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Investigation on TiO_2/graphene as resistance-based gas sensor for volatile organic compound gases detection

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ABSTRACT

Volatile organic compound (VOC) gases are usually produced from industrial activities. Short-term exposure to VOC gases can cause dizziness, headaches, nausea, and throat irritation. Years to a long time exposure to VOC gases can cause cancer and system damage in the human body. With the growth of gas sensor technology, a resistance-based gas sensor based on various structures of resistance-based gas sensors using Titanium dioxide/graphene (TiO_2/graphene) were investigated as a sensing material for detecting volatile organic compound gases, which are acetone and ethanol. The TiO_2/graphene gas sensor was deposited on a Kapton film using a screen printing technique. All TiO_2/graphene gas sensors were exposed to acetone and ethanol at room operating temperature. The results revealed that the highest response values to acetone and ethanol were produced by T99_G1_2 and T98_G2_1, respectively. It can be concluded that design 1 generated the most consistent response to acetone, while design 2 generated the most consistent response to ethanol.

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1. INTRODUCTION

Volatile organic compound (VOC) gases can easily be found in various products, such as building materials, home and personal care products, and common human activities (eg: smoking, burning, and cooking). Short-term exposure to VOC gases can cause dizziness, headaches, nausea, and throat irritation. Whereas years to a long time exposure can cause cancer and system damage in the human body. Acetone and ethanol are categorized under VOC gases. Acetone is widely used as an essential solvent and raw material in various industrial fields, such as explosives, plastics, rubber, fiber, leather, and spray paint [1]. For acetone, it is a common colorless and transparent volatile organic compound and if human exposure is more than 450 mg/m^3 (173 ppm), it can cause fatigue, headaches, and nervous system damage [2]. Whereas ethanol is an organic compound in various fields, such as biomedical, food, pharmaceutical, hygienic, cosmetic, and chemical industries [3]. However, prolonged exposure to high ethanol concentrations can easily result in uncomfortable symptoms such as skin irritation and reduce respiratory and neurological effects even with a concentration as low as 25 ppm [4]. In closed areas, VOC gases can threaten the human health [5]. In recent years, a resistance-based gas sensor has been widely studied by researchers to sense VOC gases due to its fast response, simple fabrication process, and also affordable price. Therefore, the creation of an acetone and ethanol detection device that is highly sensitive and can be used to detect ethanol or acetone leakage is crucial for both industrial safety and human health.

Recent literature has reported that various resistance-based gas sensors have been studied to sense VOC gases such as graphene [6], bismuth ferrite ($BiFeO_3$) [7], zinc oxide (ZnO) [8], indium sulfide (In_2S_3) [9], samarium oxide [10], perovskites [11], titanium dioxide (TiO_2) [12], [13], and tin dioxide [14]. In past research, TiO_2 has been reported as a sensing material for acetone detection [15] and ethanol detection [16], [17]. Furthermore, TiO_2 also has been seen as a promising material in gas sensing due to its ability to sense various types of gases such as hydrogen [18], [19], methane [20], and carbon monoxide [21]. Thus, TiO_2 has been chosen as the main sensing material to sense the VOC gases in this study since this material has been proven able to sense VOC gases. In addition to that, TiO_2 has a wide bandgap semiconductor and excellent material to sense various gases such as hydrogen, hydrogen sulfide, ammonia, and methane. Thus, this study wants to investigate the TiO_2 capability to sense VOC gases at room operating temperature. Besides, TiO_2 has the ability to sense ethanol [22]. It has been studied as a promising gas sensing material owing to its excellent electron mobility and chemical stability [23]. Furthermore, TiO_2 is very promising due to its high specific surface, low cost, and robustness in chemical/corrosive atmospheres [24].

To enhance the sensitivity, the most common method used doping material to enhance the main sensing material of the gas sensor. Therefore, this method also will be applied in this study since this method is also known as a promising technique in gas sensing applications. Thus, graphene nanoflakes has been chosen as doping material because this material can sense VOC gases which are acetone and ethanol at room operating temperature based on prior research in [6]. Graphene, on the other hand, is an atomically thin two-dimensional (2D) sheet of carbon atoms in a hexagonal lattice, has inspired a new era of research due to its exceptional properties such as extremely high mobility, very high thermal conductivity, and surface sensitivity to various molecules [25]. With this combination, TiO_2 and graphene are expected to produce a nanocomposite material with enhanced gas sensing capabilities.

