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CADMIUM DETECTION BY POLYANILINE-ZINC OXIDE COMPOSITE THIN-FILM SENSOR

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ABSTRACT

The conventional analytical techniques for detection of cadmium (Cd) requires expensive equipment and skilled operators. Although sensor technology developed nowadays are miniature and user-friendly, most of them are high in fabrication costing and complex in preparing its sensing material. As such, composite of polyaniline (PANI) and zinc oxide (ZnO) are selected to fabricate a simpler thin-film sensor with lower costing for Cd detection. Cd concentration is varied in the range from 1 ppm to 100 ppm are used to analyze the sensitivity of the sensor, and the result shows that a higher Cd concentration leads to a higher voltage reading, in the range of 18.16 mV to 65.10 mV. This is attributed to the mechanism of Cd^{2+} ions adsorption on the surface of ZnO, which induces a change in conductivity. Besides, PANI functions as a conducting polymer that stabilizes ZnO to enhance the detection performance.

Keywords: Polyaniline, zinc oxide, oxidative polymerization, cadmium, thin-film sensor

1.0 INTRODUCTION

Cd leads to poisoning when its intake is at high concentration exceeding the amount that our bodies can tolerate [1]. Applications within industrial, technological and agricultural fields have caused a wide distribution of Cd to the soil, water and air, which endangers the inhabiting living organisms [2]. Cd contaminated drinking water and food are few of the major Cd sources entering the human bodies. Cd^{2+} ions are non-biodegradable and will accumulate in human bodies and other living organism [3]. In addition, the World Health Organization (WHO) has designated Cd as one of the human carcinogens, as a long exposure to Cd may lead to cancer [4].

Therefore, the detection of Cd is crucial especially for a drinking water source. Several methods have been used for the detection of Cd to safeguard the human health and the environment. Conventional methods include atomic absorption spectroscopy (AAS), inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma optical emission spectrometry (ICP-OES) [5], but these techniques are high costing, huge, and generally require scheduled maintenance and skilled staff to operate the equipment [6].

As of now, sensor technology has been developed to overcome the limitation of these conventional methods. This is because sensors are miniature in size, making them portable and user-friendly with a lower cost. There are several types of sensor developed for Cd detection, namely: colorimetric sensor, electrochemical sensor and biosensor. Each of them can detect Cd well, but they do have limitations.

Colorimetric sensor provides an optical detection towards Cd. This sensor undergoes colour change when Cd is detected [7]. This type of sensor is based on the transduction principle, i.e. a change in light absorption of a chemochromic material [8]. However, this type of sensor usually requires huge data sets of Red-Green-Blue (RGB) values that needs to be analysed using chemometric methods [9]. In addition, complex sensing materials are usually used in sensor fabrication, such as 4-amino-3-hydrazino-5-mercapto-1,2,4-triazole [10].

Electrochemical sensor uses the concept of electrochemistry by measuring the change of conductivity in the analysed medium [11], thus it consists of three electrodes, namely; working, reference and auxiliary electrode [12]. Mercury (Hg) is widely used as the sensing material for Cd detection a decade ago, which is, however, very toxic to the user and environment [12]. Although the environmental-friendly bismuth (Bi) is later introduced as the sensing material, the preparation from its relevant oxide is complex [13].

Biosensor is a type of sensor using the enzyme as the sensing material for the detection. One of its qualities is that it is very selective in detection [11]. Its concept is similar to that of an electrochemical sensor, and its transducer functions to convert any biological event on the sensing area into a detected signal [13]. Some examples of the enzyme used for Cd detection is alkaline phosphatase [11], urease [14], invertase, and acetylcholine esterase [15], but they are not easily prepared as well.

In this study, the composite of polyaniline (PANI) and zinc oxide (ZnO) is used as the sensing material for the detection of Cd, by using the sol-gel method for thin-filming. ZnO is widely used for gas detection, such as H_2, methane and carbon monoxide [16]. It is attributed to the mechanism of O^{2-} chemisorption onto its surface which involve the transfer of charges when reaction occurred between the chemisorbed O_2 and targeted gas molecules [17]. Besides, ZnO is also able to adsorb the Cd^{2+} ions onto its surface, leading to subsequent formation of $CdOH^+$ ions or $Cd(OH)_2$ with the hydroxyl group that dissociates at the ZnO surface [18]. This study is to apply the concept of Cd^{2+} ion adsorption onto the ZnO surface into the sensor technology.

