INTERNATIONAL JOURNAL OF INTEGRATED ENGINEERING
ISSN: 2229-838X e-ISSN: 2600-7916

IJIE

Vol. 17 No. 2 (2025) 140-150
https://publisher.uthm.edu.my/ojs/index.php/ijie

Comparison of Polyaniline Performance as A Nitrate EGFET Sensor Layer with Sonication Temperature

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DOI: https://doi.org/10.30880/ijie.2025.17.02.011

Article Info

Received: 14 August 2024
Accepted: 24 June 2025
Available online: 28 July 2025

Keywords

Polyaniline, sonication temperature, nitrate, dip coating, EGFET nitrate sensor

Abstract

The performance of the nitrate Extended Gate Field Effect Transistor (EGFET) sensor has been compared using polyaniline (PANI) with different sonication temperatures, as presented in this paper. PANI was prepared with two sonication temperatures, 28 °C and 38 °C then employed as the nitrate sensor layers of the EGFET that fabricated on Zinc Oxide (ZnO) and Indium Titanium oxide (ITO) substrate using dip coating method with 15 seconds dipping times. The sensing electrode (SE) was characterized to observe the performance of the nitrate EGFET sensor by comparing PANI performance with different sonication temperatures when interacting with nitrate ions, resulting in sensitivity and linearity of the sensor toward nitrate concentrations. PANI/ZnO exhibits excellent nitrate detection capabilities when prepared at room temperature of 28°C, with a sensitivity of 58.20 mV/dec and linearity of 0.9958. To assess the long-term response of SE, drift measurements were conducted by tested sensor with a 20-ppm nitrate concentration for durations ranging from 1 to 10 minutes where ZnO/PANI SE at 28°C show response gradually decreased over time, exhibiting a drift rate of 8.9616 mV/hour over 10 minutes. Consequently, a sonication temperature of 28°C emerges as the ideal parameter for preparing PANI solutions to yield high-performance nitrate sensors.

1. Introduction

Nitrate is one of the chemical elements that cause pollution in ecosystems, especially in water and soil, caused by excessive amounts of chemical levels, especially nitrate, since it can be produced from excess growth fertilizer products or animal waste that is not handled properly and is carried by rainfall from soil to underground water since it is highly soluble and migrates very fast in water or in the atmosphere[1]. These properties of nitrate make it easy to change the concentration in soil porewater and increasing nitrate levels in the water[2]. Excessive nitrate solutions not only cause water pollution but may also cause physiological diseases in humans since the safe permissible limit for nitrate (as NO3-N) in water, as suggested by the Environmental Protection Agency (EPA), is < 10 ppm and < 11 ppm by the World Health Organization (WHO)[3].

Therefore, monitoring nitrate status is necessary to guide the efficient use of fertilizer to optimize plant growth, control farming waste management, and minimize environmental pollution[4]. Nitrate sensors are one of the analytical measurement tools to detect nitrate levels with the properties of high selectivity, fast analysis speed, simple structure, and convenient operation[5][6][7].

There are various materials that have capabilities for sensing nitrate, such as metal oxide, conducting polymers, and copper nanoparticles. Among the materials used for nitrate sensing, polyaniline (PANI) was the most conducting polymer in the field of electrochemical sensing and was considered a metal nanoparticle stabilizer and a good electrocatalyst due to its high chemical stability, high conductivity, low cost, and environmental stability[8][9]. However, its performance can be degraded through the effects of temperature, doping, diffusion, and blending of membranes[8][9]. Sonication is one method used to prepare PANI solution that involves sound vibration by breaking the large particles in solution down to nanoparticle size [10][11] and involves the application of temperature due to absorption of sound waves [11][12]. Applied to a temperature that exceeds the optimum temperature during the sonication process for preparation, PANI may affect the bond of the functional group[13] and its conductivity[14].

There are several methods for sensing nitrate, one of the commonly employed techniques in the sensor industry for nitrate sensing is electrochemical sensing. This falls under the category of chemical sensors, where an electrode serves as a transducer element in the presence of an analyte[15]. Electrochemical sensors come in various types, including potentiometric, amperometric, and conductometric [4][15] sensors which offer high usability, making them suitable for applications such as portable instruments that feature a straightforward setup, ease of sample preparation, and enhanced sensitivity or selectivity. Among the electrochemical sensors, the potentiometric type is commonly used for FET (Field Effect Transistor) applications since its reaction leads to the generation of an electrostatic potential (V)[4]. A specific example of a potentiometric sensor is the ion-sensitive field-effect transistor (ISFET). This device functions as a MOS (Metal-Oxide-Semiconductor) transistor, where the gate connection is separate from the device itself. It is linked to a reference electrode that is in contact with the solution. An insulating membrane replaces the traditional gate oxide in this setup[16].