This paper presents the investigation of TiO_2 nanoparticles doped with graphene nanoflakes with various weight concentrations as a sensing material for resistance-based gas sensors. The gas sensor was fabricated using screen-printing technology on Kapton film. All gas sensors were exposed to acetone and ethanol at room operating temperature. The gas sensor characteristic was evaluated in terms of response value. The present finding is significant for the researcher in choosing a suitable design for a resistance-based gas sensor, specifically for acetone and ethanol detection. This study also can be considered as an update of our prior work on the enhancement of acetone and ethanol detection at room operating temperature.

2. METHOD

2.1. Preparation of binder and TiO_2/graphene paste

A binder is made by mixing three materials in a specific ratio: 2 ω.t.% of ethyl cellulose, 5 ω.t.% of linseed oil, and 93 ω.t.% of terpineol. These materials were mixed in a vial with a magnetic bar and stirred on a magnetic stirrer at 40°C and 200 rpm for at least 24 hours to ensure a homogeneous binder. The TiO_2/graphene paste is prepared using three different ratios of TiO_2 and graphene as listed in Table 1. To prepare the TiO_2/Graphene mixed powder, 25 ml of acetone was added to the TiO_2 and graphene powder. The solution undergoes 10 minutes of ultrasonic cleaning at 40°C, followed by drying in an oven at 100°C until it becomes a solid powder. The resulting powder is gently ground in a mortar with a pestle. The TiO_2/graphene was prepared by mixing 55 ω.t.% of TiO_2/graphene powder with 45 ω.t.% binder. The mixed sensing material powder was gradually added to the binder while stirring on a magnetic stirrer. The paste mixing was carried out at 40°C and 80 rpm. The stirring process continues for at least 24 hours to achieve a homogeneous mixture. A similar procedure was applied for all TiO_2/graphene pastes.

2.2. Design structure and fabrication of TiO_2/graphene resistance-based gas sensor using screen-printing technique

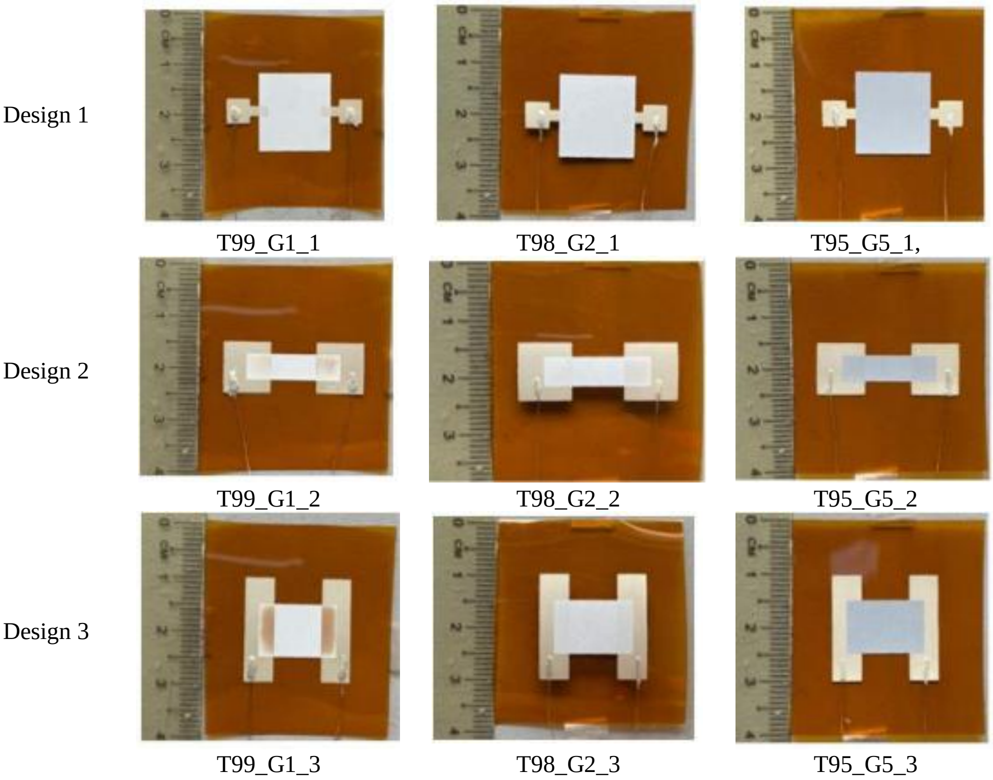
Three design structures of the resistance-based gas sensor were proposed in this study to investigate its capability to sense the VOC gases with different ratios of TiO_2/graphene. The design structures of resistance-based gas sensors were created using AutoCAD software. Table 2 outlines the area of the electrode and sensing material. Design 3 has the largest electrode area (2.4 cm^2), and Design 1 has the largest sensing layer (2.56 cm^2). Kapton film was used as a substrate with a size of 4.0 cm×4.0 cm. Firstly, the electrode based on silver paste was deposited on the Kapton film using the screen-printing technique and then dried in an oven at 150°C for 15 minutes. Next, the sensing layer (TiO_2/graphene paste) was deposited using the screen-printing technique and then annealed at 200°C for one hour. The fabricated gas sensor based on three design structures with their sample names is shown in Figure 1. All designs were adapted from other researchers, which are design 1 from [26], design 2 from [25], and design 3 from [27]. It can be seen that the color of the sensing layer becomes more greyish when the amount of graphene is higher in the paste.