ZnO layer alone is unstable when immobilized on the electrode surface, thus requiring an enzyme or conducting polymer for immobilization [19]. When ZnO is immobilized with enzyme, it can only be used to detect its respective substance. For example, glucose oxidase composite with ZnO is only able to detect glucose [20]. Hence, conducting polymer is a better option.

PANI is chosen as it is a widely used conducting polymer and can be easily prepared by oxidative polymerization [21]. Besides, PANI is low cost and simple in its fabrication [22]. Moreover, PANI can be used to detect several gas molecules, such as hydrogen sulfide (H_2S) and ammonia (NH_3), which attributed to the mechanism of chemisorption model [23]. Hence, it is possible that this would be helpful in the Cd detection. Nevertheless, PANI possess high porosity which helps in its mechanism upon detection [23], and ZnO as well [19].

The fabrication of the thin-film sensor in this study is based on the concept of sol-gel method [24], where the solution of PANI-ZnO composite dissolves in its solvent, 1-methyl-2-pyrrolidone (NMP) with thermal treatment, and then dropped onto the electrode and deposited as a thin film on it.

The aim of this study is to fabricate a PANI-ZnO composite thin-film sensor for the Cd detection, to investigate the effect of ZnO composited in PANI, and to analyse the sensitivity of the sensor by using varying concentrations of Cd (1 ppm, 25 ppm, 50 ppm, 75 ppm and 100 pm).

2.0 METHODOLOGY

The fabrication of PANI-ZnO composite thin-film sensor requires the preparation of composite sensing material and thin-filming fabrication on sensor. Besides, various concentrations of Cd (1 ppm, 25 ppm. 50 ppm, 75 ppm and 100 ppm) are needed to be prepared. The repeatability test and concentration test was carried out using the same and different concentrations of Cd respectively.

2.1 Material

Aniline (ACS reagent 99.5%), ammonium persulfate (APS, 98%), methanol (ACS reagent) and 1-methyl-2-pyyrrolidone (NMP, 99.5%) purchased from Sigma-Aldrich. Zinc oxide (ZnO, Pharmacopoeia Grade 99%) was obtained from SYSTERM. Hydrochloric acid (HCl, 37%) was purchased from RCL Labscan Limited, whereas sodium hydroxide (NaOH, ACS reagent 99%) was obtained from R&M Chemicals. Besides, conductive silver (Ag) pen was purchased from Polychem UV/EB International Corporation.

2.2 Dilution of Cadmium Solution

Different concentration of Cd (1 ppm, 25 ppm, 50 ppm, 75 ppm, and 100 ppm) was prepared by dilution method with Equation (1) shown as below:

$$M_1V_1=M_2V_2 \tag{1}$$

where M is the concentration of Cd in ppm, V is the volume of Cd in mL, 1 represent stock solution, and 2 represent the target solution.

2.3 Preparation of Polyaniline

First, 83 mL of 37% HCl stock solution was used to prepare 1000 mL of 1M HCl by using 1000 mL volumetric flask and distilled water. Then, 3.7 mL of aniline was poured into 400 mL of 1M HCl to prepare 0.1M of aniline in 1M of HCl. Later, 4.66 g of APS was poured into 400 mL of 1M HCl to prepare 0.05M of APS in 1M of HCl.

Preparation of PANI powder and PANI-ZnO composite powder is based on the process flow as shown in Figure 1.

