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GAS SENSOR ARRAY BASED ON GRAPHENE-DOPED TiO_2 FOR ACETONE VAPOR WITH DEEP NEURAL NETWORK

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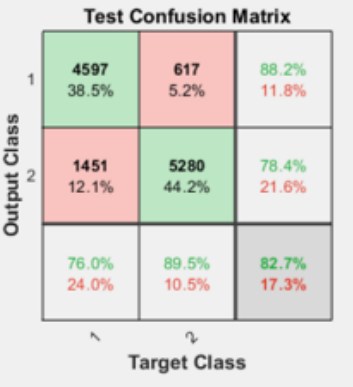
Graphical abstract



Abstract

Acetone is categorized as a volatile organic compound (VOC) gas. Exposure to it for a long period harms human health and the environment. Commonly, a single sensor is used to sense the target gas. However, it always suffered from low selectivity, making it difficult to analyze and differentiate various gases. To overcome this problem, a gas sensor array is proposed in this work to investigate the response value for each array to the acetone vapor. Six sensor arrays based on various ratios of TiO_2 doped with graphene were deposited using the screen-printing technique on an FR3-printed circuit board (PCB). The gas sensor arrays were exposed to two different concentrations of acetone vapor at room temperature. Next, the response values were classified using a deep neural network (DNN). The results revealed that the T95_G5 gas sensor responds more to 25 mL and 35 mL of acetone gas, with 1.0966 and 1.2074, respectively. The overall DNN accuracy for acetone vapor was 82.70%.

Keywords: VOC gases, gas classification, binder, screen printing, artificial intelligence

Abstrak

Aseton dikategorikan sebagai gas sebatian organik meruap (VOC). Pendedahan kepadanya untuk tempoh yang lama membahayakan kesihatan manusia dan alam sekitar. Lazimnya, satu sensor digunakan untuk mengesan gas sasaran. Walau bagaimanapun, ia sentiasa mengalami selektiviti yang rendah, menjadikannya sukar untuk menganalisis dan membezakan pelbagai gas. Untuk mengatasi masalah ini, tatasusunan sensor gas dicadangkan dalam kerja ini untuk menyiasat nilai tindak balas bagi setiap tatasusunan kepada wap aseton. Enam susunan sensor berdasarkan pelbagai nisbah TiO2 yang didopkan dengan graphene telah didepositkan menggunakan teknik percetakan skrin pada papan litar bercetak FR3 (PCB). Susunan sensor gas telah terdedah kepada dua kepekatan wap aseton yang berbeza pada suhu operasi bilik. Seterusnya, nilai tindak balas dikelaskan menggunakan rangkaian saraf dalam (DNN). Keputusan mendedahkan bahawa sensor gas T95_G5 bertindak balas lebih kepada 25 mL dan 35 mL gas aseton, masing-masing dengan 1.0966 dan 1.2074. Ketepatan DNN keseluruhan untuk wap aseton ialah 82.70%.

Kata kunci: Gas VOC, klasifikasi gas, pengikat, percetakan skrin, kecerdasan buatan

1.0 INTRODUCTION

Volatile Organic Compound (VOC) gases are hazardous to the environment and human health. Acetone is a type of VOC gas that harms human health, whether in the long or short term. Acetone is a colorless liquid that is extremely volatile and flammable and is a basic chemical used in producing plastic, textiles, medications, and other products [1]. At exposure levels of 300 to 500 ppm, it irritates the respiratory system, while at 2000 ppm, it can make people feel nauseous [1]. Acetone is an irritant and is present naturally in the environment and the human body at the minute level, and also can induce hepatotoxicity upon inhalation [2]. Acetone gas is regarded as one of the hazardous gases. Besides that, acetone has also been studied as a biomarker for diabetes [3]. Therefore, early identification of acetone fumes should be avoided as they harm the human eyes and nose, which can be accomplished using an acetone sensor, in addition to maintaining human safety.

With increasing technological development, sensors play are incredibly important role in today's world to identify and analyze physical, chemical, and biological parameters such as pressure, gas concentration, or the presence and concentration of a biological analyte. A gas sensor can detect and measure specific gases qualitatively and quantitatively. In addition to that, a gas sensor can give a quick change when exposed to the target gas. Thus, a gas sensor is proposed in this work to sense the acetone vapor. Nowadays, metals and metal oxides are the main sensing materials used in gas sensors. Their gas selectivity is restricted, and their sensitivity is insufficiently high [4]. Recent studies reported that various chemical-based gas sensors such as tin dioxide (SnO_2) [5], [6] zinc oxide (ZnO) [4], [7], [8], titanium dioxide (TiO_2) [9],[10] indium oxide (In_2O_3) [11], ceric oxide (CeO_2) [12], and nickel oxide (NiO) [13], [14], have been used to sense acetone gas at various operating temperatures. Metal-oxide-based gas sensor has the ability to sense various gases and can produce high-sensitivity gas sensors. By applying a metal-oxide semiconductor in chemiresistive gas sensors, a small size and low-cost fabrication of the gas sensor can be obtained. Reported that several metal oxides were working at high operating temperature up to 200°C, such as SnO_2 [6], ZnO [7], CeO_2 [12], and NiO [13] where which will lead to high power consumption. Even though TiO_2 also reported working in high temperature, several literatures discussed about ability of TiO_2 to operate at room operating temperature [15]–[18]. Therefore TiO_2 has been chosen as a sensing material in this study to investigate its capability to sense the acetone vapor at room temperature. With incredible advantages of TiO_2 can detect various types of gases and working at room temperature, it is believed that exploring TiO_2 as array sensors makes it possible to detect acetone gas for obtaining the desired performance.

