

Enhancement of methane detection using graphene-doped zinc oxide with Blynk internet of things for real-time monitoring

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ABSTRACT

Methane is a potent greenhouse gas, significantly contributing to global warming gases when released into the atmosphere. Methane, a naturally occurring gas, has no harmful impacts on human life at low concentrations. As concentrations rise, symptoms like fatigue, headaches, nausea, irritability, and speech difficulties increase due to asphyxiation. Zinc oxide (ZnO) and graphene-doped ZnO gas sensors were fabricated using a screen-printing technique onto a Kapton film to compare their performance to methane gas at room temperature. A silver paste was used as the interdigitated electrode, deposited on the Kapton film using a screen-printing technique, and fired at 150 °C for 15 minutes. Next, ZnO and graphene-doped ZnO pastes were deposited onto the interdigitated electrode using a screen-printing technique and became the second layer of the gas sensor. The sensing layer was annealed at 200 °C for 60 minutes. All gas sensors responded well to methane gas at room temperature. As a comparison, the graphene-doped ZnO gas sensor responded better to 6,700 ppm of methane gas than the ZnO gas sensor at room temperature. The highest response of graphene-doped ZnO to methane gas was produced by 5 wt. % of graphene doped into ZnO with a response value of 11.5.

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1. INTRODUCTION

Methane, a naturally occurring gas in the atmosphere, has no harmful effects on human life at low concentrations. As concentrations rise, it causes symptoms like fatigue, headaches, nausea, and difficulty speaking. The National Institute for Occupational Safety and Health advises against exceeding 1,000 parts per million for eight hours of labor, while 500,000 ppm can cause asphyxiation. In addition to that, methane contributes 18% of total expected global warming, with a higher global warming potential than carbon dioxide [1], [2]. Reported that in [3], hydropower dams in Peninsular Malaysia and East Malaysia produce 235.7 Gg of methane per year. Malaysia might face the risk of severe flooding and sea level rise as a result of climate change [4], [5]. Therefore, there is a need for a device to detect the methane gas that is able to monitor the methane amounts to avoid harmful effects on humans or the environment. With rapid technologies of sensor

technology with the internet of things (IoT), a gas sensor is seen as a promising device with an affordable price that can help in giving a quick sensing response and monitoring methane in real-time application.

Recent studies showed that most of the sensing material used to detect methane gas is zinc oxide (ZnO) [6]-[8], indium oxide (In_2O_3) [9], tin oxide (SnO_2) [10], [11], and vanadium oxide [12]. It can be seen that ZnO is the most common material used for methane detection, and it is also seen as a promising material for methane detection. Reported that In_2O_3 has suffered from poor selectivity and cross-selectivity, high power consumption, baseline resistance drift, and high operating temperature [13]. Meanwhile, SnO_2 had a high work temperature and low gas response [14]. Therefore, ZnO has been chosen as the main sensing material to detect methane in this study. Furthermore, ZnO has been widely used in many applications such as gas sensors [6]-[8], solar cells [15], [16], antibacterial activities [17], [18], and photodetectors [19]. ZnO has a high sensitivity, which is essential for accurate and consistent detection of methane gas, and its chemical stability supports the lifetime and durability of the sensors, making ZnO a promising candidate for detecting CH_4 [6]. Besides that ZnO, a fascinating semiconductor material with a large band gap and high extinction binding energy, is a promising gas-sensing material due to its increased sensitivity, selectivity, and thermal stability [7], [8]. In addition to that ZnO is also a surface-sensitive material, allowing for the adsorption-oxidation-desorption mechanism of gas sensing in sensing elements [20].