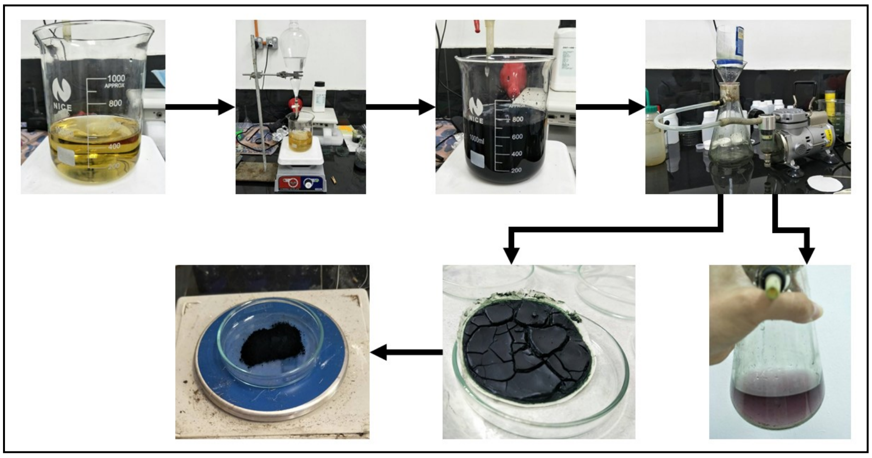

Figure 1 Process flow diagram in preparing PANI and PANI-ZnO composite powder

In the preparation of PANI, 400 mL of 0.05M APS in 1M of HCl was added dropwise into 400 mL of 0.1M aniline in 1M of HCl, and stirred vigorously for 30 minutes, followed by continuous stirring for 8 hours with moderate speed. Then, it was kept overnight and filtered afterward by vacuum filtration. Methanol was used to remove the unreacted aniline and distilled water was used to wash away the methanol during the filtration. Next, the leftover PANI cake was dried at a temperature of 80^{o} C in oven. After that, the dried PANI cake was grinded into powder using pestle and mortar.

During the preparation of PANI-ZnO composite, an additional step was required, i.e. the addition of 1.6 g ZnO (30 wt%) into the 400 mL of 0.1M aniline in 1M of HCl, followed by a stirring of 30 minutes, before adding the APS.

2.4 Fabrication of Sensor

2.4.1 Silver Electrode Fabrication

The conductive Ag pen was used to draw the working and auxiliary electrode on the glass slide and it is dried at a temperature of 110^{o} C for 15 minutes, to guarantee its best conductivity for Cd detection analysis.

2.4.2 Thin-film Fabrication

First, 40 g of NaOH pellets were dissolved in 1000 mL of distilled water to prepare 1000 mL of 1M NaOH. Then, a window for sensing area was fabricated on the glass slide by using hot glue. Next, 0.3 mL of 1M NaOH was added into 0.05 g of PANI powder, followed by 30 mL of NMP solvent with vigorously stirring. After that, 0.3 mL of the mixture was dropped onto the window of working electrode and dried at the temperature of 60^{o} C for the mixture to be evaporated, leaving only a thin-film of PANI on it.

The thin-filming of PANI-ZnO composite is carried out according to the exact same steps as preparing a PANI thin-film sensor, by using PANI-ZnO composite powder, rather than PANI powder. The process of thin-film fabrication is as shown in Figure 2.