To achieve a high-sensitivity gas sensor, doping materials are always added to the main sensing material. Various materials, such as silver and multi-walled carbon nanotubes, have been used as doping materials. Catalytic noble metals such as gold, palladium, and platinum loaded atop ordinary metal oxides (SnO_2, ZnO, In_2O_3, and TiO_2) can also increase the sensitivity of the sensor [19]. Graphene is a well-liked two-dimensional (2D) material with exceptional thermal and electrical conductivity, low electrical noise, and an ultrahigh surface-to-volume ratio, and has been thoroughly studied both theoretically and empirically since its discovery in 2004. Graphene has been added as a dopant material in other studies to improve sensor performance [20], [21]. Graphene can detect gas molecules as a sensor material because of its special characteristics [22]. The advantages that graphene-based gas sensors inherit include superior mechanical qualities that make processing a variety of electronic devices easy and a high specific surface area for adsorbing and sensing gas molecules [23]. Currently, graphene-based gas sensors have emerged as the main focus of gas-sensor research because they can mitigate the shortcomings of traditional sensors, such as large power consumption and temperature-dependency [24]. Therefore, graphene has been chosen as a doping material to be added to the TiO_2 gas sensor to enhance the gas sensor performance in this work.

Commonly, a single gas sensor is used to detect the target gas. However, it is difficult for a single gas sensor to precisely detect the target gas when additional interfering gases are present [11]. Thus, an electronic nose device is widely used as an effective technique for detecting gas analytes, which combines pattern recognition technology with gas sensor arrays [11]. An electronic nose with an array of metal-oxide-semiconductor gas sensors can be a promising platform for developing new machine-learning capabilities for odor and smell recognition [11]. To build an e-nose for the discrimination of gases, the multisensory array was generally far more successful [25], [26]. Usually, sensor arrays are utilized for gas discrimination to obtain highly sensitive and selective gas sensors to distinguish gases and their mixtures efficiently [19]. Thus, a gas sensor array is proposed in this study with the use of six gas sensors with different ratios for each sensor to compare their performance to acetone vapor.

Currently, a classification method has been applied widely in gas sensor arrays to differentiate various gases, such as principal component analysis and neural networks. Neural network-based models have gained popularity over the past ten years due to their supervised, interactive learning capability that allows them to correlate input data with the desired output [27]. The most common recognition used for gas classification is convolutional deep neural networks (CNN) [28], artificial neural networks (ANNs) [11], and deep neural networks (DNN) [27]. The DNN method which can learn features directly

from the absorption spectra after training with sufficient samples, achieves the direct identification of gas concentration free of pressure calibration and profile fitting [29]. In various pattern classification applications, the DNN method has demonstrated exceptional classification performance and emerged as a key tool for document analysis, picture identification, object detection, and video understanding [30]. The DNN method has been applied in this work to classify the two different acetone concentrations because of the advantages of the DNN method, which employs numeric data as the input data. The input data was selected based on the current value during acetone exposure.

This paper presents the development of six gas sensor arrays based on six different weight ratios of TiO_2 and graphene onto FR3 substrate. The interdigitated electrode and sensing layer were fabricated using screen-printing technology. Each gas sensor in the sensor array was exposed to two different concentrations of acetone vapors at room operating temperature. The characteristics of the gas sensor array to acetone vapor were analyzed regarding the sensing response value. By using current measurement data, these studies demonstrated the deep neural network as a classification method for two different concentrations of acetone vapor. The results showed that the DNN method can accurately classify the acetone concentrations. This study showed that this method can be applied to classify the gas concentrations for any gases. This contributes to the growing interest in e-nose applications in the gas sensing industry.