High sensitivity is always demanded for a gas sensor. The most effective method used was the doping method to enhance the gas sensitivity. Owing to the literature, most researchers used doping material to enhance sensitivity to methane such as palladium [6], [21], ferrum [22], and chromium [23]. Thus, this study will also apply the doping method to the sensing material to improve the gas sensor performance for methane. Graphene nanoflakes have been chosen as doping material added into ZnO with various weight concentrations to investigate their performance to the methane. Graphene provides incredible interest in gas sensing applications due to its large specific surface area and high electrical conductivity [24]. For graphene, the electrical properties have been reported to be strongly affected by the adsorption of external molecules onto its surface [24]. The electrical conductivity or resistance of graphene varies as the surface absorbs foreign molecules because of the adsorbate induction of a change in carrier concentration, which can be either an acceptor or a donor of electrons [15]. Therefore, it is expected that the addition of graphene into ZnO can reduce the resistivity of the ZnO gas sensor and enhance the sensing response to methane.

This paper presents the comparison of ZnO and graphene-doped ZnO gas sensors to methane at room operating temperature. The gas sensors were fabricated using a screen-printing technique on a Kapton film. The performance of gas sensors was evaluated in terms of response value, response time, and recovery time. The results revealed that the graphene-doped ZnO gas sensor showed better performance than the ZnO gas sensor to methane at room operating temperature. With the Blynk application, the sensing response of the gas sensor to the methane can be monitored in real-time monitoring and can be applied in various applications.

2. METHOD

2.1. Preparation of binder, ZnO paste, and graphene-doped ZnO paste

ZnO, ethyl cellulose, and α -terpineol were purchased from Chemiz Malaysia, Sigma-Aldrich, and, Acros Organic, respectively. All materials have been used without any purification. To make a thick film paste, two components were needed, which are a binder and a sensing powder. In this study, the binder was prepared using ethyl cellulose and α -terpineol, while the sensing powder was ZnO. The binder is made from a mixture of 5 wt. % ethyl cellulose and 95 wt. % α -terpineol. Both materials were stirred for 24 hours using a magnetic stirrer to achieve homogeneity. Next, the ZnO paste was prepared by mixing 50 wt. % of ZnO powder with 50 wt. % of binder. The ZnO paste was stirred using a magnetic stir for about 24 hours to produce a homogeneous and viscous paste.

To prepare a graphene-doped ZnO paste, a mixed powder of graphene and ZnO should be prepared. Three various weight concentrations of graphene were used: 1 wt. %, 3 wt. %, and 5 wt. %. Firstly, 1 wt. % of graphene and 99 wt. % of ZnO powder were mixed with 20 ml of acetone in a sonication bath for 30 minutes. Then, the solution was dried in an oven until it became a solid mixture powder. After drying, the mixture powder was ground into a smooth powder using a mortar and pestle. After that, the graphene-doped ZnO paste was prepared by mixing 50 wt. % graphene-doped ZnO powder with 50 wt. % binder for 24 hours. Similar methods were applied for 3 wt. % and 5 wt. % of graphene for making graphene-doped ZnO paste.

2.2. Fabrication of the ZnO and graphene-doped ZnO gas sensor

The gas sensor consists of two layers: an interdigitated electrode and a sensing layer. In this study, the interdigitated electrode will be the first layer of the gas sensor, while the sensing layer will be the second layer of the gas sensor. The design of the gas sensor was adapted from prior work [25]. The interdigitated electrode (silver paste) was deposited on the Kapton film via screen-printing using a stencil mesh thickness of 10 μm

and dried in an oven at 150 °C for 30 minutes. Next, the ZnO paste was deposited onto the interdigitated electrode using screen-printing and annealed at 200 °C for 30 minutes in an oven. The sensing layer used was 1 cm × 1 cm with a stencil mesh thickness of 10 µm. Lastly, a fine copper wire was used to make an electrical connection by attaching the wire to the leg of the IDE using a small amount of silver paste. This whole process was repeated for the graphene-doped ZnO gas sensor. ZnO gas sensor was labeled with ZnO, while graphene-doped ZnO gas sensors with 1 wt. % of graphene, 3 wt. % of graphene, and 5 wt. % of graphene were labeled as ZnOG1, ZnOG3, and ZnOG5, respectively. Figure 1 shows the fabricated ZnO and graphene-doped ZnO gas sensors after annealing treatment. Figure 1(a) displays the fabricated ZnO gas sensor and labelled as ZnO. Figure 1(b) depicts fabricated ZnO with 1 wt. % of graphene amounts and labelled as ZnOG1. Figure 1(c) illustrates ZnO with 3 wt. % of graphene amounts and labelled as ZnOG3. Figure 1(d) shows ZnO with 5 wt. % of graphene amounts and labelled as ZnOG5. It can be observed that the color of the sensing layer was darker as the concentration of graphene was increased in the graphene-doped ZnO paste.