Table 1. Ratio for TiO_2 and graphene paste

Paste name	TiO_2 (ω.t.%)	Graphene (ω.t.%)
TiO_2_G1	99	1
TiO_2_G3	98	2
TiO_2_G5	95	5

Table 2. Area (cm^2) of design

Design	Area (cm^2)	
	Electrode	Sensing material
1	0.58	2.56
2	2.00	1.00
3	2.40	1.60



T99_G1_1 T98_G2_1 T95_G5_1,

T99_G1_2 T98_G2_2 T95_G5_2

T99_G1_3 T98_G2_3 T95_G5_3

Figure 1. Fabricated TiO_2/graphene gas sensors

2.3. Experimental setup of a gas sensor for ethanol and acetone vapor detection

Figure 2 shows the experimental setup of gas sensor measurement for VOC gases. Acetone and ethanol were evaporated in glassware, and a silicone hose was used to flow the vapor into the gas chamber. To make an acetone solution, 25 ml of acetone was mixed with 25 ml of distilled water. The acetone solution is then heated to a temperature of 120°C for 30 minutes to produce acetone vapor. At first, it took 10 minutes for the gas sensor's current to stabilize at standard atmospheric pressure. The response value was then monitored within 30 minutes of acetone vapor being connected to the gas chamber's inlet. The glassware's connection to the gas chamber was severed after 30 minutes, leaving another 10 minutes for the TiO_2/graphene gas sensor to stabilize. A similar procedure was applied for ethanol vapor exposure by using a similar setup.

