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Organic Field-Effect Transistor Based on Poly 3-hexylthiophene as Ammonia Vapor Sensor

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An organic field-effect transistor (OFET) based on poly 3-hexyl thiophene (P3HT) as an active layer has been developed to detect ammonia vapor. Silicon dioxide (SiO_2) as the dielectric layer on this FET is grown on the surface of the *p*-type silicon substrate by heating in an oxygen atmosphere in the furnace at 1000 °C for 3 hours, oxygen gas (O_2) is flowed into the furnace during the heating process. The results of the energy-dispersive X-ray spectroscopy (EDS) characterization showed that the silicon substrate which had been oxidized contained about 35 % O_2. P3HT as the active layer was grown on the surface of the SiO_2 layer using the spin coating method. The *I-V* characteristics of FET show that the drain-source current (I_{DS}) is affected by changes in the gate voltage (V_G). The greater the V_G, the higher the *I*d-s produced. The effect of ammonia vapor shows that I_{DS} on the *I-V* curve decreases with increasing concentration of ammonia vapor. The dynamic response of the FET to ammonia vapor shows that the greater the V_G, the higher the sensitivity of the device. For V_G = 0 volt, the sensitivity of FET in ammonia sensing is 0.236 volt/%, meanwhile for V_G = – 8 volt, the sensitivity of FET in ammonia sensing is 0.264 volt/%.

Keywords: Ammonia, Field-effect transistor, Poly 3-hexylthiophene, SiO_2.

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1. INTRODUCTION

Ammonia with an unpleasant odor is one of the gases that can harm human health. Short-term effect may cause irritation to the respiratory tract, nose, throat and eyes [1, 2]. Contact with eyes can cause irritation to total blindness. Meanwhile, contact with the skin can cause burns. The long-term effect of inhaling high concentrations of ammonia gas continuously can cause damage to the lungs and cause death [3]. Because of the dangers that can be caused by ammonia gas, it is important to develop a system or tool that can be used to detect the presence of ammonia gas early. Ammonia gas sensors have developed rapidly in the last 50 years by using materials that respond to the presence of ammonia gas by various mechanisms [4-6].

One class of materials often used as gas sensors (including ammonia) are conducting polymers [7-9]. Conducting polymers are often used as active layers that can detect the presence of gas as well as its concentration. Conducting polymer-based sensors have many advantages over commercial sensors made of metal oxides. Conducting polymer-based sensors have high sensitivity, short response time and operate at room temperature, while metal oxide-based sensors operate at elevated temperatures [10, 11]. The high operating temperature required by gas sensors based on metal oxides causes the use of this device to be inefficient and consumes a large amount of energy [12]. Therefore, we need other materials that do not require high operating temperatures. Conducting polymers as active coatings on gas sensors have been shown to have high sensitivity in detecting gases (including ammonia), even though they operate at room temperature [13, 14].

Poly-pyrrole (Ppy) [13, 15], polyaniline (PANI) [14], polythiophene [16], and their derivatives are conducting polymers that can be used as active coatings for gas. Thiophene polymers are relatively stable in free air and in aqueous environments and have high hole mobility. Polythiophene is a type of heterocyclic aromatic conducting polymer which is almost similar to pyrrole. One of the thiophene polymers is poly 3-hexyl thiophene (P3HT), which is also often used for ammonia gas sensors [16-18].

Field-effect transistor (FET) devices can be used for ammonia gas sensors [19-23]. FET-structured sensors have more measurement parameters compared to chemiresistors, but have better sensitivity. The field effect transistor (FET) is a voltage-controlled device, meaning its output characteristics are controlled by the input voltage. FET consists of three terminals, where the conduction between the two terminals source and drain is controlled by modulation of the electric potential of the third terminal, namely the gate. The FET structure is illustrated in Fig. 1. Poly 3-hexyl thiophene (P3HT) has been used as active layer in OFET for ammonia gas sensor [24-26].

