

Graphene/PSMA composite for gas sensing application

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ABSTRACT

This paper presents the fabrication of conducting polymer sensor which comprise of graphene-poly styrene-co-maleic acid (PSMA) composite that responds to volatile organic compound (VOC) via a change in the electrical resistance of the sensors. Five sensors composed of different material ratio were fabricated to find out the most prominent weight percentage ratio (wt %) for optimum sensor response. Those materials were deposited onto silver electrode using drop-casting method. NI Card PCIE-6323 measures the voltage obtained from the sensor circuit and the resistance of the gas sensor monitored using LabVIEW instrument. It was observed from the experiments that combination of 60% graphene and 40% PSMA gives the highest sensor response.

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1. INTRODUCTION

Gas sensor refers to a device which can selectively respond to certain properties of the environment and can transfer this response into the electrical signals. This device has caught researchers' attention and has undergone a lot of development in the last few decades and now used to fulfill the industrial needs. Human nose is not suitable to be used as a gas sensor in industrial field since it is less accurate, and prone to fatigue [1]. Gas sensor in another hand has several advantages such as; it does not require properly trained personnel and not subject to individual breakdown or variation of sensitivity. In addition to that, once calibrated they can perform odor assessment on a continuous basis with a minimal cost and takes comparatively very little time. Studies on gas sensor had been conducted to widen the application for diverse purposes like medical diagnose [2-6], environmental control [7-11], and food industry [12-16]. The ability of gas sensor to measure, characterize, and discriminate between complex gas mixtures presents promising applications in those reported industries. Besides, research related to the gas sensor helps in development of multisensory communication. In current research, digital smell technology could let us transmit odors through the internet [17]. The process is by sending odor to the cloud using gas sensor, and regenerate the smell.

The common types of gas sensors that have attracted attention of many users including researchers and scientists in gas sensing field includes conducting polymer sensor, piezoelectric sensor, optical sensor, metal-oxide-semiconductor field-effect transistor (MOSFET) sensor, and conductivity sensor. Conducting polymer type gas sensor offers many advantages as compared to other types of gas sensor. Wide range of polymeric

material available in the market allows a high discrimination in array sensor since different polymer gives different response to a given odorant. Besides, this type of gas sensor requires a simpler fabrication technique, signal reproducibility, and enhanced sensitivity [18]. Conducting polymer sensor works using percolation theory principle. As the polymer composite sensor is exposed to vapor sample, some of the vapor permeates into the polymer, causing the polymer to expand. This expansion reduces the number of conducting pathway for charge carriers. Subsequently, this polymer increases the electrical resistance of the polymer composite [19].

This paper studies the effort that has been carried out to fabricate five conducting polymer sensors compose of graphene-polystyrene-co-maleic acid (PSMA) composite that compound volatile organic compounds (VOC). Graphene nanomaterial shows great promise for applications in nano-electrics, flexible electronics, sensors, and solar cells [20]. However, the percentage of combination between graphene and polymers that gives the best performance has not yet been identified. It is necessary to find the ideal weight percentage responds to volatile organic ratio of the composite for future development of gas sensor.

2. SENSOR FABRICATION AND MEASUREMENT

To analyze performance of the sensor, several experiments were performed. The block diagram of the whole setup is demonstrated in Figure 1, which consists of sensor chamber, bubbler, National Instrument (NI) my-DAQ, pump, power supply, and computer. Sample gas was tested on the sensors which located inside the metal chamber. The pump flushed the odor vapor through inlet tube. By using interface circuitry, sensor was connected to the computer via NI my-DAQ