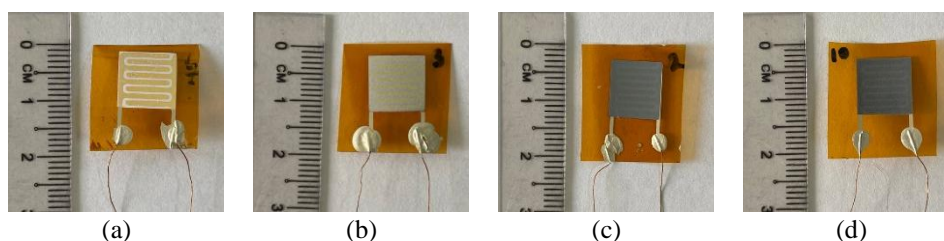


Figure 1. Fabricated ZnO and graphene-doped ZnO gas sensors; (a) ZnO, (b) ZnOG1, (c) ZnOG3, and (d) ZnOG5

2.3. Experimental setup for methane sensing

The experimental gas setup consists of a gas chamber connected to a mass flow controller and a vacuum. The sensor was placed vertically and directly to the inlet gas hose to ensure the gas flow reached the sensing layer of the gas sensor. Measurements were conducted at room operating temperature to assess the gas sensor's sensitivity to methane gas. Gas sensing measurements were carried out in a gas chamber with a methane concentration of 6,700 ppm. The carrier gas used was nitrogen at a flow rate of 50 sccm. Initially, nitrogen at a concentration of 50 sccm flowed into the gas for 30 minutes to establish a steady state before flowing into the target gas. Once a steady state had been achieved, methane gas was supplied at a concentration of 6,700 ppm for 30 minutes. A vacuum was utilized when methane flowed to the gas sensor. After that, nitrogen was flowed again to monitor the sensor's recovery. The recovery process was carried out with the vacuum on. The voltage was measured and recorded as the sensor's response using ESP32. The experimental setup for methane detection is shown in Figure 2.