2.0 METHODOLOGY

2.1 Preparation of Binder and Graphene-doped TiO_2 Paste

A thick film paste consists of a binder and sensing powder. A binder was prepared using three main materials: ethyl cellulose, linseed oil, and terpineol. Initially, 0.2 g of ethyl cellulose, 0.5 g of linseed oil, and 9.3 g of terpineol were mixed in a vial using a magnetic stirrer at 40°C and 200 rpm until it dissolved and turned into a homogenous and viscous binder. Next, six pastes of graphene-doped TiO_2 were prepared to produce six gas sensors with various ratios of TiO_2 and graphene paste to make a gas sensor array, as listed in Table 1. A mixed powder that consists of graphene powder and TiO_2 powder needs to be prepared before the paste can be prepared. Firstly, 25 ml of acetone was added to 99.50 w.t.% of TiO_2 and 0.5 w.t.% of graphene nanoflakes powder, and then sonicated using a sonication bath for 10 minutes. Then, the mixed solution was dried in the oven at 100°C until it became a solid mixture. Next, the solid mixture was crushed slowly to a mixed powder until there were no more lumps and the color was uniform by using a mortar and pestle. Then, the mixed powder was mixed with the binder according to the ratio of 55 w.t.% for TiO_2 and graphene and 45 w.t.% for the binder to make a thick film paste. To obtain a homogeneous paste and prevent agglomeration, the mixed powder was gradually added to the paste, namely T99.5_G0.5. The paste was stirred using a magnetic stirrer at 40°C with 80 rpm for 24 hours. Similar procedures were applied for the other five pastes (T99_G1, T98_G2, T97_G3, T95_G5, and T92_G8). The preparation of TiO_2 paste for the thick film gas sensors was adapted from the prior study [31].

Table 1 Ratios of graphene-doped TiO_2 for thick film paste

Paste name	TiO_2 (w.t.%)	Graphene (w.t.%)
T99.5_G0.5	99.50	0.50
T99_G1	99.00	1.00
T98_G2	98.00	2.00
T97_G3	97.00	3.00
T95_G5	95.00	5.00
T92_G8	92.00	8.00

2.2 Fabrication of Gas Sensor Array onto FR3 Substrate

Proteus software was used to design the current flow of six arrays to avoid the use of wires for six gas sensors on a board. The interdigitated electrode was deposited on the FR3 PCB for one array using a screen-printing technique and heated at 100°C for 15 minutes. The interdigitated electrode was fabricated one by one to ensure good electrical contact. Then, the pastes (T99.5_G0.5, T99_G1, T98_G2, T97_G3, T95_G5, and T92_G8) were deposited onto the interdigitated electrode for each array using a screen-printing technique, and heated at 200°C for one hour. The sensing layer was also fabricated one by one to ensure a good sensing layer of the gas sensor for the gas sensor array. Two copper wires were attached to the leg of the electrode using the silver conductive paste and heated at 100°C for 15 minutes. The other end of the copper wire and two terminal blocks have been soldered to the circuit's track. Two terminal blocks have been used; the first point is to connect to the ground point, and the second point is to connect with the supply voltage of the sensor array. Figure 1 shows the fabricated six gas sensor arrays based on graphene-doped TiO_2.

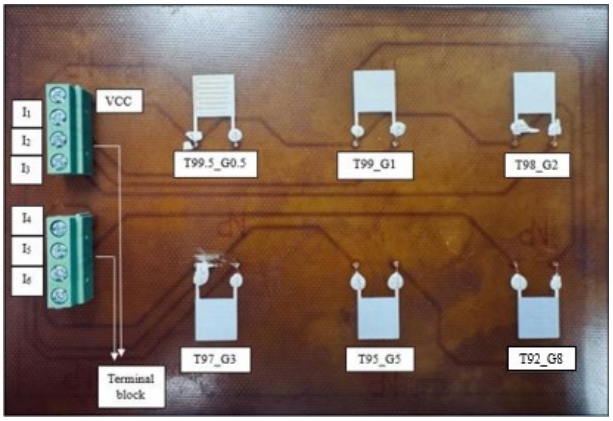


Figure 1 Fabricated gas sensor arrays based on graphene-doped TiO_2

2.3 Experimental Setup of Gas Sensor Array to Acetone Vapor

Figure 2 shows the experimental setup of the gas sensor array measurement to acetone. The gas sensor array was placed in a gas chamber and connected to the source meter. A 10V was supplied to the gas sensor, and current changes were recorded using LabVIEW 2010 software. Acetone solution is filled in glassware, and a silicone hose is used to flow the acetone vapor to the gas chamber. Two different acetone solutions will be used to produce two different concentrations of acetone vapor. The first acetone solution was prepared by mixing 25 mL of acetone with 25 mL of distilled water, while the second acetone solution was prepared by mixing 35 mL of acetone with 15 mL of distilled water. Initially, the acetone solution was stirred with a magnetic stirrer at 100 rpm for 5 minutes to ensure the solution was mixed well. To make an acetone vapor, the acetone solution was heated and stirred to a temperature of 120°C at 100 rpm for 30 minutes. For the current measurement, the gas sensor array will be exposed to the ambient air for 10 minutes for stabilization at normal air pressure, then the acetone vapor will be exposed to the gas sensor array for 30 minutes to monitor the current changes and the response time. After that, the hose connection was disconnected from the gas chamber, and the recovery period was observed for 10 minutes.

