Jurnal
Teknologi

Full Paper

INTEGRATED-ALL SOLID ELECTRODES pH SENSOR PERFORMANCE DEPENDENCY ON SILVER/SILVER CHLORIDE ELECTRODE THICKNESS

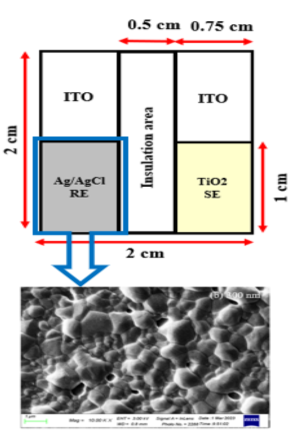
Shaiful Bakhtiar Hashim[a,b,c], Norhidayatul Hikmee Mahzan[a,b,c], Aimi Bazilah Rosli[c], Zurita Zulkifli[b*], Nurbaya Zainal[b,c], Sukreen Hana Herman[c,d]

[a]Faculty of Electrical Engineering, Universiti Teknologi MARA Cawangan Terengganu Kampus Dungun, 23000 Dungun, Terengganu, Malaysia
[b]Nano-Electronic Center (NET), Faculty of Electrical Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia
[c]Integrated Sensors Research Group, School of Electrical Engineering, College of Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia
[d]Microwave Research Institute, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia

Article history
Received
1 April 2024
Received in revised form
28 September 2024
Accepted
21 October 2024
Published Online
26 June 2025

*Corresponding author
zurita101@uitm.edu.my

Graphical abstract



Abstract

Environmental monitoring, biomedical research, and food processing require precise pH measurements; hence pH sensors are important. The glass-based reference electrode (RE) is the most common pH sensor but the fragility of the glass-based RE prevents it from being miniaturized. Thus, an integrated-all solid electrode (IASE) was fabricated where the Ag layer was thermally evaporated, followed by a chlorination process to form an Ag/AgCl RE, and a titanium dioxide (TiO_2) thin film was used as the sensing electrode (SE) to form the integrated-all solid electrode (IASE). The influence of silver/silver chloride (Ag/AgCl) thin film electrode thickness (60, 120, 180, 240, and 300 nm) on the electrochemical-based pH sensor was studied. The IASE was applied as the extended-gate of field effect transistor (EGFET). The pH sensor sensitivity and linearity were evaluated for the IASE performance. As a result, the thicker the Ag/AgCl layer the higher the sensitivity. The 300 nm Ag/AgCl thickness showed promising sensitivity and linearity with 67.8 mV/pH and 0.9846, respectively.

Keywords: Silver/silver chloride, solid reference electrode, sensitivity, linearity, pH measurement

Abstrak

Pengawasan alam sekitar, penyelidikan biomedikal, dan pemprosesan makanan memerlukan pengukuran pH yang tepat; oleh itu sensor pH adalah penting. Glass-based pH electrode adalah sensor pH yang paling biasa tetapi kerapuhan pH electrode berasaskan kaca menghalang ia daripada menjadi miniatur untuk kegunaan terutama dalam bidang perubatan. Pengaruh ketebalan Silver/Silver Chloride (Ag/ AgCl) electrode filem tipis (60, 120, 180, 240 dan 300 nm) pada sensor pH berasaskan elektrokimia telah dikaji. Lapisan Ag dipanaskan secara termal, diikuti oleh proses klorinasi untuk membentuk Ag/AgCl RE, dan titanium dioxide (TiO_2) digunakan sebagai sensing electrode (SE) untuk membentuk Integrated-all solid electrode (IASE). IASE digunakan sebagai extended-gate field effect transistor (EGFET). Sensitiviti sensor dan lineariti dinilai untuk prestasi IASE. Hasilnya, lapisan Ag/AgCl yang lebih tebal telah meningkatkan kepekaan. Ketebalan 300 nm Ag/AgCl menunjukkan sensitiviti dan lineariti yang memberikan keberkesanan dengan 67.8 mV pH dan 0.9846, masing-masing.