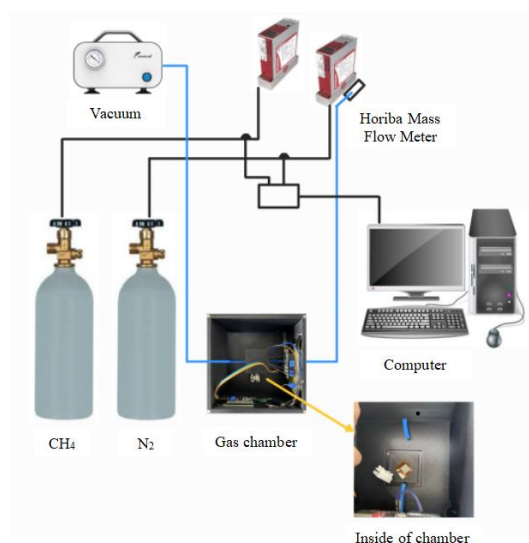


Figure 2. Experimental setup for methane detection

3. RESULTS AND DISCUSSION

3.1. Current-voltage (I-V) characteristics

To determine the conductivity of graphene gas sensors, a supply voltage in the range of -10 V to +10 V was applied to the gas sensor. The gas sensor that produced a linear graph according to Ohm's law verified that the gas sensor was resistance-based and could be exposed to methane gas. Figure 3 shows the I-V characteristics of ZnO and graphene-doped ZnO gas sensors. Figure 3(a) shows the I-V characteristics of the ZnO gas sensor. It was observed that ZnO gas sensor produced more linearity compared to the graphene-doped ZnO gas sensors. Figure 3(b) provides I-V characteristics of the graphene-doped ZnO gas sensor for 1 wt% of graphene. Figure 3(c) depicts I-V characteristics of the graphene-doped ZnO gas sensor for 3 wt. % of graphene. Figure 3(d) illustrates the I-V characteristics of a graphene-doped ZnO gas sensor for 5 wt. % of graphene. It can be observed that the I-V characteristics graph of each graphene-doped ZnO gas sensor was approaching the sinusoidal shape as the concentration of graphene increased in the graphene-doped ZnO gas sensor. The ZnOG5 gas sensor has the most sinusoidal slope due to the highest graphene amount in the sensing layer of the gas sensor. The more sinusoidal the slope of the I-V line, the lower the resistance value of the samples will be obtained. This is due to the slope of the I-V graph being inversely proportional to the resistance.

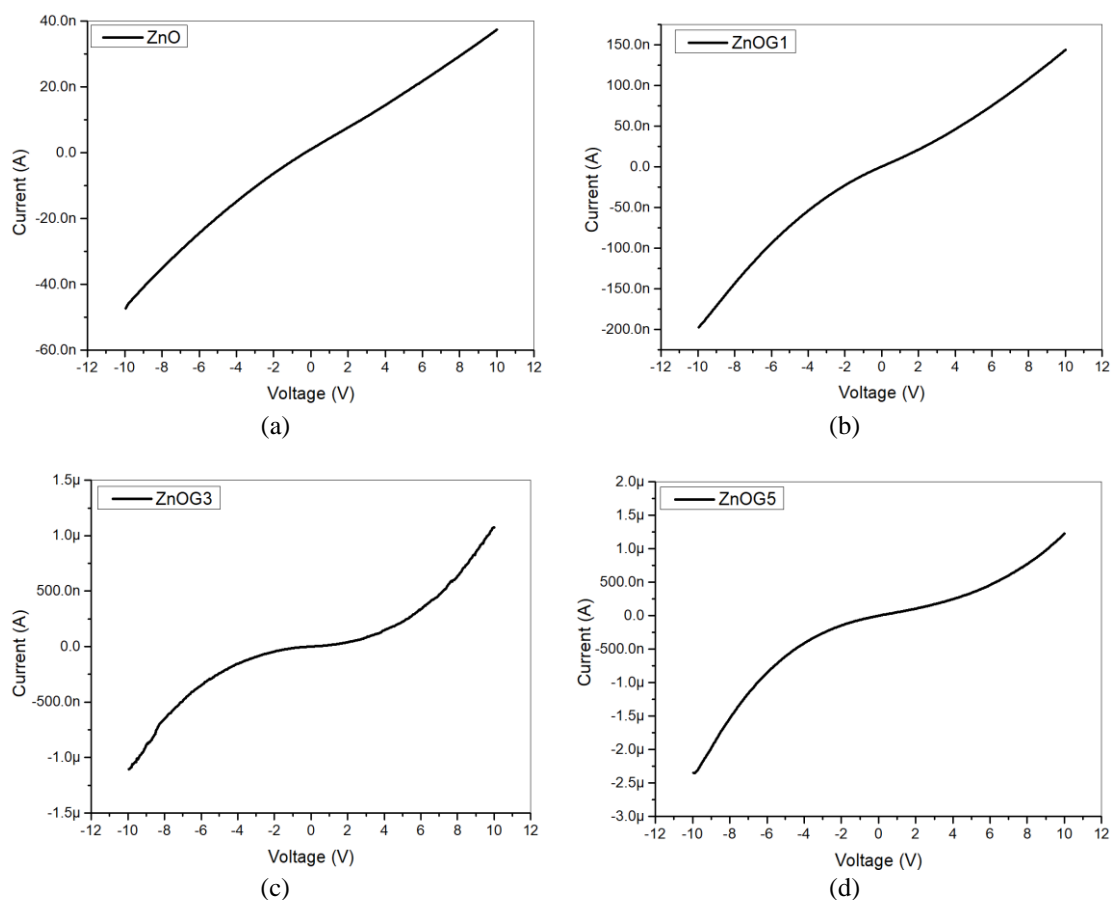


Figure 3. I-V characteristic of ZnO and graphene-doped ZnO gas sensors; (a) ZnO, (b) ZnOG1, (c) ZnOG3, and (d) ZnOG5