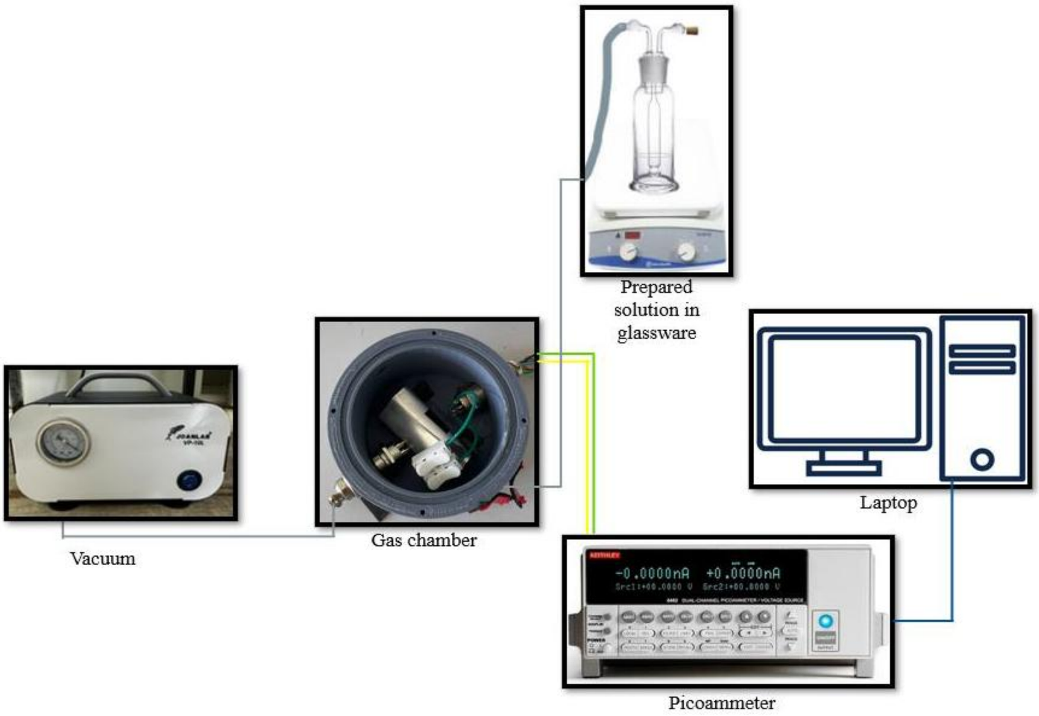


Figure 2. Experimental setup of gas sensor measurement

3. RESULTS AND DISCUSSION

3.1. Current-voltage (I-V) characteristic of TiO_2/graphene gas sensor

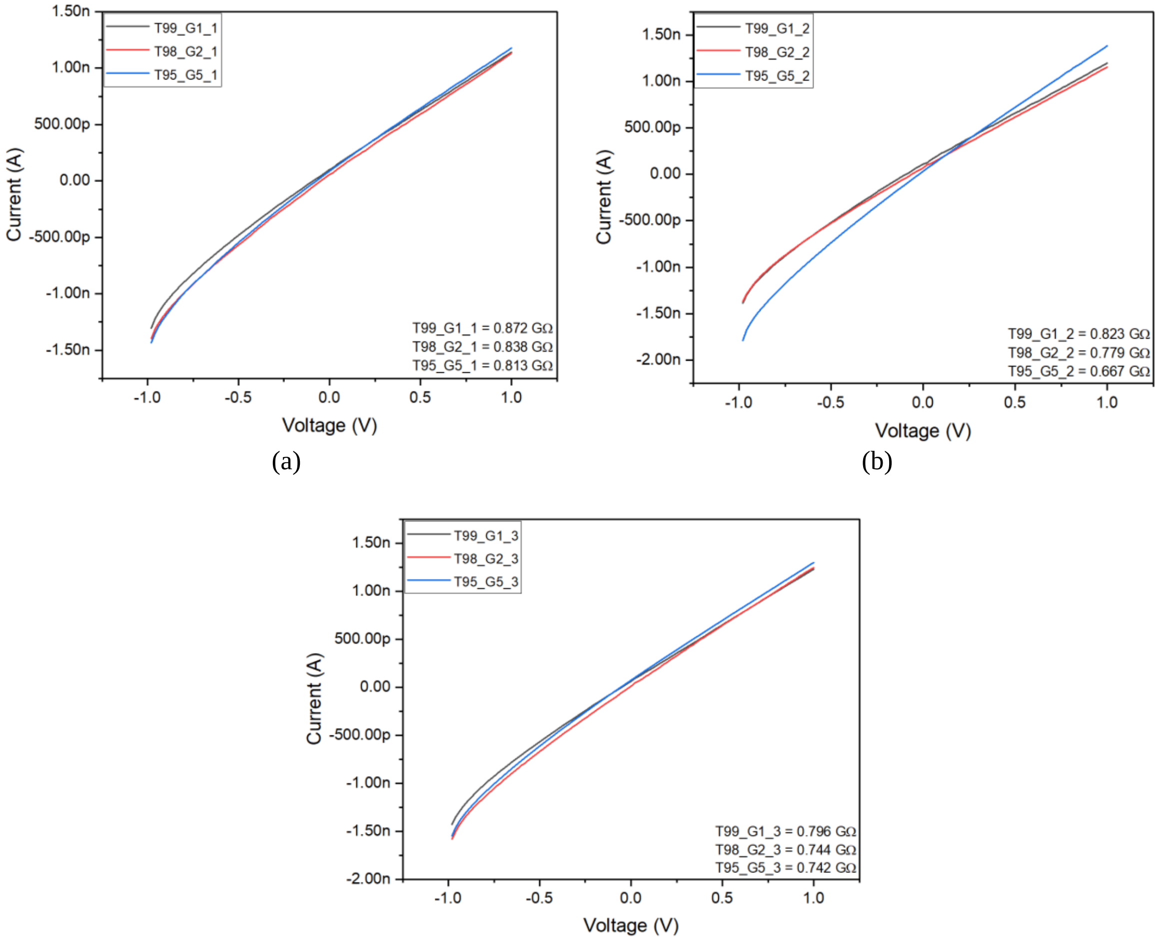
Figure 3 shows the I-V characteristics at -1V to 1V with the resistance values for all the fabricated gas sensors. Figures 3(a) to 3(c) display the I-V characteristics of T99_G1, T98_G2, and T95_G5 for design 1, design 2, and design 3, respectively. It can observed that the linear graph was produced for all gas sensors, which followed Ohm's law. Design 1 generated slightly higher resistance than designs 2 and 3, where this behavior was attributed to its larger sensing material area. The greater surface area provides more interaction points for gas molecules, resulting in noticeable alterations in electrical characteristics upon exposure. This enhanced sensitivity enables the sensor to detect lower gas concentrations, highlighting the significance of surface area in gas sensor design.