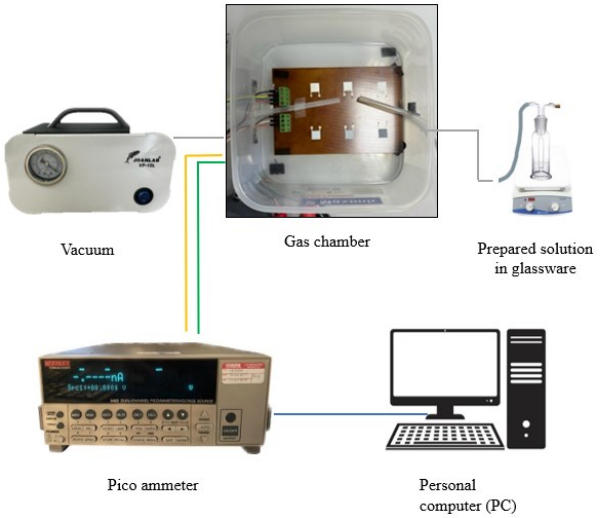


Figure 2 Experimental setup of gas sensor measurement to acetone vapor

3.0 RESULTS AND DISCUSSION

3.1 Current-Voltage (I-V) Characteristics

Current-Voltage (I-V) characteristics of the gas sensor array were measured by supplying a -1V to 1V to check the linearity of the gas sensor. If the gas sensor produced a linear graph, this result suggests that the gas sensor followed Ohm's Law, and the gas sensor can be exposed to the target gas. Figure 3 shows the I-V characteristics of the gas sensor array. The results indicated that all the gas sensors produced linearity; therefore, all gas sensors can be exposed to the acetone vapor. The resistance value for each gas sensor was calculated based on Ohm's law. The resistance values for T99.5_G0.5, T99_G1, T98_G2, T97_G3, T95_G5, and T92_G8 were approximately 7.96 GΩ, 37.28 GΩ, 38.81 GΩ, 25.03 GΩ, 33.76 GΩ, and 9.36 MΩ, respectively. The highest resistance value was produced by the T98_G2 gas sensor, while the lowest resistance value was produced by the T92_G8 gas sensor. These results revealed that the doping process was successful based on the changes in resistance value for all gas sensors. A high amount of graphene in TiO_2 will generate the lowest resistance, as measured in T92_G8, where the resistance value was approximately 9.36 MΩ.

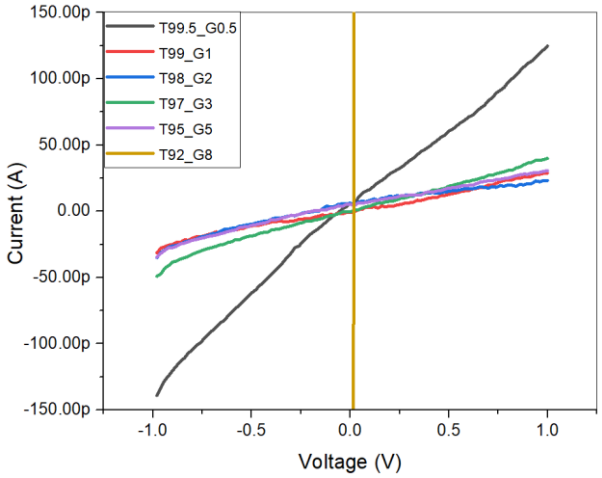


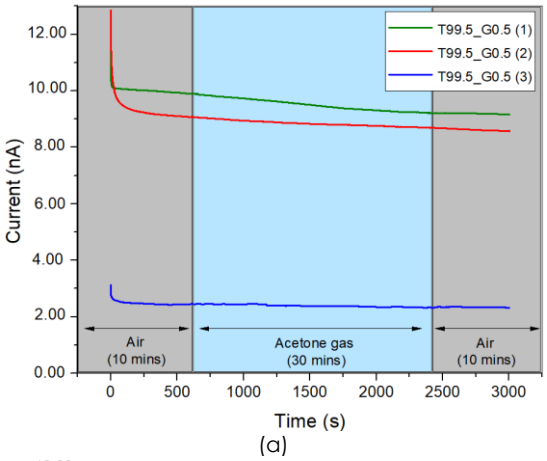
Figure 3 I-V characteristics of gas sensor array

3.2 Current Measurement of Gas Sensor Array to Acetone Vapor

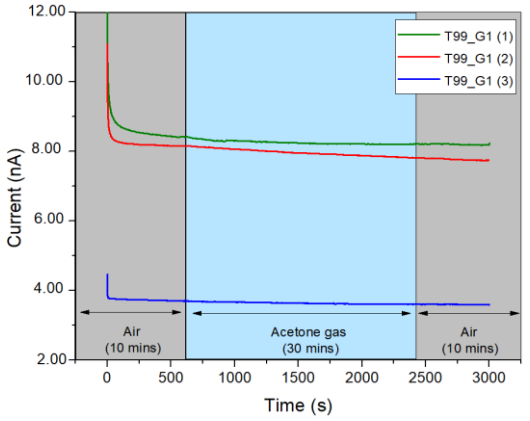
The performance of the gas sensor array to acetone vapor was evaluated in two different concentrations, which are 25 mL of acetone and 35 mL of acetone. These two concentrations need to be carried out to observe the response value of the gas sensor to the two different concentrations and to find the acetone concentration that is more responsive to acetone gas. Each gas sensor will be exposed three times to the acetone vapor to observe the response value and repeatability characteristic of the gas sensor.