However, this sensor suffers from several disadvantages, including unstable device performance and poor sensitivity [4][17]. Therefore, extended-gate field effect transistors (EGFET) serve as a solution to overcome the disadvantages of ISFETs [4][18]. The EGFET structure effectively isolates the FET from the surrounding chemical environment since the sensing electrode is deposited at the end of the signal line, extending from the FET gate electrode. The EGFET design offers advantages such as ease of characterization through simple connections at low cost, no light interference, making it less sensitive to light and temperature, and long-term stability under various environmental conditions[19][20].

This research focuses on determining the effects of sonication temperature during the preparation process of PANI solutions. The solutions were set up at temperatures of 28°C (within the range of room temperature) and 38°C, and the analysis was conducted using EGFET measurements. A temperature 38°C been selected due to the effect of temperature over 35°C reported by Hongling et al. [21] that at temperatures over 35°C, PANI becomes overoxidization and its electrochemical activity decreases. This occurs because it becomes difficult for PANI at an early stage to transition from the emeraldine form and the pernigraniline form to the leucoemeraldine form and back to the emeraldine form at higher temperatures, which also affects its ability to function as a conductive polymer. The study involved observing three structures: SE, ZnO, PANI, and PANI deposited on top of ZnO, all of which were coated onto an ITO substrate. ZnO was included in this research as the sensing material, however, it exhibits low stability. To overcome this problem, PANI was applied as a polymer layer to enhance the stability and ion selectivity of the sensor [22]. PANI also served as a sensing material due to its stability and high conductivity, making it suitable for detecting nitrate concentrations. EGFET measurements were utilized to identify the sensitivity, linearity, and drift rate of the sensor based on the resulting response, which manifests as a change in the threshold voltage when the SE reacts with the analyte.

2. Research Method

2.1 Preparation of PANI Solution

PANI (emeraldine base) powder, ([C6H4NH]2[C6H4N]2) with 0.02 mol/L molarity was used as polymer membrane material for this experiment. Two samples of 30 mL PANI solution were prepared in two laboratory bottles by mixing 0.6 g of PANI powder with 29.6 mL of dimethylformamide (DMF). The mixtures were stirred at 350 rpm for 20 minutes using a magnetic stirrer to achieve proper dilution. Subsequently, the two sample mixtures were sonicated at two distinct temperature levels which were 28°C and 38°C for 30 minutes using ultrasonic bath. Lastly, the mixtures were stirred again at 350 rpm for 1 hour. All the preparation process was shown in Fig. 1 and the complete parameters for preparing the solution were shown in Table 1.

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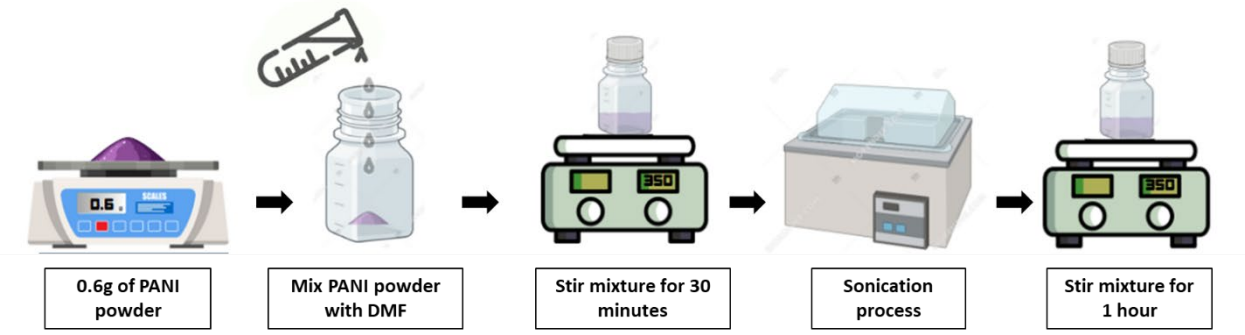

Fig. 1 *Preparation PANI solution*

Table 1 *Sample and parameter for solution preparation*

Sample	Material	Mass (g)	DMF (mL)	Stirring process		Temperature during sonication (°C)
				1st	2nd	
1	PANI	0.6	29.4	350 rpm for 30 minutes	350 rpm for 1 hour	28
2						38

