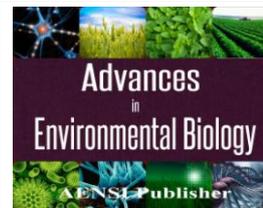




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### Integrating WO<sub>3</sub> Nanostructure on IDE for Ethylene Detection Feasibility

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#### ARTICLE INFO

##### Article history:

Received 28 February 2014

Received in revised form 25 May 2014

Accepted 6 June 2014

Available online 20 June 2014

##### Keywords:

Keyword 1 (Font: Times New Roman)

Spt) Keyword 2 Keyword 3

(At least 3 and at most 8 keywords)

#### ABSTRACT

**Background:** Ethylene, a gaseous plant hormone plays a very important role as regulator of plant growth and development. As far as precision agriculture is concern, the ability of detection ethylene will enable the complete cycle of the fruits and crops to be monitored; from flowering stage up to ripening stages. This data is very important to trigger the necessary activities such as fertilizing, pollination and harvesting to be conducted during the ideal timing to maximize the yield. **Objective:** This paper shares the effort to fabricate ethylene sensor by integrating tungsten trioxide (WO<sub>3</sub>) on silicon base interdigitated (IDE) platform via facile hydrothermal method. **Results:** This simple and medium temperature bottom up process produced sporadic multi-dimensional nanostructure across the IDE. When ethylene react with nanostructure, the potential energy barrier at the gain boundary will change hence impacts the overall resistance value of the sensor. **Conclusion:** preliminary testing at ambient condition shows that the sensor has a linear response towards low p.p.m level of ethylene. This positive result will drive for further characteristic and process optimization to produce high sensitivity, selectivity and sustainable ethylene sensor.

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**To Cite This Article:** Amirul Abd Rashid, Nor Hayati Saad, Daniel Bien Chia Sheng., Integrating WO<sub>3</sub> Nanostructure On IDE For Ethylene Detection Feasibility. *Adv. Environ. Biol.*, 8(8), 2732-2735, 2014

### INTRODUCTION

Ethylene had attracted research area particularly in precision agriculture area due to their numerous effects on the growth, development and storage life of many fresh commodities at molecular concentration [1,2]. This colourless and odourless gas is responsible for the changes in texture, softening, colour and other process involved in ripening especially for climacteric fruits includes apples, bananas, melons, apricots and tomatoes [3]. By accurately measuring the ethylene gas level, the food supply chain management is in better control to determine the correct time for harvesting as well as in controlling the logistic of the food itself. In recent review by Cristescu *et al.* [4], he suggest that there are three main categories of methods can be used to detect ethylene; gas chromatography (GC), optical and electromechanical sensing. Among them, electrochemical technologies shows better advantage in lower p.p.m level capability, good repeatability and accuracy, smaller in dimension and consume relatively low power consumption. Under this technology domain, state of the chemoresistive sensor utilizing nanotechnology sensing material starts to emerged. Esser *et al.* [5] mixed single-wall carbon nanotubes (SWNTs) with copper complex to detect the present of ethylene through the change of resistance. While the success of this sensor is proven, the processes of producing SWNTs require high temperature and high power process. Therefore, a much simple yet effective process such as hydrothermal should be explored towards producing fast response, sensitive and selective at lower production cost which matches with economic of scale required for mass sensor production [6].

#### Methodology:

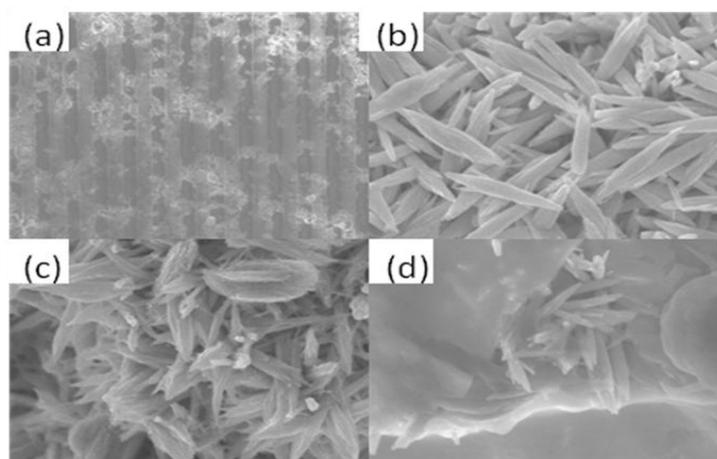
WO<sub>3</sub> nanostructure was grown on micro IDE via facile hydrothermal synthesis process following similar setup by Feng *et al.* [7]. The aqueous solutions were prepared by dissolving 8.25g of sodium tungstate dehydrate powder (Na<sub>2</sub>WO<sub>4</sub>·2H<sub>2</sub>O) in de-ionized (DI) water. The pH value was modified to 2.4 by adding oxalic acid (H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>). After 30 min stirring at ambient temperature, the IDE sensor package was immersed in the salt solution inside 100 ml Teflon lined autoclave reactor. The reactor was heat up to 130° C for 8 hours. The sensor were dried at 60° C for about 5 minutes. For morphology analysis. scanning electron microscopy (JEOL, JSM-7500 F) with 5 kV voltage acceleration was used to examine the WO<sub>3</sub> nanostructure morphology grown on IDE.