Figures 4 and 5 display the dynamic response of the gas sensor array to 25 ml of acetone and 35 ml of acetone, respectively. Figure 4(a), 4(b), 4(c), 4(d), 4(e), and 4(f) present the dynamic response of acetone for T99.5_G0.5, T99_G1, (c) T98_G2, (d) T97_G3, (e) T95_G5, and (f) T92_G8 gas sensor to 25 ml of acetone, respectively. Figure 5(a), 5(b), 5(c), 5(d), 5(e), and 5(f) present the dynamic response of acetone for T99.5_G0.5, T99_G1, (c) T98_G2, (d) T97_G3, (e) T95_G5, and (f) T92_G8 gas sensor to 35 ml of acetone, respectively.

It can be seen that the sensing response showed a downward current value for T99.5_G0.5, T99_G1, T98_G2, T97_G3, and T95_G5, while T92_G8 showed an upward current value when there was the presence of acetone gas. These results suggest that these gas sensors (T99.5_G0.5, T99_G1, T98_G2, T97_G3, T95_G5) are n-type gas sensors, while the T92_G8 gas sensor is a p-type gas sensor. The T92_G8 gas sensor has been changed from n-type to p-type due to the higher graphene concentration in the T92_G8 sensing layer, which is 8 w.t.% compared to the other gas sensors in the gas sensor array. The upward response was similar to prior research reported in Ref. [32], where the sensing layer only consists of graphene, which also supports the sensing material as a p-type gas sensor.



(a)



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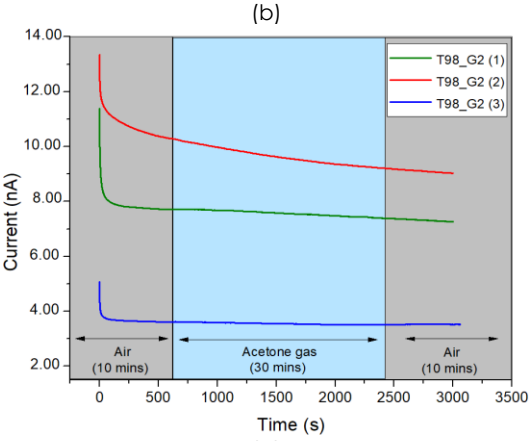

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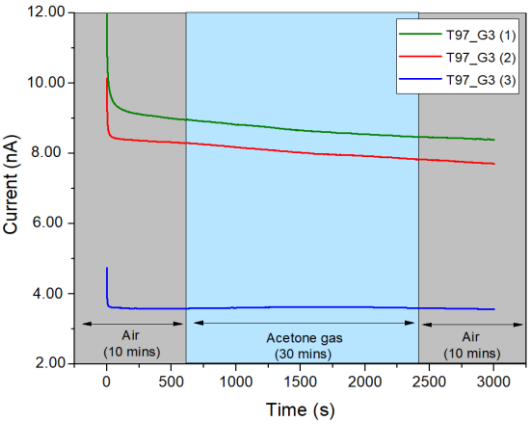