2.2 Dip Coating Method

20 mL of the two prepared samples of PANI solution have been placed in two 100 mL beakers for the dip coating method. Fig. 2 shows the overall step for the dip coating method, where two different structures of sensing electrodes (SE), bare Indium Tin Oxide (ITO) substrate and ITO coated with ZnO as sensing material by the spin coating method, are clamped onto the dip coater arm, and each electrode is coated with two conditions of PANI solution, which are differentiated by sonication temperature. The parameters of the dip coater have been set up with 5 mm/s of withdrawal speed, 20 mm/s of down speed, and a 15 s dipping times. Then, the SE be dried on hot plate for 10 minutes with temperature, 100°C. Fig. 3 shows the cross-sectional schematic diagram of a SE that has been coated with PANI and has been labelled as PANI/ZnO and PANI. The impact of 28°C and 38°C temperature to the properties of PANI been observed throughout the coloration of the PANI on SE using Olympus optical microscope.

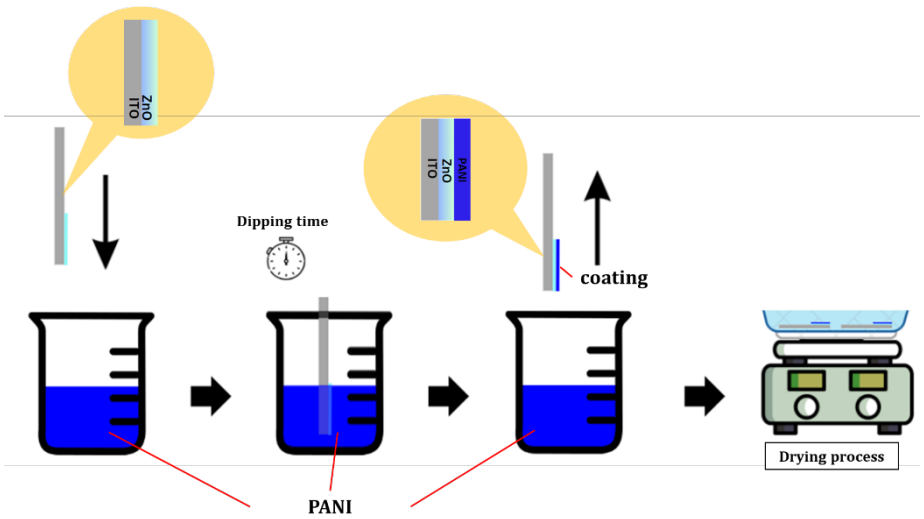

Fig. 2 *Dip coating method*

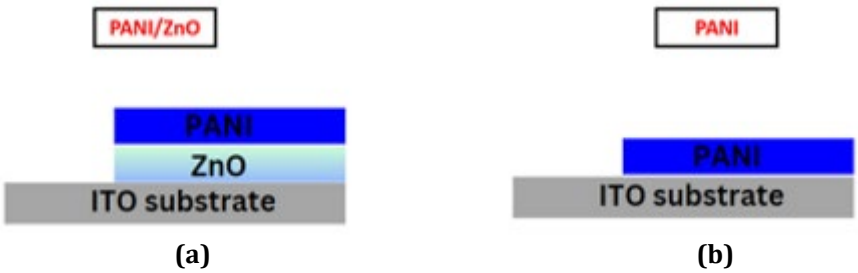

Fig. 3 *Cross-sectional view of (a) PANI and ZnO on ITO substrate; (b) PANI on ITO substrate*

2.3 EGFET Measurement

The sensitivity and linearity of the SE have been measured through an EGFET measurement setup with a Keysight B1500A Semiconductor Device Analyzer, as shown in Fig. 4. Commercialized MOSFET BS170 was used where the gate terminal was connected to SE as an extended gate immersed in nitrate solutions with concentration at range 10 to 50 ppm. The silver/silver chloride (Ag/AgCl) reference electrode (RE) was connected to the SMU 1 port of the B1500A, while SMU 2 was linked to the drain terminal of the BS170, and SMU 3 was connected to its source terminal. From this connection, the transfer and output characteristics of the sensor are displayed as a current-voltage graph (Id- Vref) in B1500A, and each curve measurement at 100 µA drain current of each nitrate concentration has been recorded for the plotted graph output gate voltage (voltage reference, Vref) against nitrate solutions. Sensor sensitivity and linearity have been calculated through the slope of the graph, Vref against nitrate concentration.