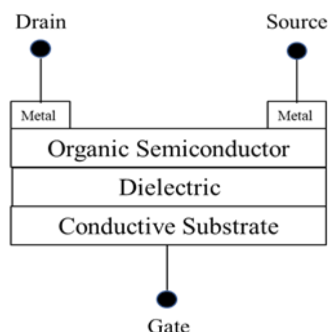

Fig. 1 – Structure of Field-Effect Transistor (FET)

FETs work by controlling the distribution of charge carriers in an active semiconductor using an internal electric field. If a *p*-type organic polymer is used, applying a negative voltage to the gate electrode will result in an accumulation of positive charges in the active layer around the dielectric surface. When sufficient charge carriers have accumulated, the conductivity of the charge accumulation region increases, consequently increasing the current between drain and source [12].

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In this research, a conducting polymer poly 3-hexylthiophene (P3HT) based organic FET will be developed to detect ammonia gas. The P3HT layer can interact with ammonia gas, so that the concentration of ammonia gas that participates in the reaction can be changed in the form of physical quantities.

2. EXPERIMENTAL DETAILS

2.1 Equipment and Materials

The equipment used in this study were beakers, measuring cups, glass slides, glass cylinders, volumetric pipettes, gloves, masks, pipettes, glass cutters, analytical balances, syringes, chambers, ultrasonic baths, hotplates, furnace, spin coater, Keithley 2400 I-V meter, and PASCO Science Workshop 750 Interface. The materials used are silicon wafer, oxygen gas, poly 3-hexylthiophene, chloroform, distilled water, dry water, H_2O_2, H_2SO_4, acetone, and ammonia gas.

2.2 Growth of SiO_2 Layer on the Surface of *p*-type Silicon Substrate

The SiO_2 layer was grown on the surface of the p-type silicon substrate by heating the silicon surface while stream oxygen gas. Initially, the p-type silicon substrate was washed with acetone in an ultrasonic bath for 30 minutes, which aims to remove dissolved organic impurities attached to the silicon. Furthermore, the silicon is immersed in peroxymonosulphuric acid for 15 minutes, this treatment aims to remove insoluble organic impurities on the silicon. Then the silicone is rinsed with distilled water. After that, the heating process was carried out in a furnace for 3 hours at a temperature of 1000 °C. Oxygen gas is streamed into the furnace during the heating process. To ensure that SiO_2 has grown on *p*-type silicon substrate, characterization was carried out using energy-dispersive X-ray spectroscopy (EDS). The EDS spectrum provides information on the presence of elements forming the SiO_2 layer, namely silicon and oxygen. The percentage of elements contained in the SiO_2 layer can be seen from the high intensity of each spectrum.

2.3 Poly 3-hexylthiophene (P3HT) Coating

The active layer of poly 3-hexylthiophene (P3HT) was coated on top of the SiO_2 surface using the spin coating method. As much as 2 mg of P3HT was dissolved in 4 ml of chloroform, then stirred using a magnetic stirrer at 400 rpm for 10 minutes. P3HT solution was dripped onto the surface of SiO_2, then rotated at 1000 rpm using a spin coater. Then it was heated with a hotplate at 80 °C for 1 hour.

2.4 Formation of Electrode Contacts

The contacts are aluminum layers deposited by the thermal evaporation method at a pressure of 10^{-5} Bar. Aluminum layer is deposited on the surface of the P3HT layer for the source and drain contacts, respectively. Meanwhile, the gate contact is deposited onto the surface of the *p*-type silicon substrate on the reverse side.

2.5 Current-Voltage Characterization of OFET

Current-voltage (*I-V*) characterization of FET is carried out using a Keithley 2400 Source Meter. The power supply is used as a voltage source. Measurements are made by connecting the negative polar of the power supply to the gate electrode and the negative polar of the *I-V* meter connected to the drain electrode. Meanwhile, the positive polar of the power supply and *I-V* meter is connected to the source electrode (Figure 2).

Fig. 2 – Circuit for measuring the *I-V* characteristic of OFET in ammonia vapor presence

2.6 Testing the Response of OFET to Ammonia Vapor

Testing the response of the OFET to ammonia vapor was carried out in the test chamber. Constant voltage is supplied to the gate (V_G). Varying voltage is supplied to the drain (V_D), while the drain current (I_D) is measured. This measurement is carried out while streaming ammonia vapor into the test chamber with a gradually increasing concentration. The drain current (I_D) will change when the active layer (P3HT) of OFET interacts with gas molecules, which increases with increasing concentration of ammonia vapor (Figure 3).