Table 1 shows the resistance values for ZnO and graphene-doped ZnO gas sensors. The resistance of the gas sensor can be calculated using Ohm's Law formula, which is $R = \frac{V}{I} = \frac{1}{\text{slope of the graph}}$. It can be seen that the ZnOG5 gas sensor generated the lowest resistance, meanwhile the highest resistance was produced by the ZnO gas sensor. This behavior might have occurred because of its rapid electron mobility; graphene is well known for having great electrical conductivity. Adding graphene to the ZnO increases the material's overall conductivity by creating a conductive network inside the composite. The electrical conductivity of a composite material can be enhanced by the addition of graphene [26]. The application of graphene lowers the overall

resistance of the composite material. This is because graphene has more conductive channels for electrons, which reduces resistance when compared to pure ZnO. The electrical resistance of ZnO composites lowers with increasing graphene content due to the creation of percolation networks of graphene sheets [27]. As reported in other literature, the addition of graphene to metal oxide can reduce the resistivity of the metal oxide [28]. Other than that, the resistance of the gas sensor can also be influenced by the amount of silver paste amount that has been used to attach the copper wire to the leg of the electrode [29].

Table 1. Resistance value for ZnO and graphene-doped ZnO gas sensors

Gas sensors	Resistance value (M Ω)
ZnO	251.00
ZnOG1	65.40
ZnOG3	12.90
ZnOG5	7.27

3.2. Performance of ZnO and graphene-doped ZnO gas sensor to methane gas

Figure 4 shows the dynamic response of ZnO and graphene-doped ZnO gas sensors to 6,700 ppm of methane. The signal strength of ZnO and graphene-doped ZnO decreased when exposed to methane, which this result suggests that all the gas sensors were n-type gas sensors. In the n-typed gas sensor, the voltage decreases in the target gas and increases when exposed to the carrier gas. This behavior was also similar to that discussed in [30], [31]. It can also be observed that all gas sensors were unable to recover to their initial value when exposed to nitrogen during the recovery process. In comparison, the ZnOG5 gas sensor produced the largest voltage changes during methane exposure, suggesting the highest response value. It can also be seen that all voltages of the gas sensors were drastically dropped when exposed to methane, where the fastest decrement was seen by ZnOG3 and ZnOG5, followed by ZnO and ZnOG1. This result revealed that the addition of graphene into the ZnO gas sensor increases the response value of the gas sensor to methane. It can be concluded that graphene as a doping material in ZnO significantly improves the sensing response, reducing resistance and improving the response time of a gas sensor's ability to detect methane due to the high conductivity and large surface area of graphene.