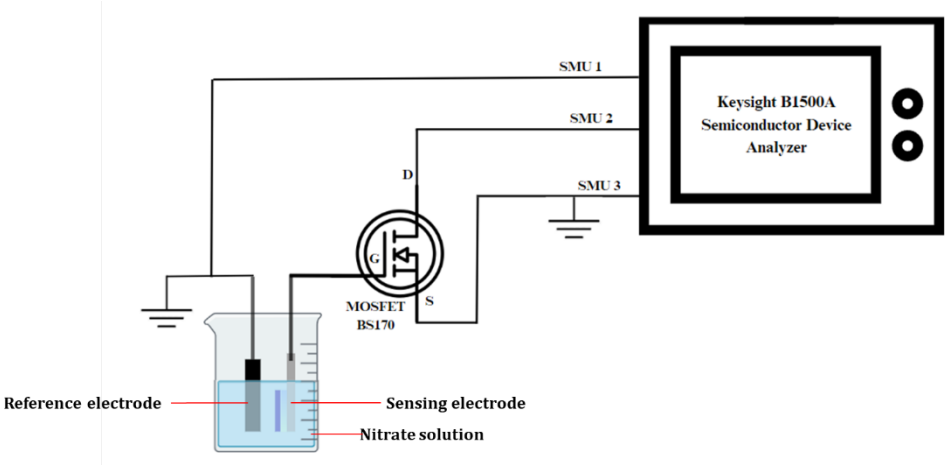

Fig. 4 *Sensitivity and linearity measurement setup*

Fig. 5 shows the experimental setup for sensor drift measurement where the commercialized MOSFET BS170 gate terminal is connected to the SE while the drain and source terminals are connected to the readout integrated circuit (ROIC). For this measurement, SE and RE (connected through the ground of ROIC) were immersed in 20 ppm of nitrate solution. 5 V has been supplied by the power supply, and the output voltage has been readout by Data Acquisition (DAQ) National Instrument through a graph of output voltage against time. The drift measurement has been recorded at 1, 3, 5, 8, and 10 minutes.

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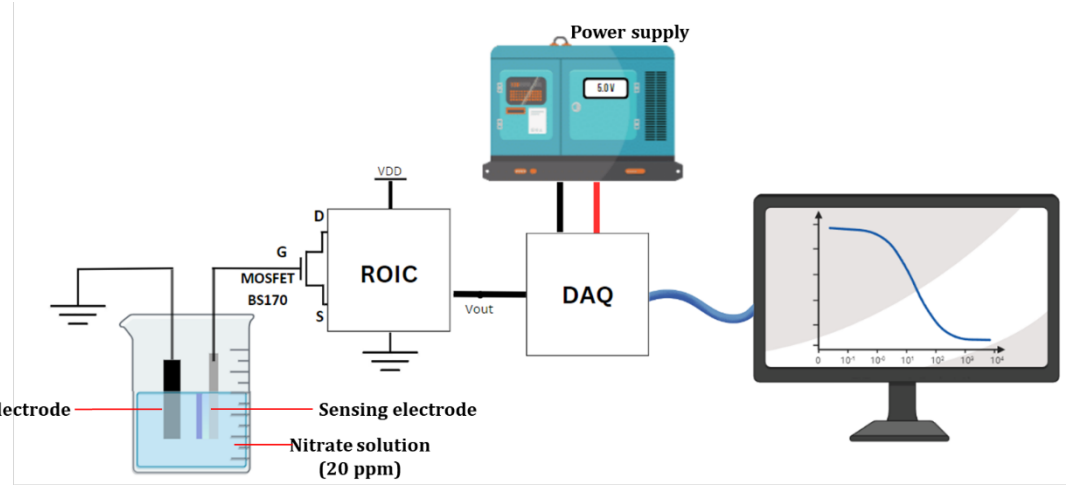
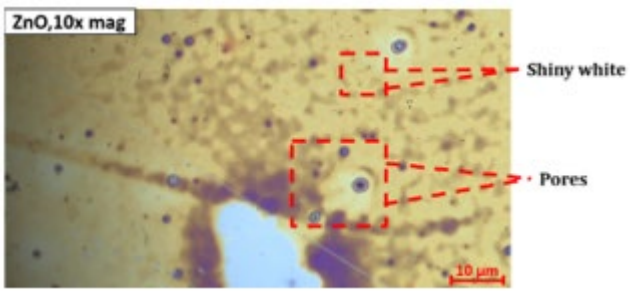