Kata kunci: Silver/silver Chloride, solid reference electrode, sensitivity, kelinearan, pengukuran pH

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1.0 INTRODUCTION

pH sensors play a vital role in various industries, including environmental monitoring, biomedical research, and food production, where precise and reliable pH measurements are essential [1]–[6]. H^+ and OH^- water molecules in most liquids interact with other molecules, changing the solution's acidity and basicity and affecting its characteristics. This interaction emphasizes pH measurement's usefulness in numerous fields.

The most widely used technique to measure pH is the glass-based pH electrode. This electrode is highly regarded for its reliability, performance, long-term stability, selectivity, and low detection limit [7]. However, there are several challenges with the current pH sensing methods. One of the most significant is the fragility of the commonly used glass-based pH reference electrode, which can limit its use and prevent it from being miniaturized for specific applications, such as medical use [2], [8].

Silver/silver chloride (Ag/AgCl) reference electrodes are a type of electrode used for pH sensing based on Ag/AgCl redox couple and several advantages, including their stability, accuracy, and wide range of use [9]–[11]. Ag/AgCl can be fabricated in the form of thin films, and this opens up miniaturization possibilities for pH sensors on top of further improved compatibility with electronic systems. In this paper, the performance of the integrated-all solid electrodes sensor was investigated based on the influences of Ag/AgCl thickness fabricated by the thermal evaporation method.

2.0 METHODOLOGY

Figure 1 shows the layout configuration of integrated-all solid electrodes (IASE). IASE was created by combining the sensing electrode (SE) and the reference electrode (RE) on a single ITO glass substrate. To establish the electrical isolation between the SE and RE, the etching process was conducted using hydrochloric acid (HCl) and zinc powder in the isolation region as shown in Figure 1. The surface areas of the SE and RE regions were 0.75 cm^2 each and were separated by an insulating area of 1 cm^2. The copper wire was attached to both uncoated conductive ITO areas. To enhance contact strength and prevent current leakage during the measurement process, copper and Kapton tape were used to encapsulate the Cu wire.

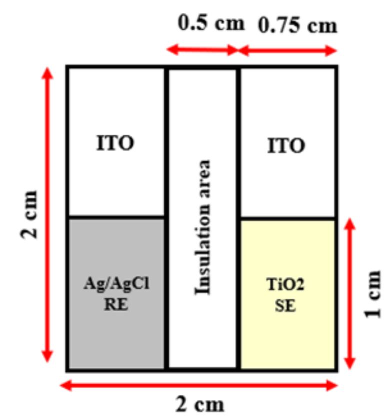

Figure 1 Layout configuration of IASE

In the preparation of IASE fabrication, the ITO substrate was through a rigorous cleaning procedure using Hwashin Technology Powersonic 405 for ultrasonic cleaning. The cleaning commenced by immersing the substrate in ethanol and deionized water, with each immersion lasting for 10 minutes. Subsequently, the substrate was carefully dried using inert nitrogen gas to ensure its cleanliness and remove residual moisture.

For the SE area, titanium dioxide (TiO_2) metal oxide material was used. The Laurell Model WS-650MZ-8NPP/LITE equipment was used to accomplish the sol-gel spin-coating process, which led to the deposition of the TiO_2 thin film. This extensively utilized method is widely recognized for its capacity to generate a uniform and homogeneous thin film [12]–[16]. The synthesis of TiO_2 involved the preparation and combination of two separate mixtures, namely Mixture A and Mixture B. Mixture A comprised titanium (IV) isopropoxide (TTIP) (Ti[OCH(CH3)2]4, Sigma-Aldrich, 97%) as the precursor, glacial acetic acid (GAA) (CH3COOH, Friedemann Schmidt, 99.8%), and absolute ethanol (C2H5OH, SYSTERM, 99.8%). Conversely, Mixture B contained deionized water (H2O, Milli-Q Advantage

A10), absolute ethanol, and Triton X-100 (C34H62O11, R&M Chemicals, 98%) as the surfactant. Each mixture was individually prepared and subjected to a stirring process for 1 hour. A further hour of stirring at room temperature continued after both separate mixtures had been merged.