3.2. Characteristics of TiO_2/graphene gas sensor to acetone and ethanol

3.2.1. Sensing response to acetone and ethanol

The sensing response for three different designs (design 1, 2, and 3) of TiO_2/graphene gas sensors (T99_G1, T98_G2, and T95_G5) to acetone vapor and ethanol vapor is presented in Figure 4 and Figure 5, respectively. Figures 4(a) to 4(c) illustrate the I-V sensing response of T99_G1, T98_G2, and T95_G5 to acetone vapor for design 1, design 2, and design 3, respectively. Whereas the Figures 5(a) to 5(c) illustrate the I-V sensing response of T99_G1, T98_G2, and T95_G5 to ethanol vapor for design 1, design 2, and design 3, respectively. The initial 10-minute period involves exposing the sensors to atmospheric pressure to achieve stability. Subsequently, acetone vapor is introduced into the gas chamber for 30 minutes, where the gas sensors exhibited a noticeable increase in current, indicative of a response to acetone. A similar pattern was observed in [28]. After exposure, the acetone source was disconnected, and the current was monitored again at atmospheric pressure for stabilization. The consistent trend observed aligns with prior research using TiO_2 as a sensing material for acetone detection, where an increase in current during acetone exposure is attributed to the adsorption and desorption of acetone molecules on the TiO_2 surface, impacting the material's conductivity and other properties.

Meanwhile, three different TiO_2/graphene gas sensor designs (1, 2, and 3) responded to ethanol vapor using pastes T99_G1, T98_G2, and T95_G5. To ensure stability, the sensors undergo a 10-minute exposure to atmospheric pressure without ethanol gas, followed by a 30-minute connection to ethanol gas and then under ambient air. The graph indicates that the sensors respond to ethanol gas with increased current, which is also a similar pattern to acetone vapor. A similar pattern was observed in [29]. All samples exposed to acetone gas follow a consistent trend observed in previous research using TiO_2 for ethanol detection. The rise in current during acetone exposure suggests interactions between acetone molecules and the TiO_2 surface, potentially affecting conductivity and other properties.