Fig. 5 *Drift measurement setup*

3. Results and Discussion

3.1 Microscopic Image

The microscopic image of the SE at X10 magnification is presented in Fig. 6, illustrating the variations in the SE structures resulting from different sonication temperatures applied during the PANI solution preparation process. The image reveals distinct differences in the coloration of the PANI layer, which is directly influenced by the temperature conditions. In Fig. 6a, the microscopic image of ZnO displays the presence of pores and a white, shiny surface. When the SE structure is coated with a PANI solution prepared at a temperature of 28°C (shown in Fig. 6b and 6c), the resulting colour is a light shade of purple, consistent with the inherent colour of the PANI solution. Additionally, the application of temperature to the PANI solution contributes to alterations in the coloration. This can be attributed to temperature-induced vibrations causing deformations in the C-H groups of the PANI quinonoid ring [13]. When changing the temperature during solution preparation, PANI molecules start to move differently, and this movement can cause the C-H groups in the PANI molecule to change shape, especially within the quinonoid ring. These structural changes in the molecule are responsible for the alterations in color that you observe in the solution [23]. Different molecular structures can absorb and reflect light differently, leading to changes in the color of the solution as a result of temperature changes. Consequently, the SE colours as shown in Fig. 6d and 6e, shifts towards a reduced intensity of purple (dark purple) when prepared with PANI under a sonication temperature of 38°C.



(a)

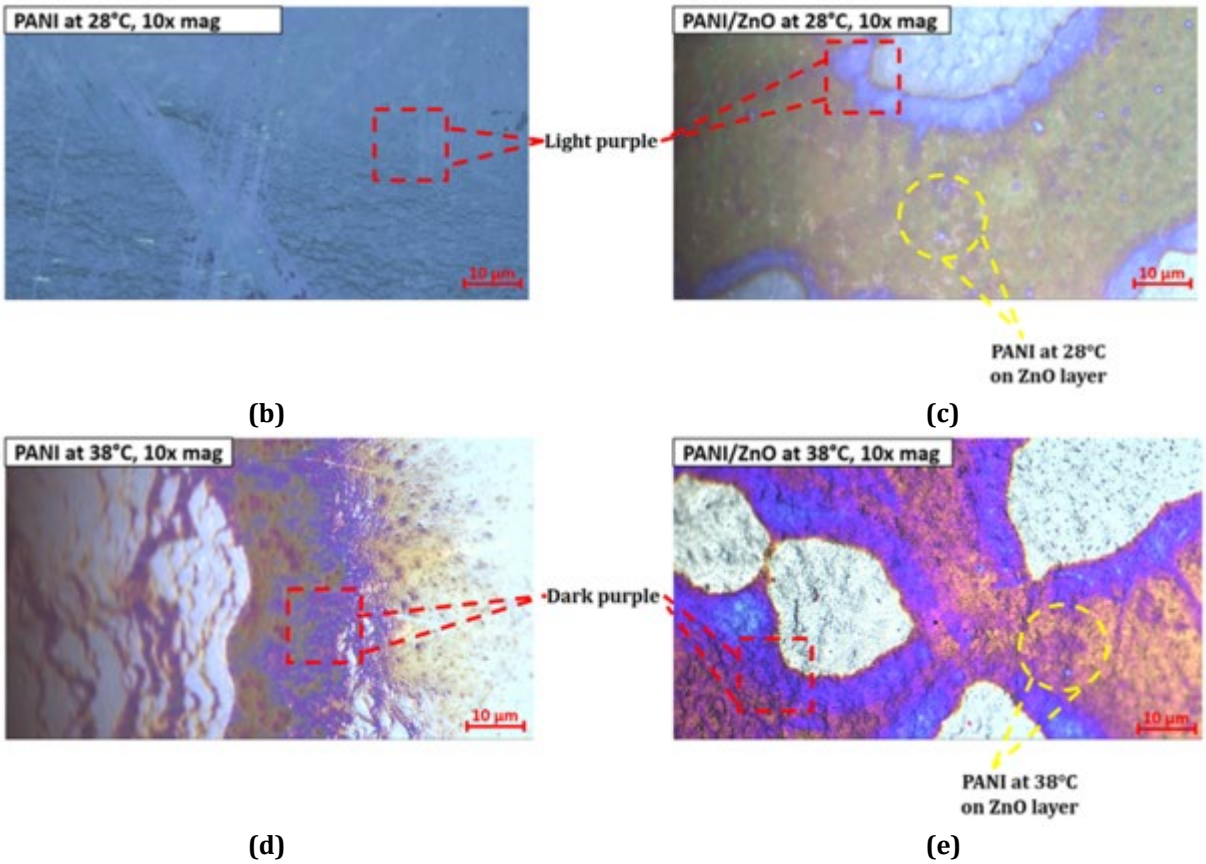


Fig. 6 *Microscopic image of (a) ZnO (b) PANI at 28 °C (c) PANI/ZnO at 28 °C (d) PANI at 38 °C (e) PANI/ZnO at 38 °C*