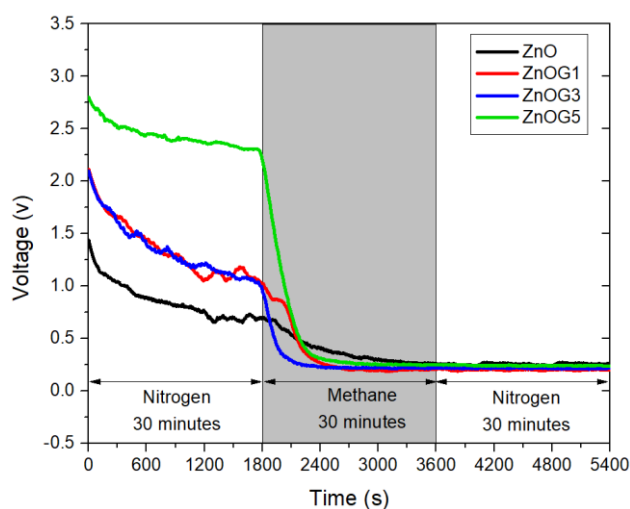


Figure 4. Methane exposure to ZnO and graphene-doped ZnO gas sensors

Table 2 lists the performance of ZnO and graphene-doped ZnO gas sensors in terms of response value and response time. The response value of the gas sensor can be calculated using a formula [32], $\text{Response} = I_{\text{nitrogen}}/I_{\text{methane}}$. The results revealed that ZnOG5 produced the highest response value to the methane, which indicates that a higher amount of graphene in ZnO helps the adsorption of methane gas on the surface of the gas sensor, thus producing a high response value. Whereas ZnOG3 produced the fastest response time to methane compared to other gas sensors, with a response time was approximately 390 s. This result indicated that a higher amount of graphene does not have a large impact on improving the response time to the target gas; however, it can influence the sensitivity of the gas sensor.

Table 2. Performance of ZnO and graphene-doped ZnO gas sensors

Gas sensor	Response value	Response time (s)
ZnO	2.69	1,110
ZnOG1	5.63	750
ZnOG3	8.50	390
ZnOG5	11.50	770

3.3. Interface of ZnO and graphene-doped ZnO gas sensor with Blynk application

To monitor the voltage changes upon exposure to nitrogen and methane gas, a real-time monitoring application was developed using the Blynk application. Figure 5 presents real-time data from ZnO and graphene-doped ZnO gas sensors. Figure 5(a) displays changes in voltage recorded by the Blynk IoT based on the ZnO gas sensor. Figure 5(b) shows changes in voltage recorded by the Blynk IoT based on a graphene-doped ZnO gas sensor for 1 wt. % of graphene (ZnOG1). Figure 5(c) depicts changes in voltage recorded by Blynk IoT based on a graphene-doped ZnO gas sensor for 3 wt. % of graphene (ZnOG3). Figure 5(d) illustrates changes in voltage recorded by Blynk IoT based on a graphene-doped ZnO gas sensor for 5 wt. % of graphene (ZnOG5). It can be observed that the voltages recorded using the Blynk application were similar to the voltages recorded using ESP32 (Figure 4). A similar pattern was also observed using Blynk, in which the voltage was decreased when exposed to the methane. This feature has enabled the real-time monitoring of gas sensors in the future and has allowed people to monitor the gas changes via any platform, such as computer or mobile.