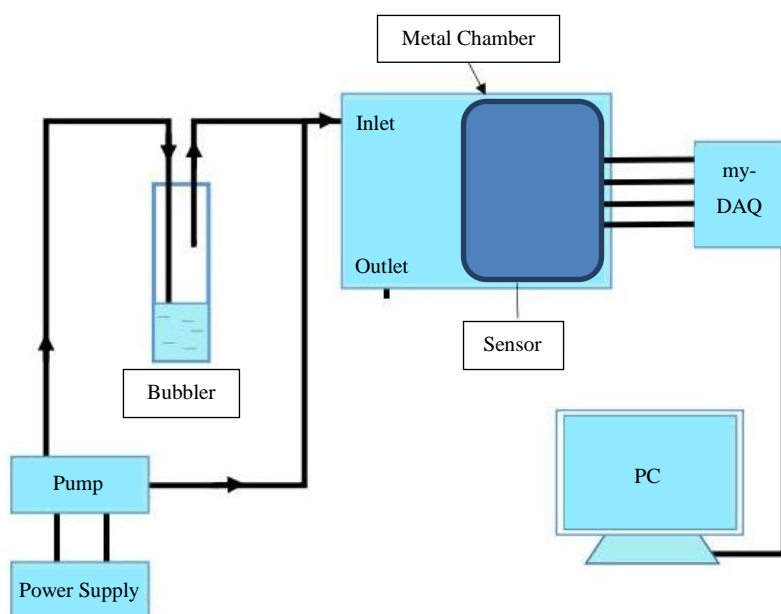


Figure 1. Schematic diagram of experiment

In order to measure resistance changes upon odor exposure, sensor circuit which consists of Wheatstone Bridge was designed. When the bridge is fully balanced, the right-side resistors which are identical to the left side resistors ($R_1=R_2$, and $R_3=R_x$), and the voltage across the bridge is zero. However, with a small change in resistance on one of the resistors, the bridge becomes unbalanced, and a voltage difference appears. This feature of Wheatstone Bridge makes it suitable to be used as measuring circuit for sensors. The sensing material was deposited onto the interdigitated electrode using drop casting technique up to five layers, but it cannot be seen considering the color of graphene-PSMA mixture is crystal clear.

2.1. Chemical preparation

Ultrapure pristine graphene monolayer flakes (dispersion in ethanol) with concentration of 1 mg/L were procured from graphene supermarket containing average flake thickness 0.35 nm, and average particle (lateral) size ~550 nm (150-3000) nm. PSMA ($M_w=65,000$) was purchased from Sigma-Aldrich. Since PSMA is in solid form, it needs to be dissolved with ethanol. The ratio of graphene weight is too small

as compared to PSMA weight. Hence PSMA solution was diluted for two times, to ensure that PSMA weight is small enough to be mixed with graphene. Polymer solutions were prepared from 10 mg PSMA and 10 ml ethanol. After being completely dissolved, 1 ml of the mixture was added to 9 ml ethanol to dilute the solution. They were homogeneously dispersed by physical agitation for 30 minutes with the aid of digital ultrasonic cleaner at room temperature. Graphene was used as received without any further treatment. Five types of sensor were fabricated with different graphene/PSMA ratio. The identification name of each sensor and their respective ratio are listed in Table 1. Amount of material to be deposited was calculated based on its weight percentage (wt %), where:

$$\text{weight percentage (wt\%)} = \frac{\text{weight of solute}}{\text{weight of solution}} \times 100\%$$

Table 1. Ratio and quantity of composite material

Label	Graphene ratio (%)	Graphene quantity (ml)	PSMA Ratio (%)	PSMA volume (ul)
Sensor A	70	0.5	30	2.1
Sensor B	60	0.5	40	3.3
Sensor C	50	0.5	50	5.0
Sensor D	30	0.5	70	12
Sensor E	20	0.5	80	20

Drop casting technique was deployed using micro pipette with different ranges. Two ul of the solution was deposited on a sensor substrate with an active area (0.25”×0.25”) consisting of Au interdigitated electrodes. This process was repeated until resistance was between 3 M ohm and 9 M ohm. As-deposited composite films were dried at room temp for 1 hour. Figure 2 shows the complete setup for this experiment. Sensor was placed into a metal chamber. Microdiaphragm pump (Model NMP 30 KNDC, KNF Neuberger Inc., Trenton, NJ) was connected by polyethylene tubing to a bubbler. A laboratory DC power supply was used to supply 5V voltage, and data acquisition hardware (NI, my-DAQ) was used to measure the voltage drops across the sensors.