3.2 Sensitivity and Linearity

The transfer characteristic, sensitivity, and linearity of the sensing element (SE) were determined by analysing the current-voltage characteristics of the EGFET nitrate sensor. The output voltage corresponding to nitrate concentrations ranging from 10 to 50 ppm was recorded from the ID-Vref graph at a current value (IDS) of 100 µA using the Keysight B1500A Semiconductor Device Analyzer. This recorded data was then plotted to establish the relationship between Vref and nitrate concentration, allowing us to determine sensitivity and linearity based on the slope of the graph. Fig. 7 show the correlation between Vref and nitrate concentrations for different SE structures of the EGFET nitrate sensor. For ZnO, the sensitivity and linearity are 58.20 mV/dec and 0.9755, respectively (Fig. 7a). These results indicate that the SE utilizing ZnO approaches the optimal sensitivity for the nitrate sensor [24], with linearity close to 1.

For SE that been applied with two different PANI solution, show the varies result in sensitivity and linearity for nitrate sensor. SE composed of an ITO substrate and PANI (as the sensing layer) at 28°C yields sensitivity and linearity values of 82.70 mV/dec and 0.9781, respectively. Conversely, utilizing PANI at a temperature of 38°C results in a sensitivity of 145.30 mV/dec and linearity of 0.8285, as demonstrated in Fig.s 7b and 7c. Fig.s 7d and 7e show the Vref versus nitrate concentration graph for SEs consisting of PANI (at sonication temperatures of 28°C and 38°C) as a polymer layer on top of ZnO, PANI/ZnO. At a temperature of 28°C, the sensitivity and linearity are 58.20 mV/dec and 0.9958, respectively, while at 38°C, the sensitivity and linearity values are 98.90 mV/dec and 0.9874. This comparative analysis of different PANI conditions underscores the effect of temperature on the performance of PANI both as a sensing layer and a polymer layer for nitrate sensing.

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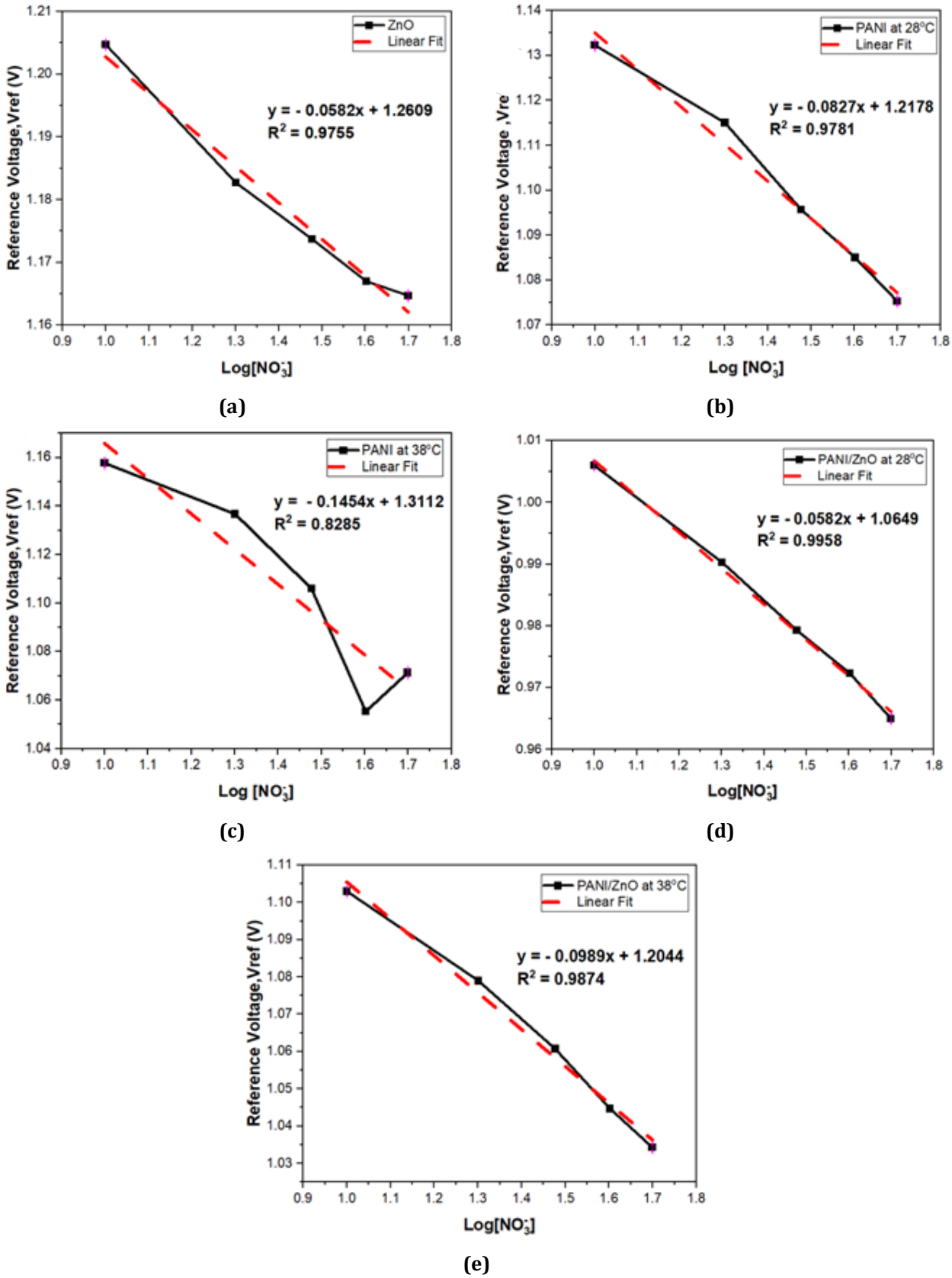