The TiO_2 thin film deposition starts with 10 drops of the prepared TiO_2 mixture deposited onto the ITO substrate on the spin coater chuck. The substrate was then spun at 500 rpm for 10 seconds before being increased to 3000 rpm for one minute. The solution was able to disperse uniformly throughout the substrate surface due to the high-speed spinning, producing a uniformly thin layer. To remove any remaining solvents, like water and ethanol, the thin film was dried for 10 minutes at 100 °C. Then, a single layer of thin TiO_2 film, about 23 nm thick, was produced after annealing at 400 °C for 15 minutes.

Following the deposition of an Ag layer via thermal evaporation (TE), a 5-second chlorination process was conducted using FeCl3 to produce an Ag/AgCl RE. Table 1 shows the varied Ag/AgCl thickness list in this experiment. The thickness was controlled during the TE process.

The deposited samples were characterized in terms of physical and sensing properties. By utilizing an EGFET pH sensor setup, the sensitivity and linearity were assessed.

The surface morphology of the deposited Ag/AgCl was examined for the physical properties using field emission scanning electron microscopy (FESEM) at 10k magnification. The Ag/AgCl thin film that was deposited was examined for crystallinity using X-ray diffraction (XRD).

Table 1 List of varied Ag/AgCl thickness

Ag/AgCl Thickness (nm)
60
120
180
240
300

Various pH buffer solutions were employed to explore the sensor's response capabilities. The IASE was immersed in different pH buffer solutions, including those with 2, 4, 7, 10, and 12 pH values. The transfer characteristic (drain current, I_D versus reference voltage, V_{REF}) and the output characteristic (drain current, I_D versus drain-source voltage, V_{DS}) were obtained from these measurements. During transfer characteristic measurement, the V_D was set at 100 mV and V_{REF} sweeps from 0 to 3 V. To assess the pH sensitivity and linearity performance of the fabricated IASE, a graph depicting the relationship between the drain current and the reference voltage was evaluated. The output voltage was extracted from the I_D versus V_{REF} curve as the obtained I_D of 100 µA corresponding to an applied reference voltage.

Linearity in pH sensors shows how well the sensor's output matches changes in pH, with a straight line indicating a strong relationship. A sensor with high linearity ensures the output signal changes consistently with each pH unit across the range. In an ideal sensor, this would result in a constant slope when plotting output versus pH. Linearity is measured using the coefficient of determination (R^2), where values closer to 1 indicate a more accurate linear relationship.

3.0 RESULTS AND DISCUSSION

The surface morphology of the Ag/AgCl thin film was characterized between the lowest and the highest thickness of the thin film as shown in Figure 2 (a) and (b), respectively. As a result, the Ag/AgCl thin films with 60 nm thickness exhibited a granular morphology characterized by a notable separation between the granules. In contrast, samples with a thickness of 300 nm, demonstrated a dense morphology structure, with each granule closely linked to one another. The chlorination process may result in the AgCl grain being etched away at lower thicknesses due to lower Ag deposition. At higher thicknesses, the chlorination can induce significant alterations in the surface morphology of Ag/AgCl thin films. The treatment may cause the merging and coalescence of adjacent AgCl grains, resulting in a more continuous and interconnected morphology. The results of this SEM at a thickness of 300 nm exhibit similarities to the findings of T. Rahman *et al.* [17].

The XRD analysis was used to determine the chemical composition and crystal structure between Ag^+ and Cl^-. The XRD scan was conducted over a range of 20° to 80°. As seen in Figure 3, the XRD spectrum is displayed at a various thickness. Based on the XRD analysis, it shows clearly three notable peaks at 2θ values of approximately 38.27, 44.47, and 77.53, corresponding to the (111), (200), and (420) orientations, respectively. The diffraction peaks observed in the sample exhibit a high degree of sharpness, suggesting a well-defined crystalline structure. Furthermore, no additional impurity peaks are detected, indicating that the sample possesses a remarkable level of quality and purity [18]. These orientations indicate the presence of Ag for (111) and (200), while the (420) orientation indicates AgCl [19]. The XRD peaks demonstrate that increasing the thickness results in higher intensity of the diffraction peaks. Specifically, at a thickness of 60 nm, a broad peak around 35.36 can be attributed to the (200) orientation of AgCl [20]. However, due to the higher intensity of the Ag orientation, the AgCl peak orientation is not visible in Figure 3. The intensity of XRD peaks reflects the amount and orientation of crystalline material in the thin film.