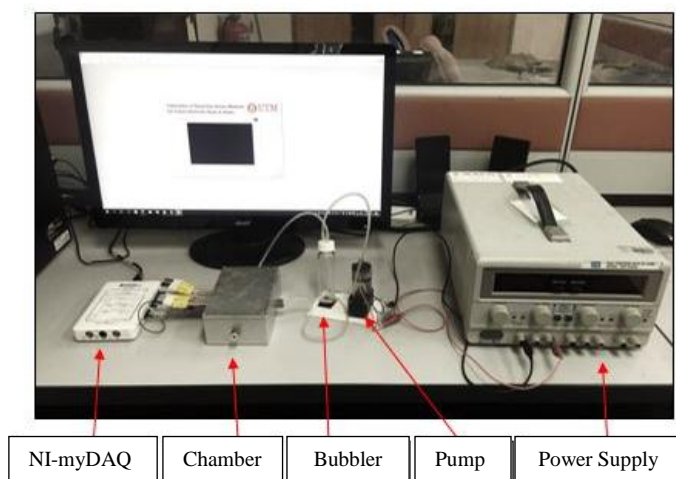


Figure 2. Complete setup of experiment

2.2. Experimental procedure

In this experiment, Pucelle perfume was tested as the vapor samples, and environment air was used as the baseline. This sensor was designed for gas sensor application, and hence VOC was tested instead of identifying single compound. 3 mL of the vapor samples were transferred to screw-capped 10 mL bottles bubbler. The pump flown the reference gas to the chamber for 20 seconds to create a baseline voltage, followed by gas under test for 30 seconds, and flow reference gas to clean the chamber for 20 seconds. The perfume concentration was controlled in each experiment by delivering the same pump voltage specifically 5 V. The time gap between each repetition is set to 5 minutes, and the same sensor was deployed in each repetition. The baseline voltage for all five sensors is 2.5 V.

3. RESULTS AND DISCUSSION

Each sensor was deposited with graphene/PSMA composite and with different weight percentage (wt %). To test the sensor performance, Pucelle perfume was utilized as the subject for this experiment. The response magnitude for each sensor was observed to investigate graphene/PSMA sensing capability towards odorant. In addition, the response time and recovery time for each sensor was determined and discussed. The fractional baseline manipulation was calculated to verify which sensor gives the best response towards Pucelle perfume.

3.1. Characteristics of graphene/PSMA sensor

In each experiment, three readings were taken and named as Repetition 1, Repetition 2, and Repetition 3. From Figure 3, Repetition 1 was labeled with blue colored line, Repetition 2 labeled with orange colored line, and Repetition 3 labeled with grey colored line. The voltage measurement of sensor deposited with 70% Graphene (0.5ml) and 30% PSMA (2.1 ul) labelled as Sensor A has been carried out with three repetitions and the results are shown in Figure 3.