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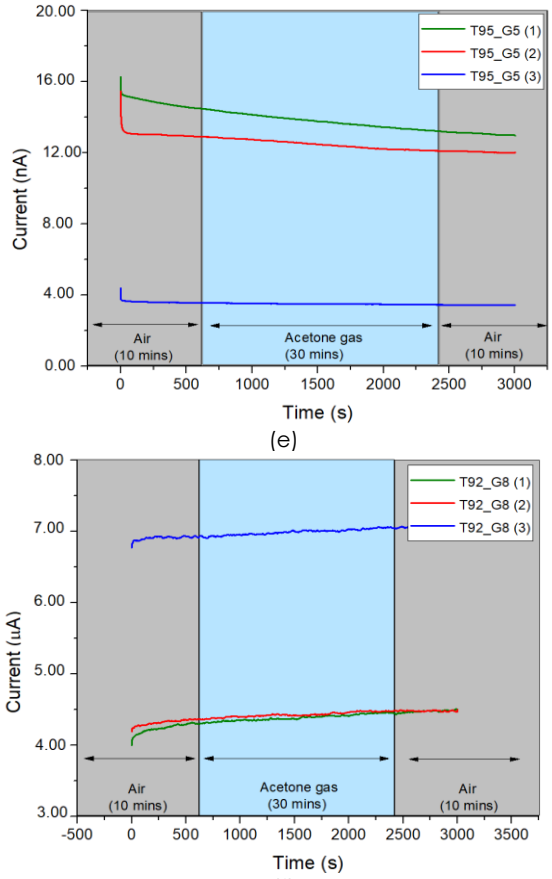
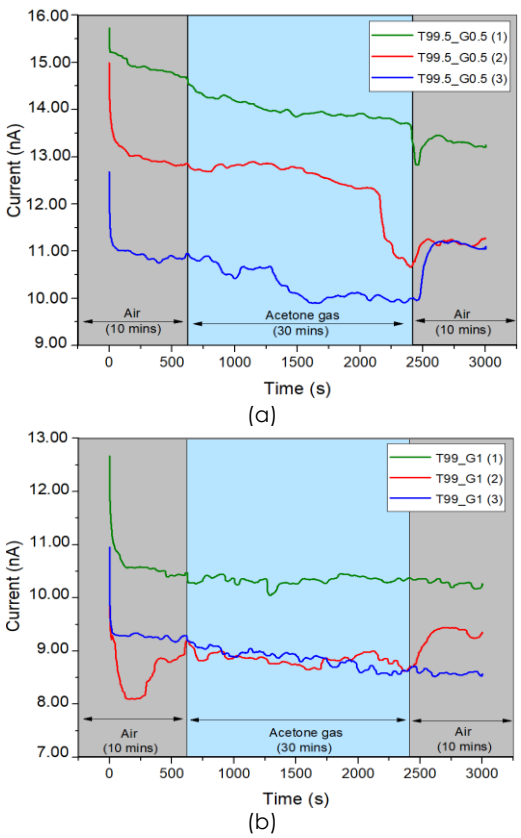


Figure 4 Response of gas sensor array to 25 mL of acetone (a) T99.5_G0.5, (b) T99_G1, (c) T98_G2, (d) T97_G3, (e) T95_G5, and (f) T92_G8



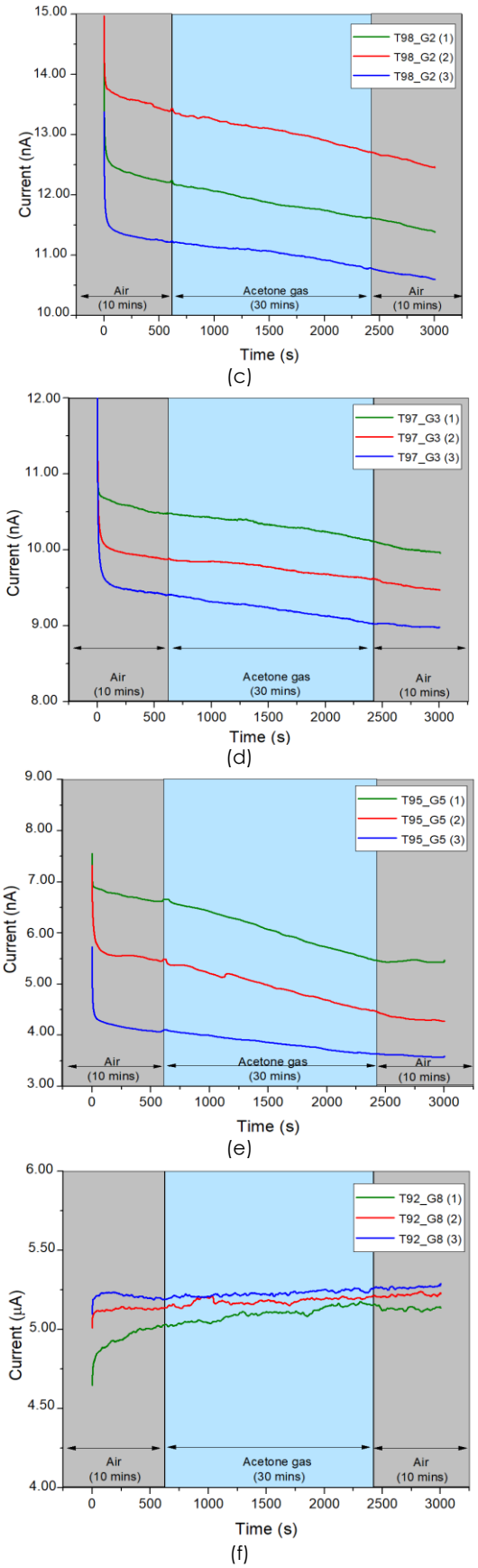


Figure 5 Response of gas sensor array to 35 mL of acetone (a) T99.5_G0.5, (b) T99_G1, (c) T98_G2, (d) T97_G3, (e) T95_G5, and (f) T92_G8