Fig. 7 *References voltage versus nitrate concentration of (a) ZnO (b) PANI at 28 °C (c) PANI at 38 °C (d) PANI/ZnO at 28 °C and (e) PANI/ZnO at 38 °C*

Table 2 *Summarize the sensitivity and linearity of ZnO and PANI under temperatures 28 °C and 38 °C on the top of ZnO and ITO subsrate*

Type of SE	ZnO	PANI at 28 °C	PANI at 38 °C	PANI/ZnO at 28 °C	PANI/ZnO at 38 °C
Sensitivity (mV/dec)	58.20	82.70	145.30	58.20	98.90
Linearity (R^2)	0.9755	0.9781	0.8285	0.9958	0.9874

The summary of sensitivity and linearity results for SEs involving ZnO, PANI, and PANI/ZnO (at sonication temperatures of 28°C and 38°C) has been organized in Table 2. The utilization of PANI atop ITO as a polymer layer highlights PANI's potential in nitrate sensing. However, the sensitivity values for both samples fall considerably short of approaching the optimal sensitivity required for a nitrate sensor. In the case of SEs with PANI as the polymer layer, PANI/ZnO at 28°C demonstrates an approach to the optimal sensitivity for nitrate sensing according to Nernstian response limit, accompanied by the highest linearity, which closely approximates a value of 1. For instance, Carol et al. [25] conducted a study on a printed potentiometric nitrate sensor using PVB that had been sonicated for 30 minutes in an ice bath (at a temperature of 10 to 15 °C). The sensor exhibited an optimal Nernstian response with a sensitivity of 48.0 mV/dec across the nitrate concentration range of 0.62 to 6200 ppm in solution. Conversely, PANI/ZnO at 38°C exhibits elevated sensitivity but fails to reach the optimal sensitivity level for nitrate sensing. Additionally, its linearity is slightly lower compared to that at 28°C but still maintains proximity to a value of 1. For SEs incorporating PANI at 38°C, the sensitivity outperforms those with PANI at 28°C. This difference in sensitivity is attributed to the deformation of the C-H group [13] of PANI at 38°C which enhances the interactions between the C-H group and nitrate ions, resulting in high in sensitivity [4].

3.3 Drift

The drift characteristics represent the stability of sensor in constantly sensing the nitrate concentration and been evaluated through the drift rate. This measurement has focused on sensing 20 ppm nitrate solution at times 1, 3, 5, 8, and 10 minutes. The change Vref (ΔVref) is equal to Vref(t) minus Vref(0) and drift property is compared by calculating the drift rate (ΔVref/h) at high time setup for drift [26].