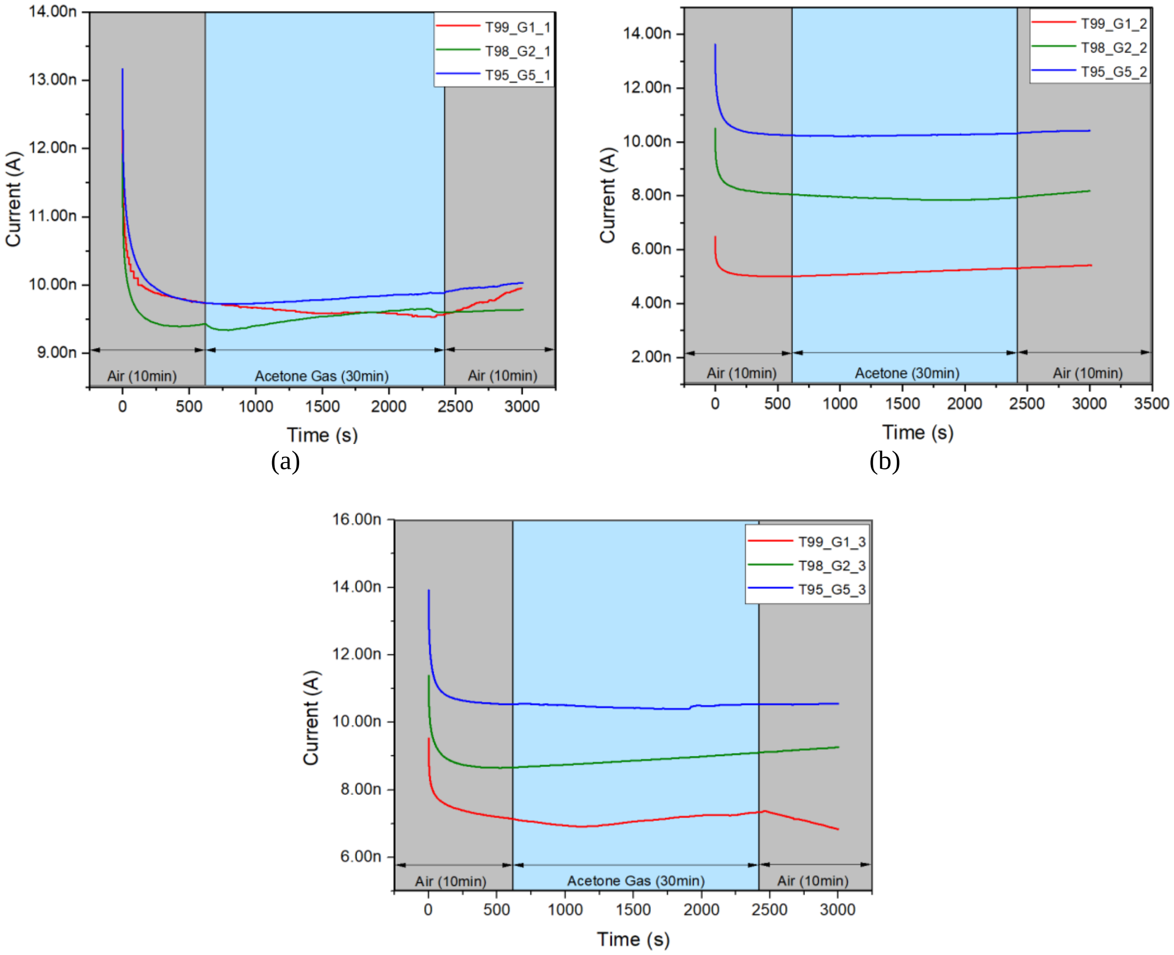
(a) (b)

(c)