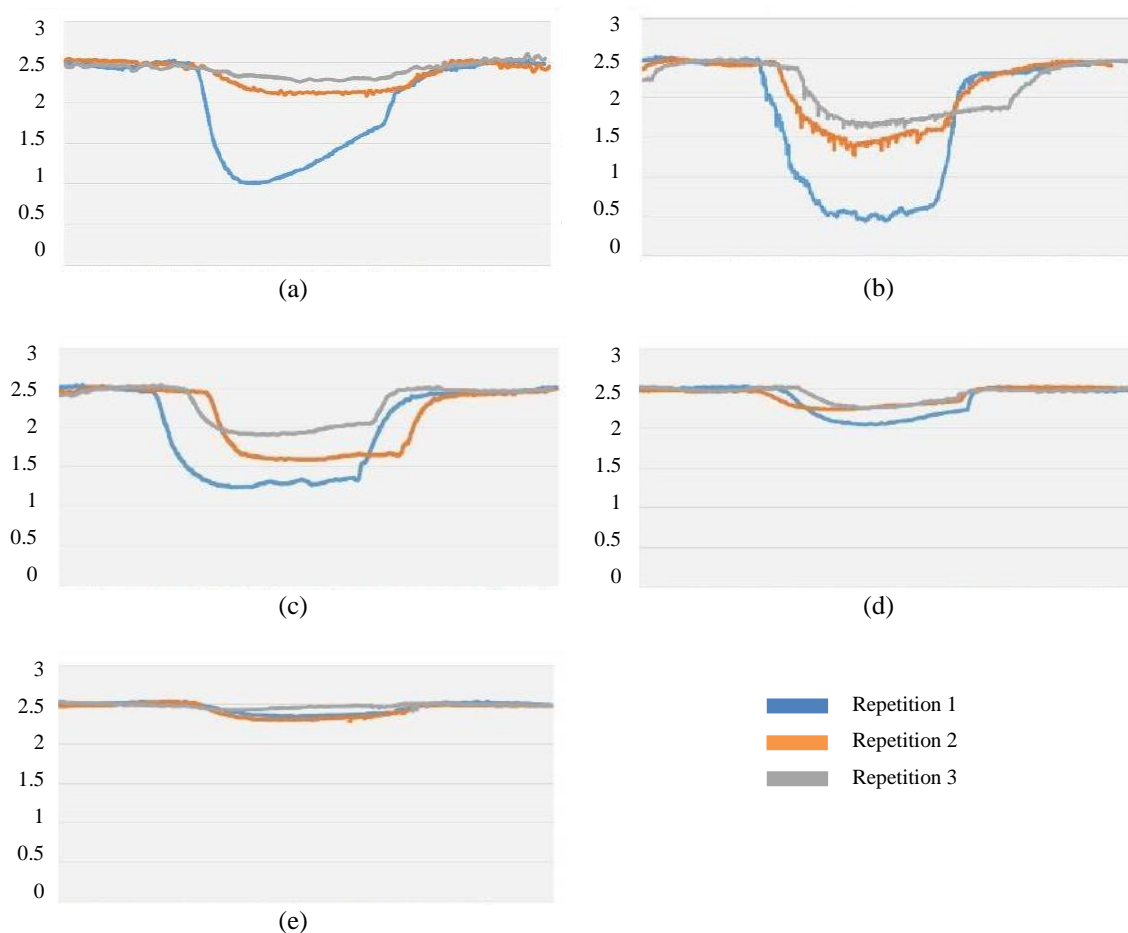


Figure 3. The characteristics of five sensors, (a) Sensor A (70 % graphene, 30% PSMA), (b) Sensor B (60% Graphene, 40% PSMA), (c) Sensor C (50% graphene, 50% PSMA), (d) Sensor D (30% graphene, 70% PSMA), (e) Sensor E (20% graphene, 80% PSMA)

From Figure 3(a), the voltage achieves the highest peak of 1 V when exposed to perfume vapor and drop to its baseline value (2.5 V) as reference air was flushed into it. According to [21], the general experimental voltage reading should rise when exposed for testing gas and slump as reference gas is flushed into the chamber. In this experiment, the first repetition illustrates a slight decline before the reference gas is flushed into the chamber. This fall off may due to graphene/PSMA structure itself. As the graphene content in the composite is dominant, a small expansion of polymer would disrupt the conducting pathway between the charge carriers and hence increase the electrical resistance between electrodes. Besides, this reduction

may occur due to sensor poison. The response time, recovery time, and sensitivity of each repetition was calculated and recorded in Table 2. Repetition 1 has the shortest response time (91 s) as compared to Repetition 2 (129 s) and Repetition 3 (177 s). However, it takes a longer time to reach baseline value when flushed with reference air (165 s). The sensitivity is calculated to be 0.6, which is much greater than Repetition 2 (0.17) and Repetition 3 (0.10).

Table 2. Characteristics of Sensor A

Repetition	Response time (s)	Recovery time (s)	Sensitivity (%)
1	91	165	0.60
2	129	67	0.17
3	177	154	0.10

Responds for Sensor B which deposited with 60% graphene (0.5 ml) and 40% PSMA (3.3 ul) was illustrated in Figure 3(b). For Repetition 1, the voltage dropped to 0.43 V when exposed to perfume vapor. This sensor shows the largest voltage difference (2.07 V) as compared to the other sensors. Hence, it was considered as the most sensitive sensor for detection of Pucelle perfume. The summary of response time, recovery time, and sensitivity of each repetition was shown in Table 3. Although Repetition 1 has a longer response time (112 s) as compared to Repetition 3 (99 s), it still gives the highest sensitivity (0.83). As for recovery time, Repetition 3 shows the fastest (179 s) and Repetition 1 is the slowest (217 s).