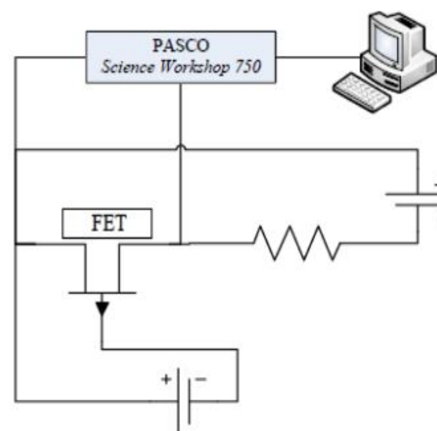


Fig. 3 – Circuit for measuring the response of OFET to ammonia vapor

Testing the dynamic response of the FET is carried out by inserting the FET device into the test chamber and then applying a voltage using a 9-volt battery. The gate electrode is connected to the power supply. The FET device is connected in parallel with a voltage sensor that

has been connected to an interface (PASCO Science-Workshop 750) which is connected to a personal computer (PC) to retrieve data. The tests were carried out in two different conditions, namely when the gate voltage was not applied (V_G = 0 volt) and when the gate voltage was given (V_G = – 8 volt).

At first the sensor was run in condition without ammonia vapor, then inserted ammonia vapor using a syringe into the chamber and held for a while. The voltage rises to a stationary state, this is caused by an increase in resistance in the P3HT active layer [22]. After that, the concentration of ammonia vapor is increased with the same concentration as the first concentration so that the concentration of ammonia gas in the chamber becomes twice the initial concentration of ammonia vapor and is held for a while. The voltage rises again until it reaches a stationary state. Then the addition is the same as the second addition so that the concentration of ammonia vapor in the chamber becomes three times the initial concentration of ammonia vapor and is held for a while. The voltage rises again until it reaches a stationary state. Then the chamber lid is opened so that the ammonia vapor in the chamber comes out until it runs out and the ammonia gas concentration in the chamber becomes zero, which causes the voltage to drop back to its original state, so that the dynamic response curve of the OFET device to the ammonia vapor concentration is obtained.

3. RESULTS AND DISCUSSION

3.1 SiO_2 Layer

A layer of silicon dioxide (SiO_2) as a dielectric layer on the FET device is grown on the surface of the silicon substrate by heating in an oxygen atmosphere. The silicon substrate is heated in the furnace at 1000 °C for 3 hours, while streaming oxygen gas (O_2) into the furnace. The presence of the SiO_2 layer was confirmed by characterization using EDS. Figure 4 shows the formation of a SiO_2 layer on a p-type silicon substrate after being heated in an oxygen atmosphere, which is indicated by a brownish yellow color on the surface of the p-type silicon substrate.

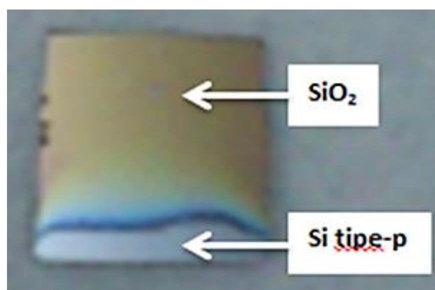
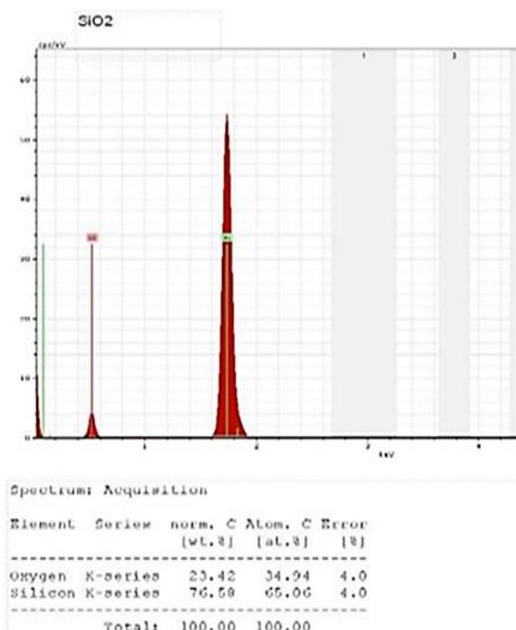