Figure 3. I-V characteristics (a) design 1, (b) design 2, and (c) design 3

3.2.2. Response value to acetone and ethanol

Table 3 presents the response values for acetone and ethanol vapor for three different designs of TiO_2/graphene gas sensors. In design 1, 2 ω.t.% of graphene, specifically T98_G2_1, exhibited the highest response for acetone at 1.0182, surpassing T95_G5_1 and T99_G1_1 with values of 1.0147 and 1.0021, respectively. Design 2 showcased that T99_G1_2 to acetone, with 1.0605, had the superior response, making 1 ω.t.% graphene preferable other than gas sensors. In design 3, T98_G2_3 stood out to acetone with a response value of 1.0509, outperforming T99_G1_3 and T95_G5_3 at 1.0255 and 1.0001, respectively. Whereas the response values to ethanol vapor indicate distinct reactions among the designs. Design 1 stands out with T98_G2_1 exhibiting the highest response (1.0906) to ethanol, surpassing T99_G1_1 (1.0698) and T95_G5_1 (1.0126). In design 2, T95_G5_2 displays the strongest response (1.0777) to ethanol, outperforming T99_G1_2 and T98_G2_2 at 1.0616 and 1.0366, respectively, suggesting 5 ω.t.% graphene enhances ethanol vapor detection. Design 3 showed the T98_G2_3 leading with a response of 1.0669, surpassing T99_G1_3 and T95_G5_3 at 1.0073 and 1.0276. Overall, the highest response values to acetone and ethanol were produced by T99_G1_2 and T98_G2_1, respectively. It can be concluded that all three designs of resistance-based gas sensors based on TiO_2/graphene have the capability to sense acetone and ethanol vapor at room operating temperature. In addition, the graphene amount doped into TiO_2 also did not have much impact on the design of the resistance-based gas sensor proposed in this study, since the percentage of differences between 1 ω.t.% to 5 ω.t.% of graphene were approximately 6.04% and 8.27% to the acetone and ethanol vapor, respectively.



(a) (b)

(c)

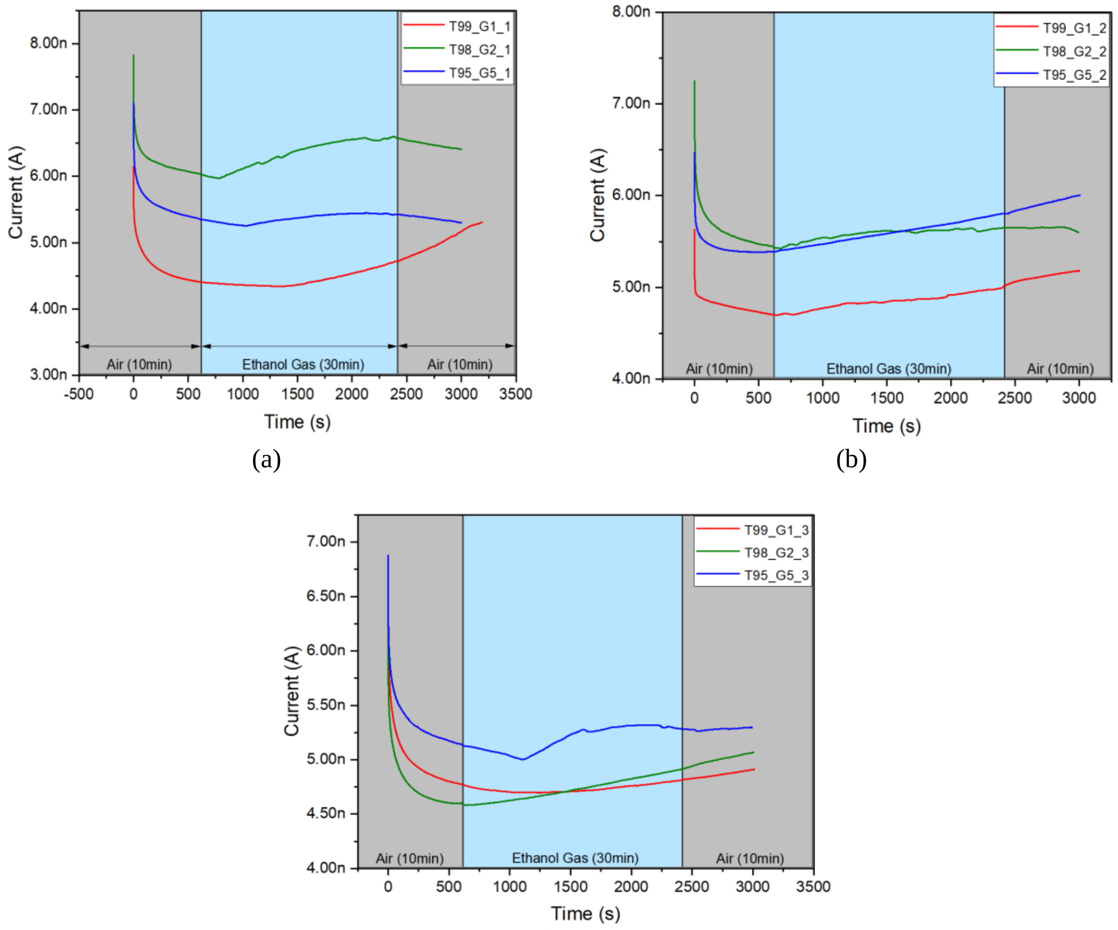
Figure 4. Sensing response of TiO_2/graphene gas sensor to acetone vapor (a) design 1, (b) design 2, and (c) design 3