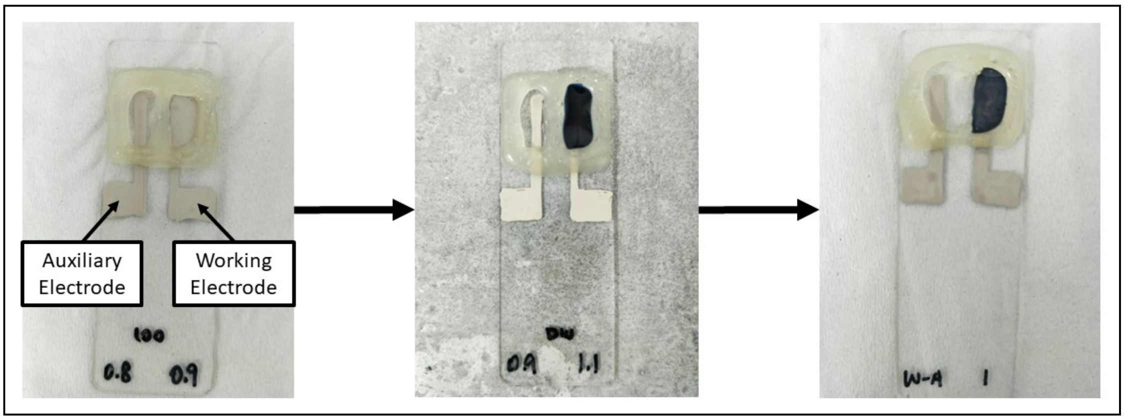

Figure 2 Thin-film fabrication of sensor

2.5 Analysis by Repeatability and Concentration Test

The fabricated PANI thin-film sensor and PANI-ZnO composite thin-film sensor was used to detect various concentration of Cd (1 ppm, 25 ppm, 50 ppm, 75 ppm and 100 ppm). LabVIEW 2011 with NI DAQ was used for Cd detection, and the apparatus set up is shown in Figure 3. Distilled water was used to obtain the reference voltage at the beginning for each sensor.

Then, 0.5 mL of Cd solution was used for the repeatability test with the same sensor to detect the same Cd concentration for three times, consuming about five to ten minutes for each detection. Distilled water was used to clean the surface of sensor after every detection.

0.5 mL of Cd solution was used as well for the concentration test with the same sensor (if it is repeatable), to detect different concentration of Cd in descending order for three cycles. Distilled water was used to clean the surface of sensor after every detection as well. The detection procedure was similar to that of the repeatability test.

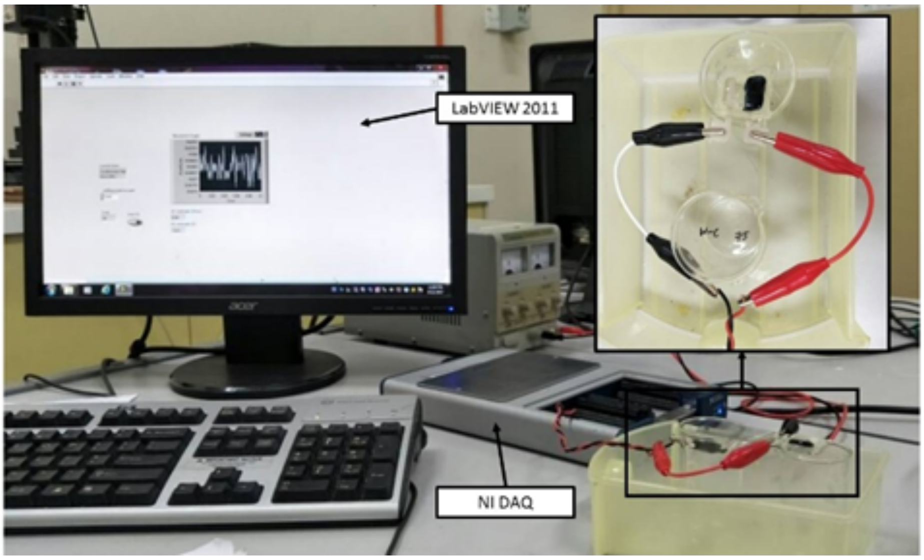

Figure 3 Apparatus set up for analysis procedure

3.0 RESULTS AND DISCUSSION

3.1 Oxidative Polymerization of Aniline

PANI is synthesized through addition polymerization by using aniline as the monomer, and APS as the strong oxidant for oxidizing all the intermediates of PANI during the synthesis [25]. The structure of PANI is shown in Figure 4.

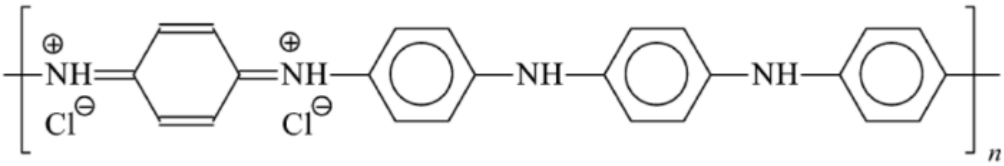

Figure 4 PANI (emeraldine) HCl [26]

3.2 Polyaniline-Zinc Oxide Composite

The composite of PANI-ZnO powder had the same colour as PANI, which is dark green in colour. Its emeraldine base structure of N=Q=N, which the Q represents the quinoid [27], as shown in Figure 5.