Table 2 shows the sensing response value of each gas sensor array to 25 mL and 35 mL of acetone. The sensing response values for T99.5_G0.5, T99_G1, T98_G2, T97_G3, T95_G5 gas sensors were calculated using equation (1), while for the T92_G8 gas sensor was calculated using equation (2) below:

$$Sensing\ response = \frac{I_{air}}{I_{gas}} \quad (1)$$

$$Sensing\ response = \frac{I_{gas}}{I_{air}} \quad (2)$$

Where I_g is the current during acetone flow and I_{air} is the current during air flow. I_g value was taken at the time of first exposure to acetone gas, which occurred at t = 600 s, and I_{air} value was taken at the end time of air flow during the first cycle, which occurred at t = 2400 s for T99.5_G0.5, T99_G1, T98_G2, T97_G3, T95_G5 gas sensors. Whereas the I_g value was taken at the end time of acetone exposure, which occurred at t = 2400 s, and the I_{air} value was taken at the end time of air exposure in the first cycle, which occurred at t = 600 s for the T92_G8 gas sensor. The taken value of I_g and I_{air} was different because the T92_G8 gas sensor is a p-type gas sensor, while T99.5_G0.5, T99_G1, T98_G2, T97_G3, and T95_G5 gas sensors are n-type gas sensors.

Table 2 Sensing response value for each running time of a gas sensor to acetone

Sample name	Sensing response value					
	25 mL acetone			35 mL acetone		
Running time	1	2	3	1	2	3
T99.5_G0.5	1.0735	1.0433	1.0460	1.0746	1.2044	1.0856
T99_G1	1.0251	1.0433	1.0251	1.0062	1.0331	1.0766
T98_G2	1.0421	1.1163	1.0277	1.0499	1.0519	1.0408
T97_G3	1.0584	1.0589	1.0038	1.0349	1.0278	1.0435
T95_G5	1.0966	1.0712	1.0301	1.2074	1.2032	1.1241
T92_G8	1.0356	1.0262	1.0181	1.0269	1.0137	1.0122

It can be seen that the T95_G5 gas sensor has a consistent and higher response to 25 mL of acetone, with response values of 1.0966, 1.0712, and 1.0301 for all three response readings compared to other sensor arrays. Meanwhile, the T92_G8 gas sensor has a lower response than other gas sensors, with 1.0356, 1.0262, and 1.0181 for all three measurements. Therefore, the T95_G5 gas sensor is suitable for the concentration of 25 ml of acetone gas because it produced the most consistent and highest response compared to other sensor arrays. Meanwhile, for 35 mL of acetone, the T95_G5 gas sensor has a consistent and higher response than other sensor arrays, with response values of 1.2074, 1.2032, and 1.1241 for all three measurements. Whereas the T92_G8 gas sensor has a lower response than other gas sensors, with 1.0269, 1.0137, and 1.0122 for all three measurements. Thus, it can be concluded that the T95_G5 gas sensor is the responsive gas sensor for the concentration of 25 mL and 35 mL of acetone gas because it produces the most consistent and highest response compared to other gas sensors.

3.3 Error Bar for Gas Sensor Arrays

Figure 6 shows the histogram for error bars of 25 mL and 35 mL of acetone gas for gas sensor arrays. It can be observed that the average value of sensing response for the gas sensor array showed almost a similar value, thus the error bar generated a slightly different value for 25 mL of acetone, while the average value of sensing response for the gas sensor array showed different changes for 35 mL of acetone, thus the error bar showed significant changes as compared with all gas sensors. This result found that the deviation for 35 mL of acetone was higher than 25 mL of acetone. This result suggests that the gas sensor array was more responsive to 35 mL of acetone vapor compared to 25 mL of acetone vapor, based on the calculated error bar shown in the histogram.