3.2.3. Comparison of response value to acetone and ethanol with previous studies

Table 4 lists the comparison of the response value of the TiO_2/graphene gas sensor to acetone and ethanol in this study with our prior works. The result revealed that the use of metal oxide (TiO_2) in the gas sensor increased the sensing response to the ethanol and acetone at room operating temperature. Used of screen-printing technology as a deposition technique for the resistance-based gas sensor also showed better performance of the gas sensor to the target gas. It also can be observed that the area of the sensing material is not affected by the response value of the gas sensor at room temperature. Therefore, this finding implies that the selection of sensing material is essential to enhance the sensitivity of a gas sensor to the target gas.

Table 3. Response value to acetone and ethanol vapor

Design	Sample name	Response value	
		Acetone	Ethanol
	T99_G1_1	1.0021	1.0698
1	T98_G2_1	1.0182	1.0906
	T95_G5_1	1.0147	1.0126
	T99_G1_2	1.0605	1.0616
2	T98_G2_2	1.0001	1.0366
	T95_G5_2	1.0059	1.0777
	T99_G1_3	1.0255	1.0073
3	T98_G2_3	1.0509	1.0669
	T95_G5_3	1.0001	1.0276



(a) (b)

(c)

Figure 5. Sensing response of TiO_2/graphene gas sensor to ethanol vapor (a) design 1, (b) design 2, and (c) design 3

Table 4. Comparison of response value to acetone and ethanol with previous studies

Sensing material	Area of sensing layer (cm^2)	Deposition technique	Response value		Ref.
			Acetone	Ethanol	
Graphene SG(G)	1.00	Screen-printing	1.024	1.004	[6]
Graphene (DG(G)-1)	1.00	Doctor Blade	-	1.20	[30]
Graphene (DK(G)-1)	1.00	Doctor Blade	-	1.00	[30]
TiO_2/graphene (T98_G2_1)	2.56	Screen-printing	1.0182	1.0906	This study
TiO_2/grapheme (T99_G1_2)	1.00	Screen-printing	1.0605	1.0616	This study

4. CONCLUSION

In conclusion, resistance-based gas sensors have been successfully fabricated using screen-printing technology with three design structures using TiO_2 doped with various weight ratios of graphene. All the gas sensors responded well to acetone and ethanol vapor at room operating temperature. The results revealed that the T99_G1_2 gas sensor produced the highest response value of 1.0605 to the acetone vapor and the T98_G2_1 gas sensor produced the highest response value of 1.0906 to ethanol vapor. The obtained results gave a path for the development of a resistance-based gas sensor for ethanol and acetone detection operable at room temperature. This finding also can be reproduced with the use of other metal-oxide material as sensing material applied for resistance-based gas sensors for sensitivity enhancement to acetone and ethanol vapor in room temperature sensing.

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