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To investigate ethylene detection capability, the sensor was connected to LCR meter and initial resistance in air reading ( $R_a$ ) was recorded. Subsequently, the sensor was located horizontally inside the chamber which contains specific p.p.m level of ethylene which was measured by industrial grade commercial Ethylene sensor (Detcon, USA) earlier. Once the resistance reading was stable, the value ( $R_g$ ) was recorded and the sensitivity of the as-synthesize nanostructure on IDE was calculated. The gas sensitivity ( $S$ ) of the sensor toward Ethylene gas is defined as the ratio of the stationary electrical resistance of the sensing materials in the test gas ( $R_g$ ) to the resistance in air ( $R_a$ ) or initial resistance; that is  $S = R_g / R_a$ . The plot of sensitivity versus ethylene p.p.m level was plotted accordingly.

## RESULTS AND DISCUSSION

Figure 1 shows the SEM of  $WO_3$  nanostructure layered on IDE surface. In general it can be seen that the nanostructure was sporadically distributed in between the IDE (Figure 1 (a)). At higher magnification, the nanostructure actually contains multidimensional structure which consist of 1D (nanorods) 2D (thick film slab) and 3D (nanoflower like).



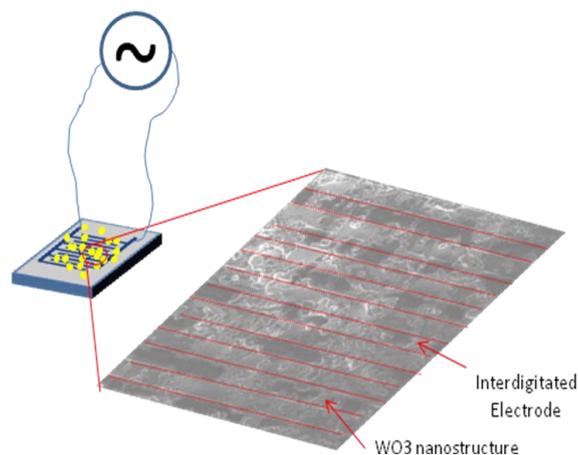
**Fig. 1:** Sem images of the structure shows distribution on IDE (a), nanorods (b), nanoflower like (c) and thick film (d).

The resistance measurement of such integrated IDE was performed following the electrical connection illustrated in Figure 2. The sensor was connected with voltage sources of 1 V at 100 kHz and the LCR meter will show in-situ resistance reading on air as well as when the sensor is located inside the testing chamber. The resistance value versus the p.p.m level of ethylene was plotted in Figure 3. Based on 3 point measurement, the resistance of the sensor increased linearly with the increased of ethylene p.p.m. More points of measurement were needed to confirm the actual resistance behaviour of the sensor.

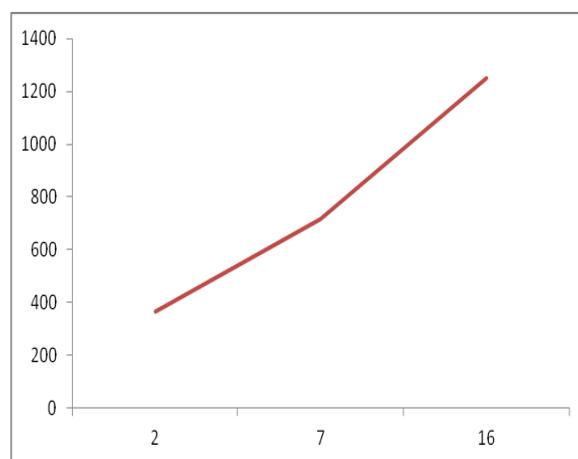
In this feasibility study, the testing was done at ambient condition. On top of that, the ethylene was measured not in continuous manner because the sensor was removed from the chamber while the p.p.m level was being adjusted. It is proposed to conduct continuous monitoring to avoid any resistance variation due to taking in and out of the sensor because it may have reacted with surrounding during that time. At this point of time, the sensitivity of the sensor towards 3 different p.p.m levels as tabulated in Table 1.

**Table 1:** Sensitivity of  $WO_3$  nanostructured IDE sensor to different ethylene concentration.

Ethylene Concentration (p.p.m)	2	7	16
$R_a$	260	220	246
$R_g$	366	715	1250
Sensitivity	1.4	3.25	5.08



**Fig. 2:** Schematic of electrical connection of the sensor to measure resistance.



**Fig. 3:** Plot of resistance value Vs Ethylene p.p.m level.

#### Conclusion:

This paper reports the initial development of chemoresistive sensor to detect low concentration ethylene.  $\text{WO}_3$  with multidimensional nanostructure was successfully integrated with silicon IDE platform via facile hydrothermal process. Despite the non-homogenous nanostructure observed on the device, the fabricated sensor shows a good response towards different ethylene concentration. Further characteristic study can venture base on this preliminary work towards fabrication of ultra sensitive, selective and sustainable ethylene sensor in the future.

#### ACKNOWLEDGEMENTS

The authors wish to thank MIMOS Berhad, Universiti Teknologi MARA (UiTM) and Ministry of Higher Education Malaysia (MOE) for the financial support under ERGS grant (File No : 600-RMI/ERGS 5/3 (18/2013)).

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