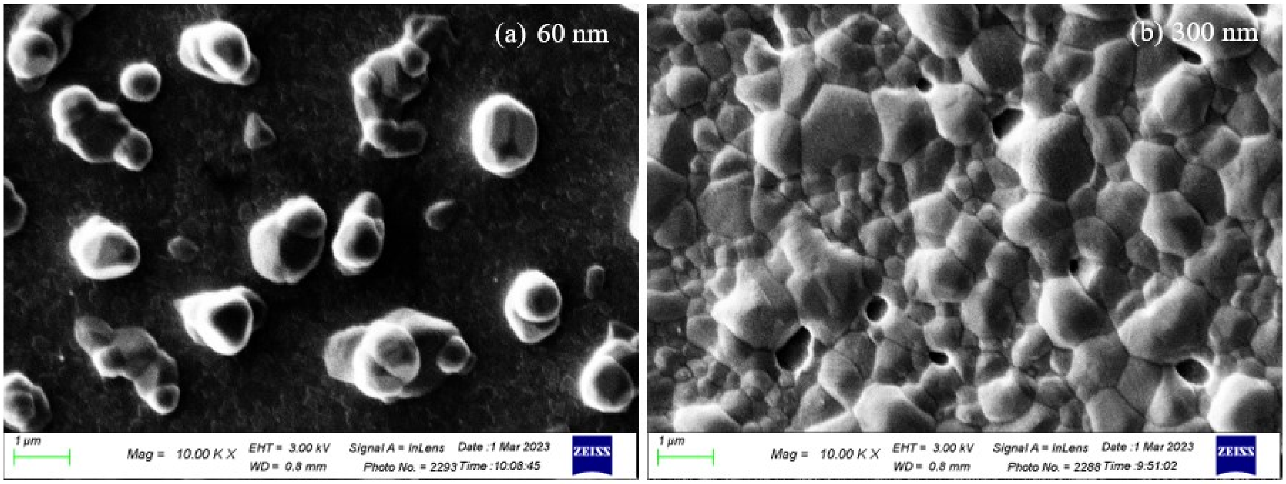

Figure 2 FESEM images of Ag/AgCl thin film deposited at different thicknesses (a) 60 nm and (b) 300 nm