Table 3. Characteristics of Sensor B

Repetition	Response time (s)	Recovery time (s)	Sensitivity (%)
1	112	217	0.83
2	124	188	0.44
3	99	179	0.33

The voltage measurement of sensor deposited with 50% Graphene (0.5 ml) and 50% PSMA (5 ul) labelled as Sensor C had been carried out with three repetitions and the result is shown in Figure 3(c). The voltage achieved the highest peak of 1.2 V when exposed to perfume vapor and drop to its baseline value (2.5 V) as reference air was flushed into the chamber. The voltage difference for Repetition 1 is 1.27 V. The summary of response time, recovery time, and sensitivity of each repetition was shown in Table 4. For response time, Repetition 2 shows the shortest (52 s), followed by Repetition 3 (76 s), and Repetition 1 (107 s). As for recovery time, the shortest is Repetition 3 (46 s), followed by Repetition 2 (206 s), and Repetition 1 (248 s). However, Repetition 1 shows the highest sensitivity (0.51) as compared to Repetition 2 and 3.

Table 4. Characteristics of Sensor C

Repetition	Response time (s)	Recovery time (s)	Sensitivity (%)
1	107	248	0.51
2	52	206	0.37
3	76	46	0.24

Figure 3(d) shows the result for Sensor D which deposited with 30% graphene (0.5 ml), and 70% PSMA (12 ul). For Repetition 1, the respond fell from 2.5 V to 2.04 V, which shows a voltage difference of 0.46 V. The summary of response time, recovery time, and sensitivity of each repetition was shown in Table 5. Repetition 3 has the shortest response time (87 s), followed by Repetition 2 (96 s), and Repetition 1 (133s). However, Repetition 1 has the shortest recovery time (27 s) and the highest sensitivity (0.18).

Table 5. Characteristics of Sensor D

Repetition	Response time (s)	Recovery time (s)	Sensitivity (%)
1	133	27	0.18
2	96	44	0.11
3	87	55	0.10

Figure 3(e) shows the result for Sensor E which deposited with 20% graphene (0.5 ml), and 80% PSMA (20 ul). The voltage for Repetition 1 reduced from 2.5 V to 2.33 V, which shows a voltage difference of 0.17 V. The summary of response time, recovery time, and sensitivity of each repetition was shown in Table 6. Repetition 1 has the longest response time (169 s) as compared to Repetition 2 (110 s) and Repetition 3 (51 s). It has the longest recovery time (83 s) as well. The sensitivity is not much different from Repetition 2 and Repetition 3.

Table 6. Characteristics of Sensor E

Repetition	Response time (s)	Recovery time (s)	Sensitivity (%)
1	169	83	0.07
2	110	78	0.09
3	51	54	0.04

Figure 4 illustrates the response magnitude of five kinds of sensors when exposed to Pucelle perfume. Each colored line represents a separate sensor output from each sensor type. Sensor A represented by the green colored line, red colored line represents Sensor B, while yellow colored line represents Sensor C. Sensor D represented by purple colored line and Sensor E represented by blue colored line. Generally, each sensor shows similar trend which comparable to each other.