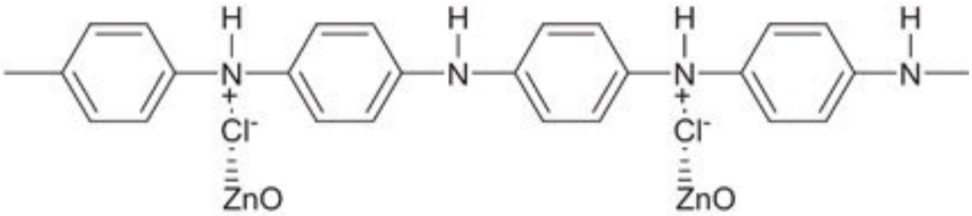

Figure 5 Structure of PANI-ZnO composite [27]

3.3 Thin-film Fabrication

The PANI and PANI-ZnO thin-film sensor can be distinguished by the naked eye, the thin-film of PANI-ZnO composite is dark blue in colour, but the thin-film of PANI is dark green in colour, as shown in Figure 6.

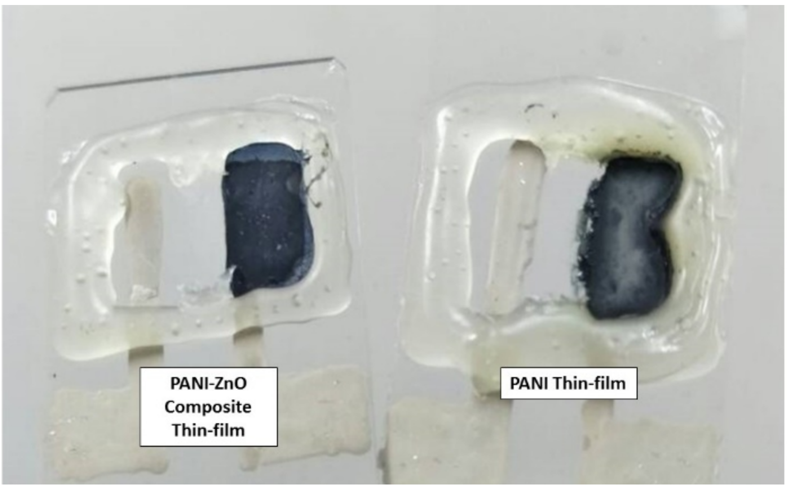

Figure 6 Thin-film fabrication by PANI and PANI-ZnO composite

3.4 Repeatability Test

Both PANI and PANI-ZnO thin-film sensors were tested with the same concentration of Cd for three times. The results shown that the sensor fabricated is available for repeatable usage, and the data were tabulated in Table 1 and Table 2 for PANI and PANI-ZnO composite thin-film sensor respectively.

Table 1 Experimental data of PANI thin-film sensor for repeatability test

Sensor	Concentration Used	Voltage (mV)		
		1st Run	2nd Run	3rd Run
DW-1.8-1.9	100 ppm	-35.14	-34.31	-33.50
25-1.8-1.9	100 ppm	-32.79	-34.24	-35.30

Table 2 Experimental data of PANI-ZnO composite thin-film sensor for repeatability test

Sensor	Concentration Used	Voltage (mV)		
		1st Run	2nd Run	3rd Run
W-A-1	1 ppm	-33.35	-37.67	-37.13
W-B-X	100 ppm	10.95	10.33	9.94

Graph analysis is performed as well, as shown in Figure 7 and Figure 8 for PANI and PANI-ZnO composite thin-film sensor respectively. Based on the graphs plotted with raw data from LabVIEW, it can be concluded that the sensors are able to respond within ten seconds and obtain a constant reading within five to ten minutes as the equilibrium between the sensing material with Cd^{2+} ions is reached.