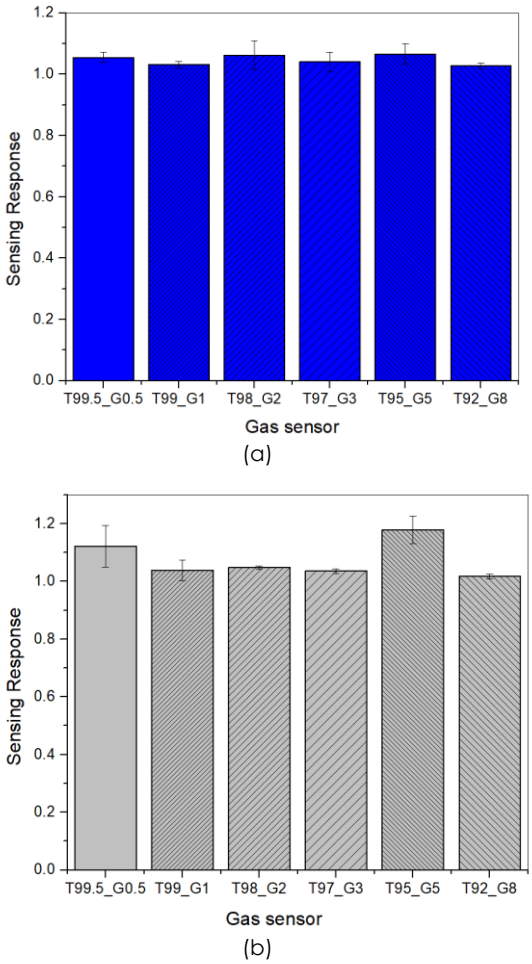


Figure 6 Error bars of 25 mL and 35 mL of acetone vapor for gas sensor arrays

3.3 Classification of Acetone Concentrations using Deep Neural Network (DNN)

Classification of two different acetone concentrations was carried out using a deep neural network. The parameters for DNN are listed in Table 3.

Table 3 Parameters for DNN

Parameter	Quantity
Number of neurons	50
Number of epochs	1000
Number of hidden layers	5

Classification results based on the test confusion matrix are shown in Figure 7. There were 79,632 data points for each concentration to train, validate, and test the process. It can be seen that the class 1 group was classified correctly with 76.0% (specificity) in the test confusion matrix. The class 2 group was classified correctly with 89.5% (sensitivity). It can be observed that 4597 data points were classified correctly in class 1, and 1451 data points were misclassified in class 2. Meanwhile, 5280 data points were classified correctly in class 2, and 617 data points were misclassified in class 1. Therefore, the overall correct classification (accuracy) for the two classes using the deep neural network method was 82.7%.

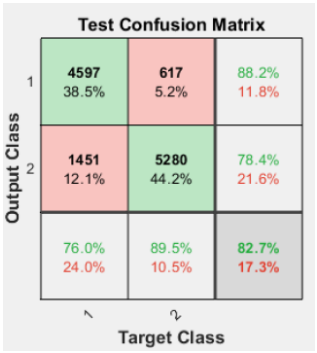


Figure 7 The test confusion matrix of acetone vapor using DNN

3.4 Comparison of Classification Methods for Gas Concentrations

Table 4 lists the comparison of the gas classification method with other studies. Most of the studies showed an accuracy above 90%. As a comparison, this study used a higher number of data points; thus, the produced accuracy for this study is acceptable, which is above 80%. A higher data points will contribute to the accuracy of the model, and the number of misplaced can also be higher, as compared with other classification models. Even though the accuracy is not larger than 90% in this study, this DNN method can be used for gas classification, specifically for numeric data as the input, where current data has been used. Furthermore, the model accuracy is relatively lower (82.7%) in this study compared to other work because the sensing response value is not showed a significant changes for 25 ml acetone and 35 ml acetone. Therefore, it might contributed to the low accuracy. High different of sensing response value for different concentrations will help to enhance the model accuracy in gas classification.

Table 4 Comparison of the gas classification method with other studies

Sensing material	Number of sensor arrays	Type of gases	Method	Data points	Accuracy (%)	Ref.
In_2O_3, SnO_2, and TiO_2	5	carbon dioxide, nitrogen oxide and ammonia	DNN and CNN	350	100 (75 s) 85 (15 s)	[19]
TGS 2600, TGS2602, TGS 2610, TGS 2620	4	nitrogen dioxide and carbon monoxide	CNN	300	100	[33]
CeO_2 and CeO_2-In	4	trimethylamine, acetone, ethanol and isopropanol	random forest	200	97.5	[34]
In_2O_3	-	acetone and ethanol	eXtreme Gradient Boosting (XGB)	1739	99.2	[35]
WS_2	-	nitrogen oxide and ammonia	selective denoising autoencoder	180	98.75	[36]
TGS2611, MG-812, 4NO2-20, 4NH3-100	4	Ammonia, methane, carbon dioxide and nitrogen oxide	bidirectional recurrent neural network	521	98.93	[37]
TiO_2 and TiO_2-graphene	6	acetone	DNN	79,632	82.7	This study

4.0 CONCLUSION

The gas sensor array based on six different ratios of TiO2 and graphene has been successfully designed and fabricated onto the FR3 PCB substrate using screen-printing technology. All the gas sensors were able to respond to acetone vapor. It can be seen that the T95_G5 gas sensor shows a consistent and higher response to the concentration of 25 ml and 35 ml acetone gas compared to other arrays, with response values were approximately 1.0966 and 1.2074, respectively. The DNN models, which are the training models, have been applied for both acetone concentrations to attain the capability of selective identification for specific gases, with the overall accuracy for the classification of acetone vapor being 82.7%. With six types of sensing materials

in a gas sensor, this gas sensor array offers an efficient solution without the need for various gas sensors to identify the concentration of acetone vapors. These results also demonstrated that this method could be potentially utilized for e-nose commercialization in the electronics industry. For future works, the array sensor will be fabricated on Kapton film to enhance the sensing response at room temperature, and then a classification model, such as CNN, will be implemented to obtain a high-accuracy model.

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Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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