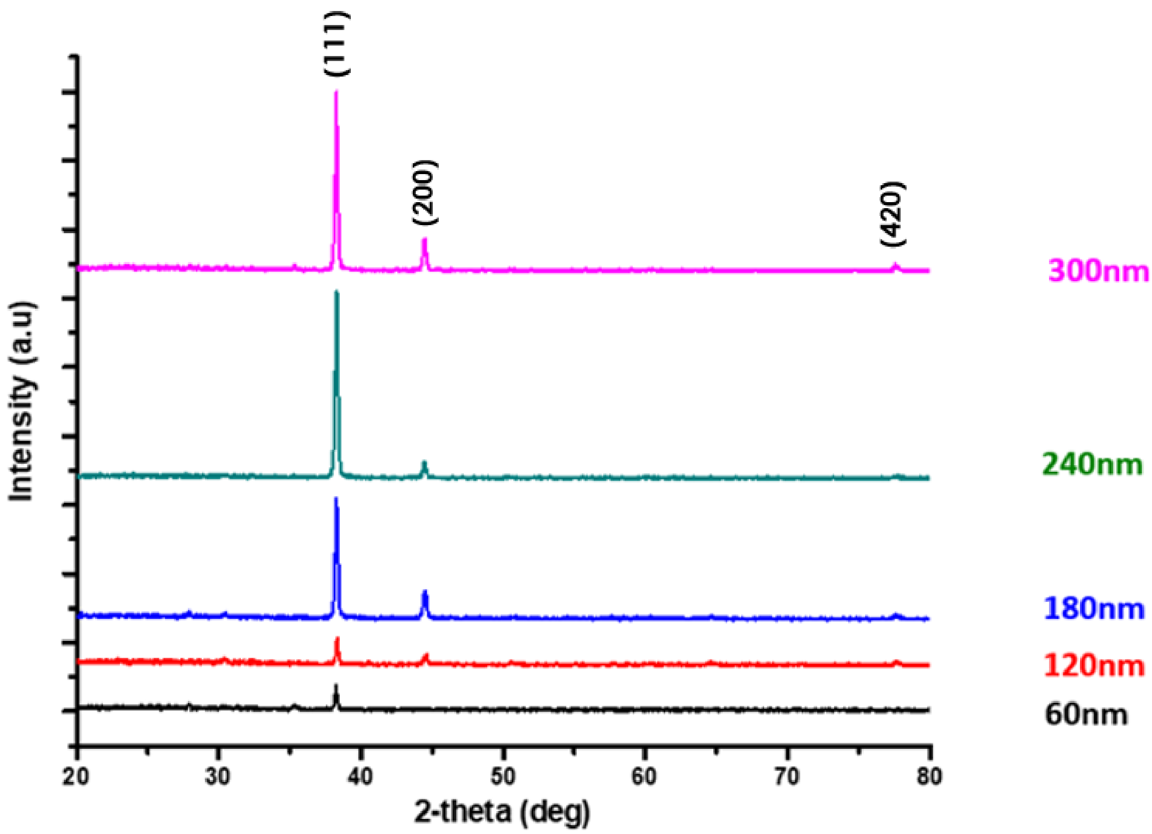

Figure 3 XRD spectrum of the fabricated Ag/AgCl at different thickness

Figures 4 until 6 show the EGFET transfer characteristic and output voltage versus pH for the 60, 180, and 300 nm Ag/AgCl RE, respectively. The transfer characteristics were taken with the IASE immersed in pH buffer solutions (pH2 – 12) at room temperature condition. It can be seen that the threshold voltage for each measurement in each pH buffer is shifted to the right as the pH value increases. The output voltage versus pH graph was plotted by taking the reference voltage value at 100 µA drain current from the transfer characteristic for each of the pH values. The sensitivity is then taken from the graph slope, and linearity is the line linear regression.

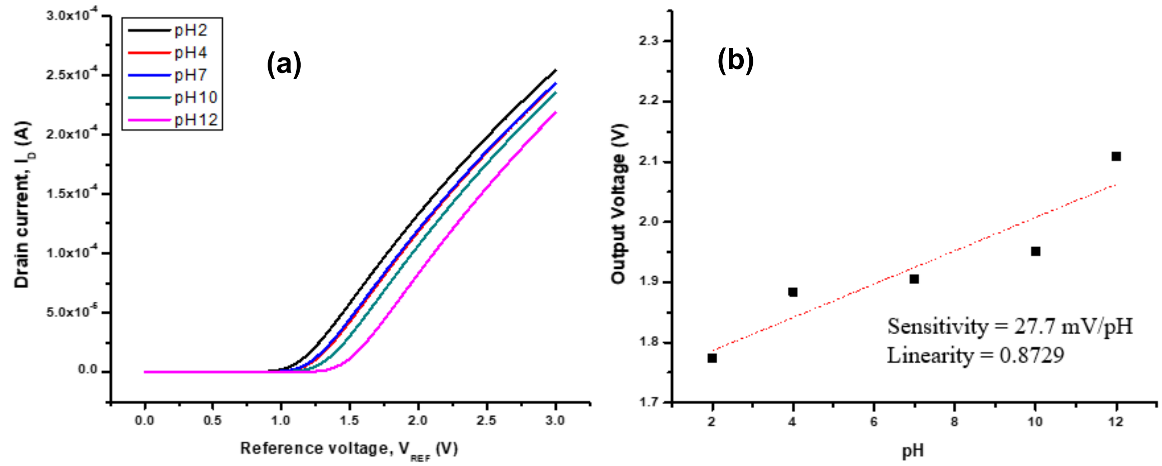

Figure 4 Ag/AgCl RE thickness 60 nm (a) transfer characteristics and (b) output voltage versus pH at drain current 100 μA