Fig. 4 –*p*-type silicon substrate after oxidized

Figure 5 shows the EDS spectrum of oxidized *p*-type silicon surface. The EDS spectrum indicates the presence of elemental oxygen on the surface of *p*-type silicon which has been oxidized to form a SiO_2 layer. The percentage of elemental oxygen on the oxidized surface is known to be about 35 % and that of elemental silicon is about 65 %. Thus, a SiO_2 layer has been successfully grown on the surface of a *p*-type silicon substrate.



Spectrum: Acquisition

Element	Series	norm. C [wt.%]	Atom. C [at.%]	Error [%]
Oxygen	K-series	23.42	34.94	4.0
Silicon	K-series	76.58	65.06	4.0
	Total:	100.00	100.00	

Fig. 5 – EDS spectrum of SiO_2 layer on *p*-type silicon

3.2 *I-V* Characteristics of OFET

I-V characterization was carried out using a Keithley 2400 Source-Meter (*I-V* meter). The drain and source electrodes are connected to the *I-V* meter, the gate electrode is connected to the power supply. The supply voltage at the drain varies from 0 to – 10 volts and the voltage at the gate varies from 0 to – 8 volts. The *I-V* characteristics of OFET can be seen from the curve of the drain-source current (I_{DS}) to the drain-source voltage (V_{DS}) by providing a variation of the gate voltage (V_G).

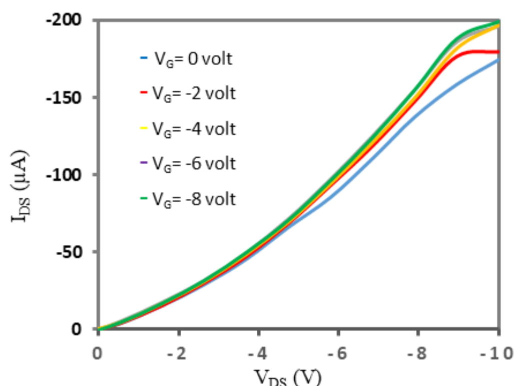


Fig. 6 – I_{DS} vs V_{DS} curves of OFET at varied gate voltage (V_G)

Figure 6 shows the *I-V* curve of OFET when the gate voltage (V_G) is applied, the drain-source current (I_{DS}) changes exponentially. The greater the gate voltage (V_G), the greater the drain-source current (I_{DS}) will be. This is because the active layer P3HT is a *p*-type conducting polymer, so that when a negative voltage is applied to the gate, a positive charge accumulates in the active layer around the dielectric surface [12]. This accumulated charge causes the drain-source current (I_{DS}) to increase.

3.3 *I-V* Characteristics of OFET in Presence of Ammonia Gas

This test is carried out by placing the OFET into the test chamber and then applying a constant voltage to the gate electrode of – 8 volt (V_G) using a power supply. The FET is connected to the *I-V* meter which is already connected to a personal computer (PC). *I-V* data were measured by varying V_{DS} while recording the I_{DS}, at constant gate voltage at V_G = – 8 V. *I-V* data were taken for each varied ammonia gas concentrations, start from 0 %, 1.887 %, 3.774 % and 5.660 %, respectively.

Figure 7 shows the drain-source current (*I*d-s) to drain-source voltage (V_{DS}) curve with increasing ammonia gas concentration, or a fixed gate voltage (V_G = – 8 V). When the gas is exposed, the curve of drain-source current (I_{DS}) to the drain-source voltage (V_{DS}) changes. The I_{DS} vs V_{DS} curve (Fig. 7) shows that the slope of drain-source current (I_{DS}) vs drain-source voltage (V_{DS}) is decreasing as the concentration of ammonia gas increases. The decrease in the slope of the drain-source current (I_{DS}) at constant gate voltage (V_G = – 8 V) is caused by an increase in the resistance of the P3HT active layer.