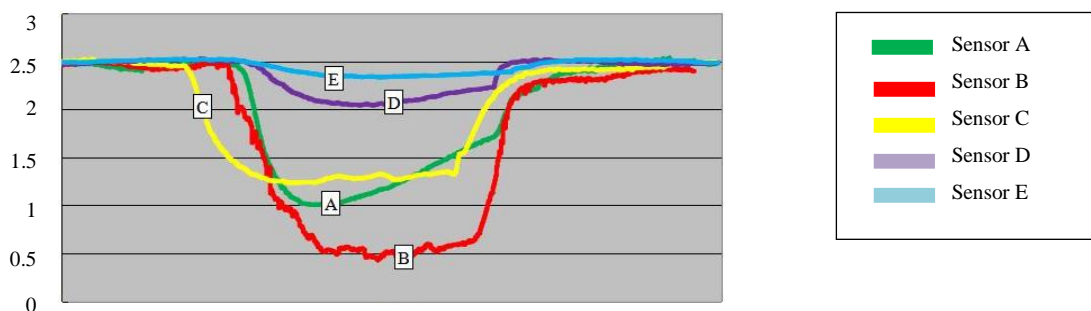


Figure 4. Comparison of five sensors

It can be clearly seen from the graph that different sensors provide different response towards the same odorant. Each sensor proved the capability of graphene/PSMA based sensor to detect presence of Pucelle perfume. However, their sensitivity, response time and recovery time varied due to different weight percentage (wt %) of graphene and PSMA mixture. The resistivity of each sensor results from the expansion of polymer when exposed to odorant. This expansion reduces the number of conducting pathways for charge carriers that naturally present in this composite. For this reason, the weight percentage (wt %) of polymer in the graphene-polymer composite consequently affect the resistivity of the sensor. As reported in [19], responds of each sensor are different for different odorant. Some of the sensors might be more sensitive for specific odorant but less sensitive for another odorant. By comparing all five sensors in this experiment, sensor B which deposited with 60% graphene and 40% PSMA gives the highest sensor response towards Pucelle perfume. It could be concluded as the dominant sensor to detect the presence of Pucelle perfume's vapor. However, this sensor might not be the best sensor to detect another odorant.

Sensor A which deposited with 70% graphene and 30% PSMA has the steepest gradient of response compared to the other four sensors, which means Sensor A has the fastest response time towards perfume vapor (91 s). It was then followed by Sensor C (107 s), Sensor B (112 s), Sensor D (133 s) and Sensor E (169 s). As for recovery time, Sensor D has shortest time (27 s), followed by Sensor E (83 s), Sensor A (165 s), sensor B (217 s), and Sensor C (248 s). The summary of response time, recovery time, and sensitivity for each sensor was reported in Table 7.

Table 7. Summary for each sensor response

Sensor	Response time (s)	Recovery time (s)	Sensitivity (%)
A	91	165	0.60
B	112	217	0.83
C	107	248	0.51
D	133	27	0.18
E	169	83	0.07

4. CONCLUSION

Utilization of graphene/PSMA based gas sensor in this study has provided sufficiently strong sensor response to detect organic volatile compound. In addition, results from experiments demonstrated that combination of 60% graphene and 40% PSMA gives the highest sensor response for measurement of the subject. This result was concluded prior to three times experiment repetition.

As an extension to improve and optimize the sensor system, it would be interesting to diversify the type of polymer to produce a new sensor. Compilation of multiple weight percentage of graphene with different polymers may be investigated in future works, to find out whether 60% graphene and 40% PSMA combination still works for another graphene-polymer combination. In term of fabrication technique, problem arises when graphene acquired from the market has a very low concentration. It is difficult to quantify the polymer volume in order to obtain the desired wt % ratio. Therefore, in future research graphene may be produced via other methods such as Hummers Offeman's method which has been used by another researcher [22, 23]. In another hand, condition of the experiment should be strictly maintained to get a more accurate result. In particular, the humidity and flow pressure should be continuously monitored. Sensitivity of the sensor significantly affected as this control keeps changing. Hence to overcome this problem, a humidity sensor, and pressure sensor should be installed inside the sensor chamber in future research. Besides, inkjet printing technique should be implemented to replace drop-casting technique. Although using this technique is quite complex and expensive, it would give a very high precision material deposition process and able to control the deposition process in a more detailed way. Li *et al.* [24] and Secor *et al.* [25] in their research use this inkjet printing method for deposition of graphene. This inkjet printing technology allows a wide range of substrate includes cloth and transparent paper. Hence, a flexible graphene-based gas sensor could be fabricated in the future study.

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