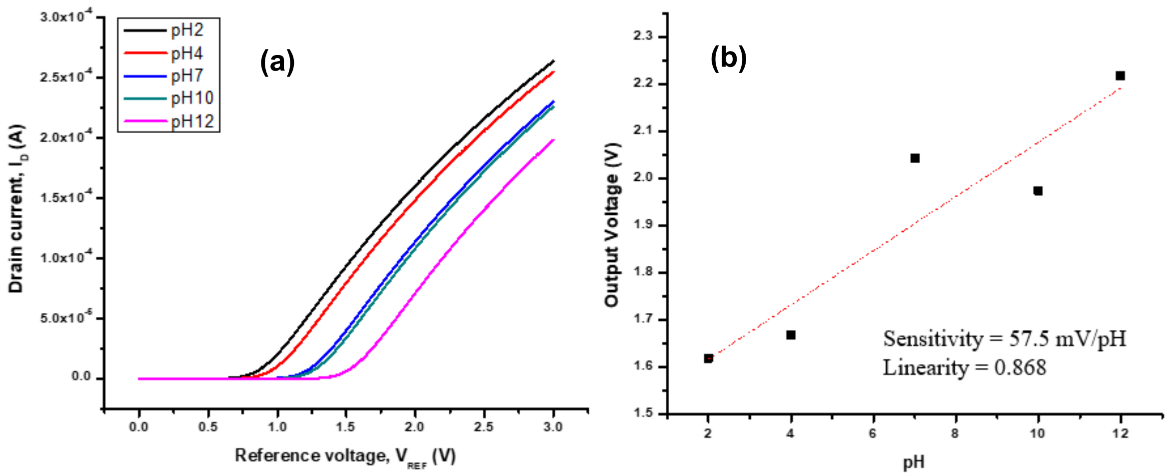

Figure 5 Ag/AgCl RE thickness 180 nm (a) transfer characteristics and (b) output voltage versus pH at drain current 100 μA

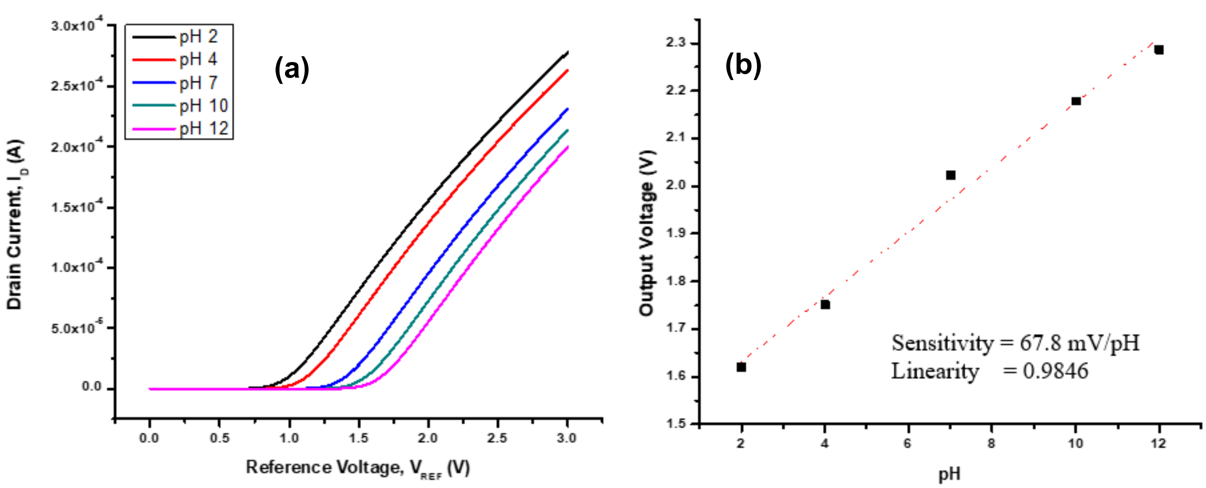

Figure 6 Ag/AgCl RE thickness 300 nm (a) transfer characteristics and (b) output voltage versus pH at drain current 100 μA