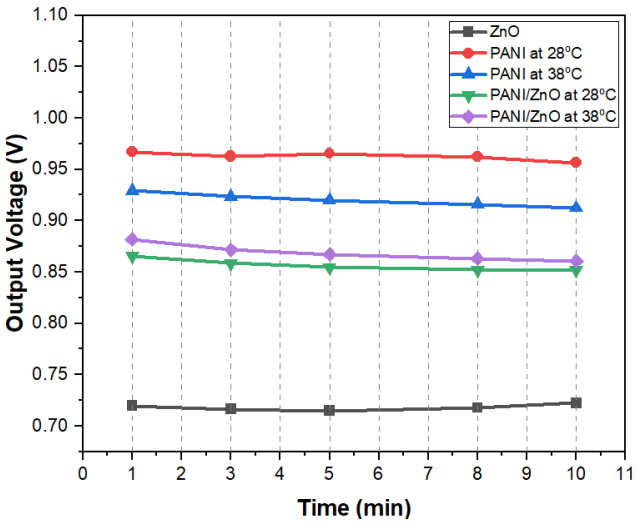

Fig. 8 *Drift characteristic of ZnO, PANI and PANI/ZnO at 20 ppm*

Table 3 *Drift rate of ZnO and PANI under temperature 28 °C and 38 °C on the top of ZnO and ITO substrate*

Type of SE	ZnO	PANI at 28 °C	PANI at 38 °C	PANI/ZnO at 28 °C	PANI/ZnO at 38 °C
Drift rate (mV/h)	1.5240	6.4260	9.9598	8.3616	12.6780

Table 3 show SE drift rate of ZnO, PANI at 28 °C and 38 °C and PANI/ZnO at at 28 °C and 38 °C were around 1.5240 mV/h, 6.4260 mV/h, 9.9598 mV/h, 8.3616 mV/h and 12.6780 mV/h. SE ZnO and PANI at 28 °C result in lowest drift rate, however the output voltage for time implement in drift measurement (shown in Fig. 8) show

that unstable reading for both SE. For example, Kamarozaman et al. [26] fabricated PANI-TiO2 composition resulting in long stability response under pH 4, 7, and 10 with sensitivity and linearity of 66.1 mV/pH. Conversely, the SE involving PANI/ZnO at 28°C exhibited a gradual decrease in output voltage over the 1 to 10-minute time span, characterized by a drift rate of 8.9616 mV/h. Notably, this trend indicated a relatively stable decreasing reading compared to the other SEs. On the other hand, for the SE utilizing PANI at 38°C, a substantial reduction in output voltage occurred during the initial 1 to 3 minutes of the drift measurement. This behavior might be attributed to the impact of high temperature on the conductivity of PANI, resulting in a decreasing conductivity level when subjected to elevated temperatures [14] due to a slow loss of the structure of the solvent caused by the degradation condition [27].

4. Conclusion

PANI with sonication temperature, 28 °C is successfully fabricated on the top of ITO substrate and ZnO using dip coating method. From the result, solution PANI prepared at sonication temperature 28 °C (PANI/ZnO at 28 °C) result in sensitivity at approaching optimal sensitivity of nitrate sensor and has higher linearity value that close to 1. It also result in low drift rate which represent the strengthen of the structure to rapid reaction with analyte in long term response. In conclusion, the performance of PANI as EGFET sensor-based nitrate sensor layer performance better on 28 °C with improved sensitivity, near-optimal linearity, and enhanced stability. The successful fabrication of PANI at a sonication temperature of 28°C onto both ITO substrate and ZnO substrates through the dip coating method underscores its potential as a highly effective nitrate sensing layer.

Acknowledgement

The research project is funded by the Ministry of Higher Education (MOHE) under the Fundamental Research Grant Scheme (FRGS)(FRGS/1/2022/TK07/UITM/02/38) and supported by the College of Engineering, Universiti Teknologi MARA.

Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Nor Farhana Abdullah, Robaiah Mamat, Muhammad Izzat Alif Muslan;* ***data collection:*** *Nor Farhana Abdullah, Robaiah Mamat;* ***analysis and interpretation of results:*** *Nor Farhana Abdullah;* ***draft manuscript preparation:*** *Nor Farhana Abdullah, Azrif Manut;* ***project supervision:*** *Wan Fazlida Hanim Abdullah. All authors reviewed the results and approved the final version of the manuscript.*

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