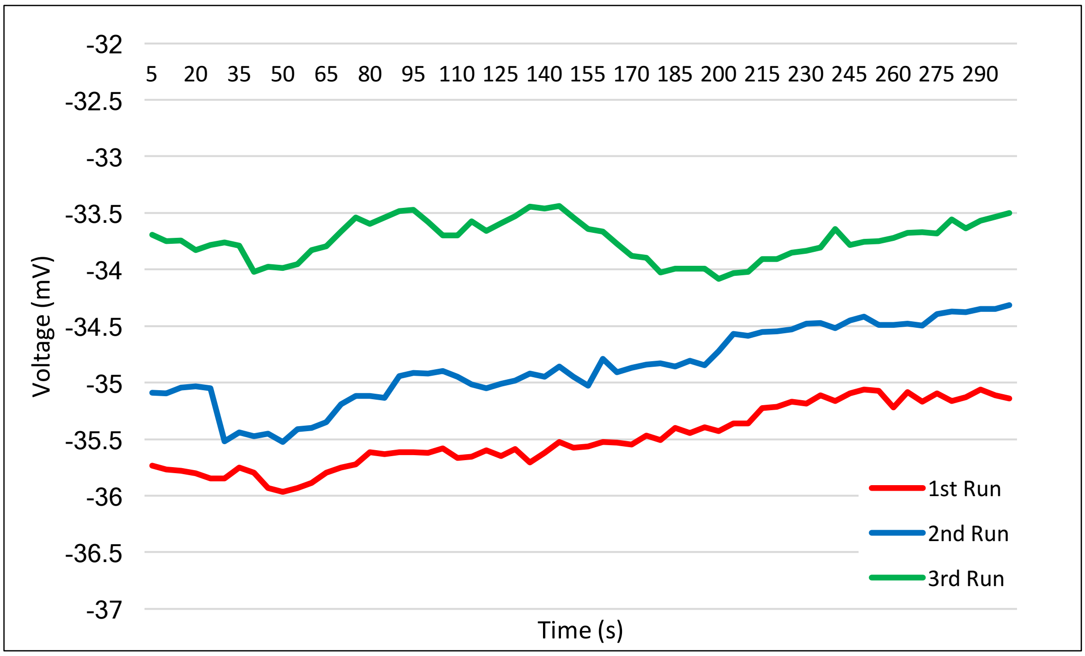

Figure 7 Voltage (mV) against time (s) for sensor DW-1.8-1.9 with 100 ppm of Cd solution

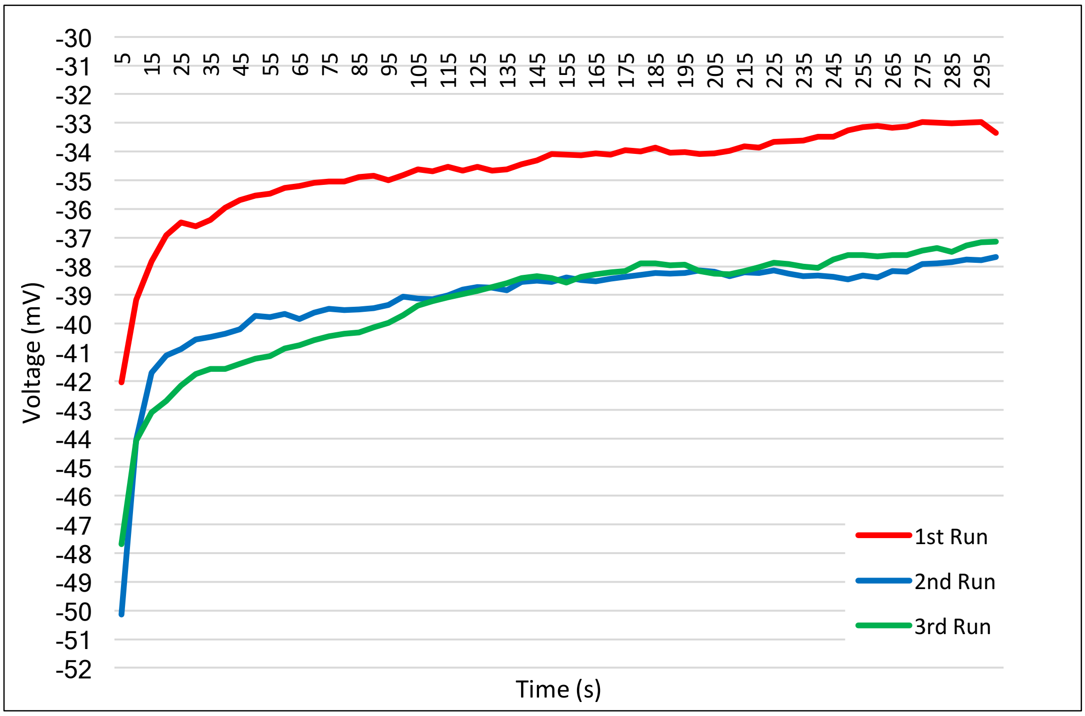

Figure 8 Voltage (mV) against time (s) for sensor W-A-1 with 1 ppm of Cd solution

3.5 Concentration Test

Both PANI and PANI-ZnO thin-film sensors were tested with different concentration of Cd in the range of 1 ppm to 100 ppm for three cycles, and the results obtained were tabulated in Table 3 and Table 4 respectively.