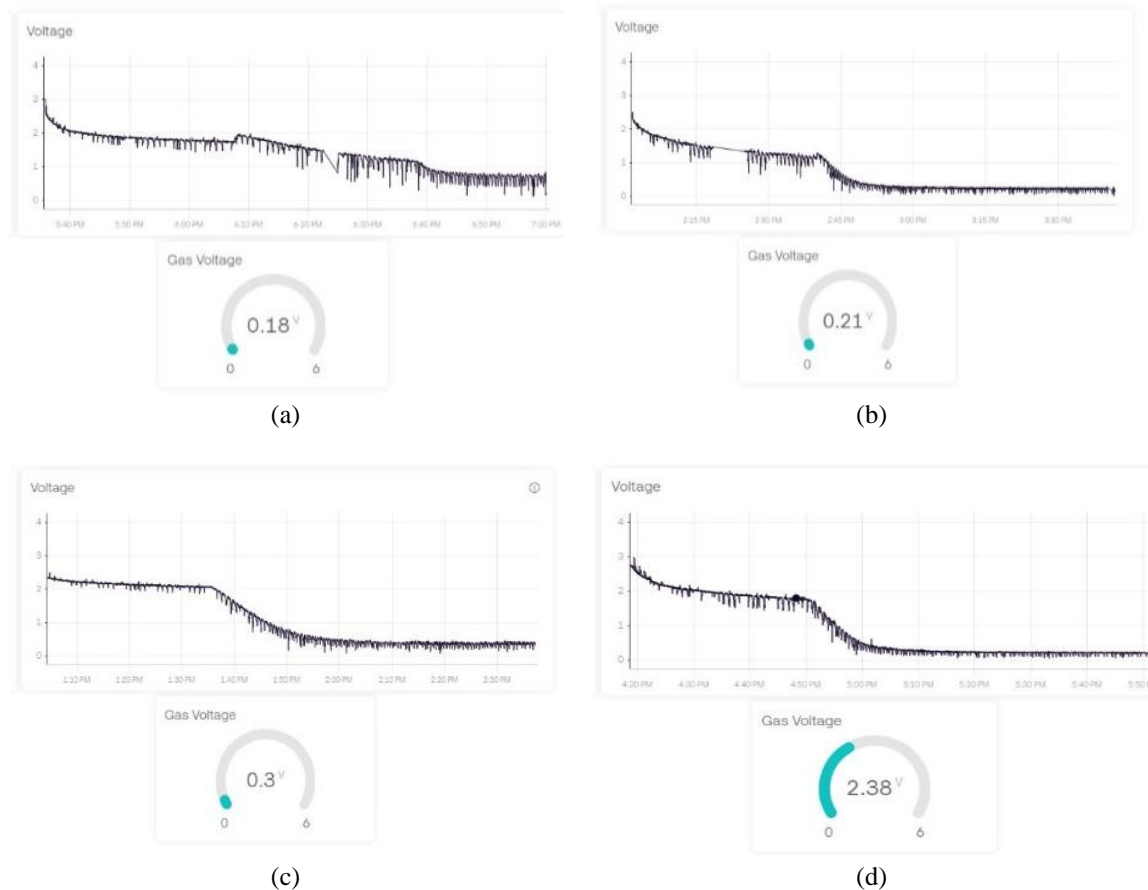


Figure 5. The interface of Blynk IoT during methane exposure for ZnO and graphene-doped ZnO gas sensors; (a) ZnO, (b) ZnOG1, (c) ZnOG3, and (d) ZnOG5

4. CONCLUSION

Resistance-based gas sensors using ZnO and graphene-doped ZnO have been successfully fabricated using screen-printing technology and exposed to 6,700 ppm of methane at room operating temperature. The experimental results revealed that ZnO and graphene-doped ZnO gas sensors were sensitive to methane at room

temperature. The highest sensing response was produced by ZnOG5, with a response value was 11.5, while the fastest response time was recorded by ZnOG3, with a value was 390 s. The addition of graphene into ZnO proved that higher sensing response to methane, which shows that graphene is a promising doping candidate for the enhancement of methane detection. The results also revealed that all the gas sensors had the most stability during methane exposure. For future work, higher graphene amounts will be added into ZnO to investigate their effect on methane sensing. With the use of the Blynk application as an IoT platform, this gas sensor can be applied in a real environment of gas sensing.

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AUTHOR CONTRIBUTIONS STATEMENT

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Name of Author	C	M	So	Va	Fo	I	R	D	O	E	Vi	Su	P	Fu
Siti Amaniah Mohd Chachuli	✓	✓		✓		✓			✓	✓				
Fatin Liyana Syakinah Mohd Yatim					✓			✓	✓					
Nur Hazahsha Shamsudin						✓	✓		✓					
Omer Coban	✓		✓							✓				

C : **C**onceptualization

M : **M**ethodology

So : **S**oftware

Va : **V**alidation

Fo : **F**ormal analysis

I : **I**nterpretation

R : **R**esources

D : **D**ata Curation

O : **O**riginal Draft

E : **E**diting

Vi : **V**isualization

Su : **S**upervision

P : **P**roject administration

Fu : **F**unding acquisition

CONFLICT OF INTEREST STATEMENT

Authors state no conflict of interest.

INFORMED CONSENT

We have obtained informed consent from all individuals included in this study.

DATA AVAILABILITY





The data that support the findings of this study are available from the corresponding author, [Siti Amaniah Mohd Chachuli], upon reasonable request.

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