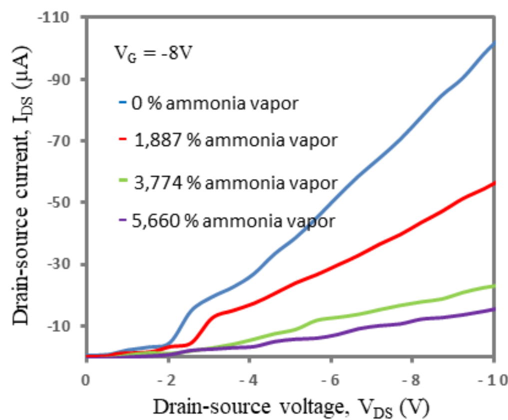


Fig. 7 – Curves of I_{DS} vs V_{DS} at constant gate voltage (V_G = –8 V) for varied ammonia gas concentrations (0 %, 1.887 %, 3.774 % and 5.660 %)

Figure 8 shows graph between I_{DS} with ammonia concentration at V_{DS} = – 10 V for V_G = –8 V which taken from *I-V* data (Fig. 7) for each varied ammonia concentration exposed. The graph shows a decreasing linear curve for lower concentration of ammonia vapor with slope of about 25 μA (I_{DS})/% ammonia concentration. This means that the P3HT active layer-based OFET has highly sensitive response to the ammonia vapor.

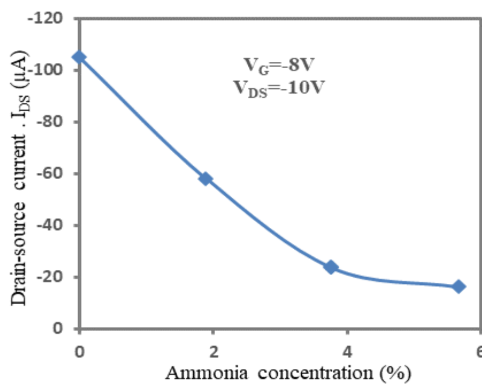


Fig. 8 – Graph between I_{DS} at V_{DS} = – 10 V vs concentration of ammonia gas for V_G = – 8 V

3.4 Dynamics Response of FET in Varied Concentration of Ammonia Vapor

Figure 9 shows the dynamic response of the FET to varying concentrations of ammonia gas. When exposed ammonia vapor with a concentration of 1.887 % and held for a while, the voltage rises to a stationary state. On the addition of ammonia vapor so that the concentration becomes 3.774 % and is held for a while, there is also an increase in the voltage up to a stationary state. Likewise with the addition of ammonia vapor concentration with a concentration of 5.660 %. When the chamber is opened, ammonia vapor comes out of the chamber, so that the concentration of ammonia vapor in the chamber decreases, which causes the voltage to drop back to its original state. Based on the dynamic response curve (Figure 9), it can be found that the applied gate voltage (V_G) influences the output voltage. The greater the gate voltage (V_G) applied, the higher the resulting output voltage.

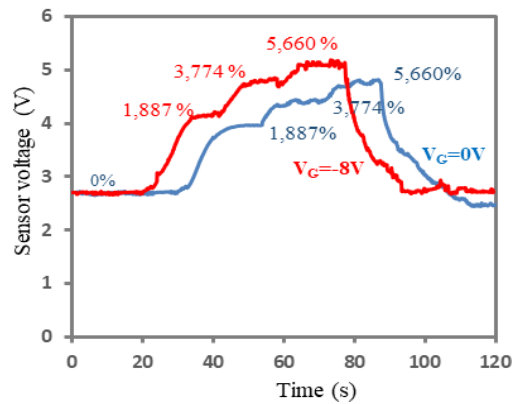


Fig. 9 – Dynamics response of OFET at V_G = 0 V and V_G = – 8 V in varied ammonia gas concentration (%)

Based on the dynamic response curve, it appears that three values of stationary voltage increase with increasing concentration of ammonia gas. From these data, a stationary voltage curve for the concentration of ammonia gas is made. Then the sensitivity value of the FET device to the concentration of ammonia gas can be determined from the slope of the curve that is ratio of the change in output voltage to the concentration of ammonia vapor.