Table 3 Experimental data of PANI thin-film sensor for sensor DW-1.8-1.9 with different concentration of Cd solution

		Voltage (mV)				
Run	**Ref.**	**100 ppm**	**75 ppm**	**50 ppm**	**25 ppm**	**1 ppm**
1	-42.18	8.11	9.30	11.30	12.43	1.80
2	-42.54	11.84	12.21	15.78	15.70	3.45
3	-34.61	2.62	11.46	12.08	14.25	5.95

Table 4 Experimental data of PANI-ZnO composite thin-film sensor for sensor W-A-1 with different concentration of Cd solution

		Voltage (mV)				
Run	**Ref.**	**100 ppm**	**75 ppm**	**50 ppm**	**25 ppm**	**1 ppm**
1	-42.44	53.83	52.61	51.27	49.43	13.33
2	-51.18	63.03	61.05	60.44	58.25	14.56
3	-53.64	65.10	64.46	62.77	61.50	18.16

A bar chart is illustrated for both PANI and PANI-ZnO thin-film sensors, as shown in Figure 9 and Figure 10 respectively. Based on the result in Table 3, it shows that the PANI thin-film sensor was invalid for Cd detection. This is because the voltage reading was erratic fluctuating during the Cd detection of 1 ppm. This means that the adsorption of Cd^{2+} ion cannot contribute to the Cd detection although PANI has high porosity.

As for the PANI-ZnO composite thin film sensor, a valid result was obtained based on Table 4. This is because the voltage reading showed a trend where a higher concentration of Cd corresponding to a higher voltage reading, and vice versa [18]. The mechanism of O_2 chemisorption [28] and Cd^{2+} ions adsorption [18] onto the ZnO surface was discussed.

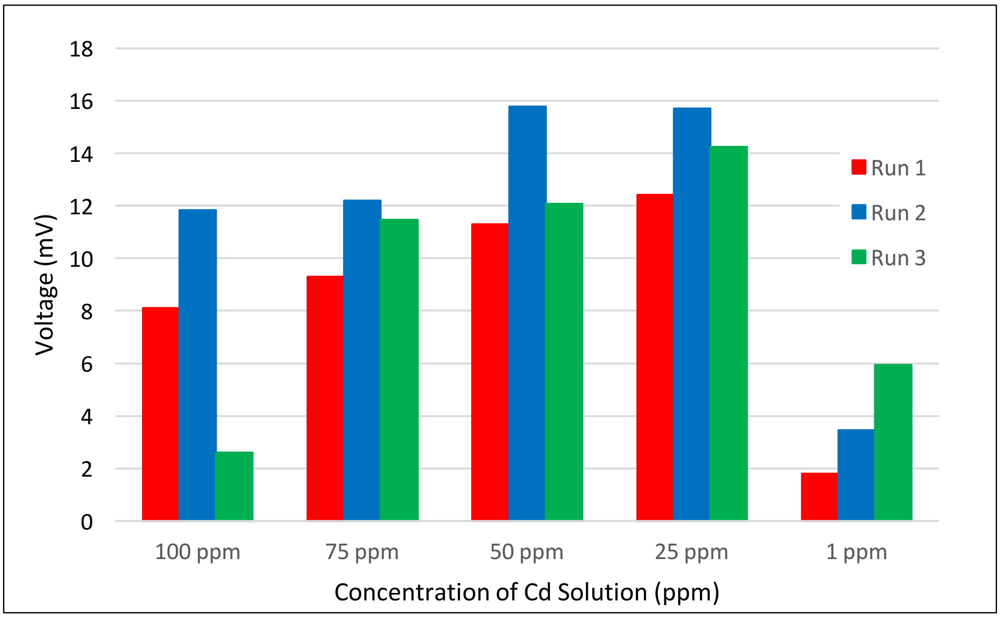

Figure 9 Voltage (mV) against concentration of Cd solution (ppm) for sensor DW-1.8-1.9

