezoelectric effect. The sensor is made up of a thin quartz crystal sandwiched between two electrodes. The detection of gas is measured based on the changes of resonance frequencies and mass on the sensor surface. The response of the sensor depends closely on the type of crystal cut and sensitive materials for gas absorption (Hu, Wan, Jian, Ren, Jin, Su, & Bai, 2019).

The changes in resonance frequencies are altered by the increase of mass that occurs due to the absorption of gas onto the surface of the crystal during exposure to vapour. The sensor can work at room temperature and in high selectivity conditions. However, the QCM sensor is commonly used as a supportive element of sensor array because it cannot perform a stand-alone operation, has a complex fabrication process and has poor signal due to noise (Arshak et al., 2004).

2.5.3 Field-Effect Transistors Based Gas Sensor

Field-Effect Transistors (FET) gas sensor consists of source and drains electrodes with a channel material as a bridge between the two electrodes FET works by controlling the flow of the carrier at the conductive channel from source to drain and influenced by the applied voltage at the gate and source terminal. FET sensor has been successfully reported in diagnosed respiratory diseases such as lung and gastric cancer(Hu, Wan, Jian, Ren, Jin, Su, Bai, et al., 2019). The detection occurs when gas molecules interact with the active surface or conductive channel, thus leading to a change in the drain-source current and the gate voltage. FET sensor is a promising sensor in gas detection since it has low operating temperature, high sensitivity, can be monitored as a single sensor as it allowed more freedom for pattern recognition due to its ability to extract multiparameter and can detect target analytes from gas or liquid.

However, the FET sensor is very sensitive to humidity. Y. Deng et al in their research regarding flexible sensors for the detection of volatile biomarkers had used modified Polyaniline (PANI) film as hydrophobic channels to minimize the effect of humidity and mechanical bending of the sensor (Deng et al., 2018).

Different types of sensors are different in their response, sensitivity, selectivity, and detection range. Table 2.2 summarizes the different types of sensors as mentioned previously based on their advantages and disadvantages.