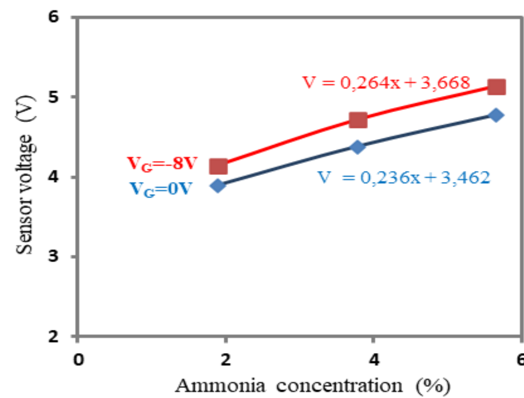


Fig. 10 – Stationary voltage vs ammonia concentration at V_G = 0 V and V_G = – 8 V

Figure 10 shows the curve between voltage and ammonia gas concentration. Based on the curve, in the

range taken, the stationary voltage is directly proportional to the applied gate voltage (V_G). The greater the gate voltage, the higher the sensitivity of the FET device. When given $V_G = -8$ V, for every 1 % change in concentration, there will be an increase in voltage of 0.264 V. Meanwhile, when $V_G = 0$ volt, for every 1 % change in concentration, there is an increase in voltage of 0.236 V.

4. CONCLUSIONS

The organic FET was successfully made by coating an active layer of poly 3-hexylthiophene (P3HT) on the surface of SiO_2 as dielectric layer on *p*-type silicon substrate using the spin coating method. A SiO_2 dielectric layer was grown on the *p*-type silicon substrate by heating method. The *I-V* characteristic curve shows the drain-source current (I_{DS}) is affected by the gate voltage (V_G). The greater the V_G, the more the drain-source current (I_{DS}) increased. The response of the organic FET to ammonia gas is based on the *I-V* characteristic curve of the organic FET and the dynamic response of the organic FET to the gas concentration of ammonia vapor. The effect of ammonia vapor on the *I-V* characteristic curve shows that the I_{DS} decreases as the concentration of ammonia vapor increases. The dynamic response of the organic FET to ammonia vapor shows that the greater the V_G, the more sensitive the organic FET sensor is. When supplied $V_G = -8$ V, every 1 % increase in ammonia concentration results in an increase in voltage of 0.264 volt. When supplied $V_G = 0$ V, every 1 % increase in ammonia concentration results in a voltage increase of 0.236 V.

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Органічний польовий транзистор на основі полі-3-гексилтіофену як датчик парів аміаку

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Органічний польовий транзистор (OFET) на основі полі-3-гексилтіофену (P3HT) як активного шару був розроблений для виявлення парів аміаку. Діоксид кремнію (SiO_2) як шар діелектрика на цьому польовому транзисторі вирощується на поверхні кремнієвої підкладки *p*-типу шляхом нагрівання в атмосфері кисню в печі при 1000 °C протягом 3 годин, газоподібний кисень (O_2) надходить у піч під час процесу нагрівання. Результати характеристик енергодисперсійної рентгенівської спектроскопії (EDS) показали, що кремнієва підкладка, яка була окислена, містила близько 35 % O_2. P3HT як активний шар був виро-

щений на поверхні шару SiO_2 за допомогою методу центрифугування. ВАХ польового транзистора показує, що на струм стік-витік (IDS) впливає зміна напруги затвора (V_G). Чим більша V_G, тим вищий отриманий ідентифікатор. Вплив парів аміаку показує, що вплив IDS на ВАХ зменшується зі збільшенням концентрації парів аміаку. Динамічний відгук польового транзистора на пари аміаку показує, що чим більше V_G, тим вище чутливість пристрою. Для V_G = 0 вольт чутливість польового транзистора при вимірюванні аміаку становить 0,236 вольт/%, тоді як для V_G = – 8 вольт чутливість польового транзистора при вимірюванні аміаку становить 0,264 вольт/%.

Ключові слова: Аміак, Польовий транзистор, Полі-3-гексилтіофен, SiO_2.