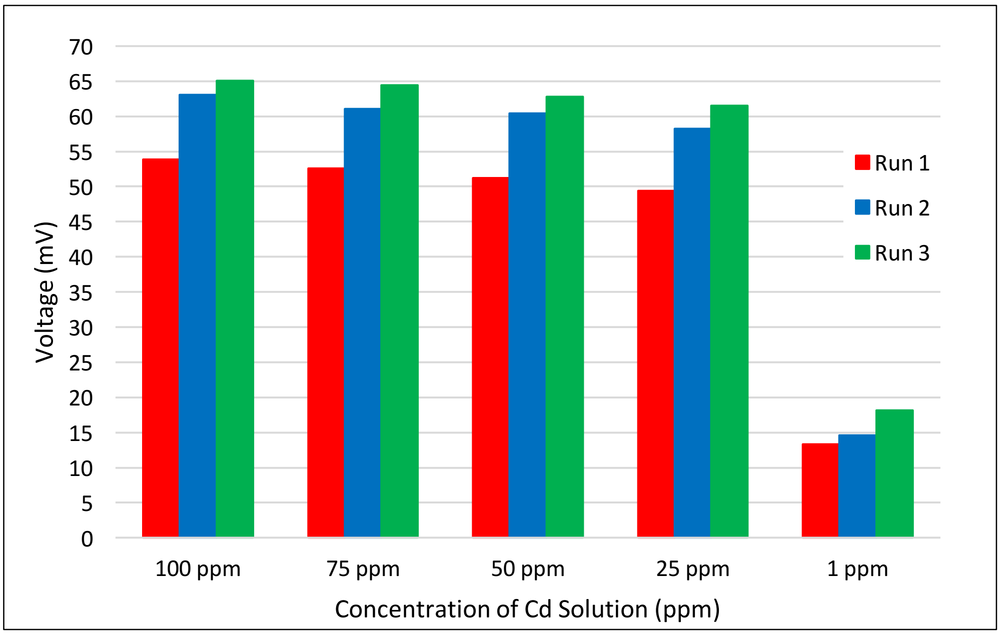

Figure 10 Voltage (mV) against concentration of Cd solution (ppm) for sensor W-A-1

The change in conductivity that leads to an increase in voltage reading is attributed to the mechanism of Cd^{2+} adsorption onto the ZnO surface. When Cd^{2+} ions are adsorbed onto the ZnO surface, they will react with the hydroxyl group that dissociates at the surface, forming $CdOH^{+}$ ions or $Cd(OH)_2$ [18]. Besides, the adsorption of Cd^{2+} ions are more favourable in a high Cd concentration which tends to increase the voltage [18].

The chemisorbed O^{2-} ions tend to affect the result obtained during the first run, which showed a lower voltage reading compare to second and third run. This is because of the exposure of sensor with surrounding air and failure to wash them away by distilled water. Therefore, the Cd^{2+} ions took time to replace the chemisorbed O^{2-} ions by adsorbing themselves onto the ZnO surface, thus causing a lower voltage detected at the early stage. Nevertheless, the sensor is still repeatable as the Cd^{2+} ions that absorbed on the ZnO surface can be washed away by distilled water.

4.0 CONCLUSION

This study presented a PANI-ZnO composite thin-film sensor for Cd detection which was developed and fabricated successfully. The PANI-ZnO composite thin-film sensor gave acceptable results in detecting varying Cd concentration, but PANI thin-film sensor failed. ZnO plays an important role in Cd detection, due to the mechanism of Cd^{2+} ions adsorption onto its surface, with PANI as the agent for immobilization. The PANI-ZnO composite thin-film sensor successfully showed that a higher Cd concentration leads to a higher voltage reading detected, and vice versa. In addition, the fabrication of PANI-ZnO composite thin-film is simpler and had lower costing. Overall, it can be concluded that this type of sensor is user-friendly, portable and responds rapidly.

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