In Figure 4, the result indicates the value of sensitivity and linearity is 27.7 mV/pH and 0.8729, respectively, for a 60 nm thickness of Ag/AgCl. These values suggest a notably low sensitivity compared to the expected sensitivity based on the Nernst equation formula [21]–[23]. However, when the thickness of Ag/AgCl is increased to 180 nm, the sensitivity improves to 57.5 mV/pH, while the linearity remains at approximately 0.868. Subsequently, a further increase in thickness to 300 nm significantly enhances the sensitivity (67.8 mV/pH) and linearity (0.9846). These findings indicate that the thickness of Ag/AgCl significantly impacts the sensitivity of pH measurements.

The sensitivity and linearity results of IASE at different thicknesses were tabulated in Table 2.

Table 2 Sensitivity and linearity value of IASE at different Ag/AgCl thickness

Ag/AgCl Thickness (nm)	**60**	**120**	**180**	**240**	**300**
Sensitivity (mV/pH)	27.7	56.7	57.5	58.7	67.8
Linearity	0.8729	0.9654	0.868	0.9741	0.9846

The lowest sensitivity was around 27.7 mV/pH for the 60 nm Ag/AgCl, and as the thickness increased, the sensitivity also increased to the highest, 67.8 mV/pH for 300 nm Ag/AgCl. The observed phenomenon at 300 nm, can be attributed to the enhanced sensor sensitivity resulting from the lower resistance and improved morphology of the thicker Ag/AgCl reference electrode (RE).

Table 3 shows a comparative table summarizing key parameters such as sensitivity, linearity, reproducibility, and other relevant factors from similar works.

Table 3 Comparison of sensitivity, linearity, and reproducibility for different sensor configurations

Reference	**Materials**	**Sensitivity (mV/pH)**	**Linearity (R^2)**	**Repoducibility**	**Other Notable Feature**
Current study	TiO_2 (Sol-gel) + Ag/AgCl	67.8	0.9846	Yes	Simple fabrication, cost-effective, stable response
Eka Maulana et. al [24]	TiO_2 + Ag/AgCl (Spin Coating)	45.229	0.9396	Not Specified	Lower sensitivity and linearity
T.S.V Nguyen et. al [25]	Ag/AgCl with Graphite oxide	150.36	0.9899	Not Specified	Requires additional graphite oxide layer, more complex technique

Eka Maulana *et al.* [24] reported similar works to the current study using a spin coating method resulting in sensitivity and linearity around 45.229 mV/pH and 0.9396, respectively. While T.S.V Nguyen *et al.* [25] study employed Ag/AgCl with graphite oxide, demonstrating a higher sensitivity of 150.36 mV/pH and linearity of 0.9899. However, it requires an additional graphite oxide layer, potentially increasing fabrication complexity and cost. A higher sensitivity (150.36 mV/pH) may be beneficial in certain high-precision applications, but the sensitivity of 67.8 mV/pH reported in the current study is still within an acceptable range for many real-world applications and can provide reliable and stable performance.

4.0 CONCLUSION

As a conclusion, thin film REs made of Ag/AgCl have been successfully produced on an ITO-coated glass substrate at a variety of thicknesses. It has been demonstrated that the thick layer of Ag/AgCl that was deposited is sensitive to variations in pH. Furthermore, the sensitivity of the IASE has been significantly increased as a result of increasing the thickness of the thin film composed of Ag/AgCl materials. The linearity of the IASE increased from 60 nm to 120 nm but slightly decreased at 180 nm before further increased to 300 nm. The highest sensitivity achieved was 67.8 mV/pH, and the linearity was 0.9846 at 300 nm thickness. The FESEM images show the morphology related to the effect of poor sensitivity at lower thicknesses and better sensitivity at higher thicknesses.

Acknowledgement

The authors would like to express their appreciation to Universiti Teknologi MARA for the financial support and Ministry of Higher Education Malaysia under the Fundamental Research Grant Scheme [Project Code: FRGS/1/2021/TK0/UITM/02/50] for the experimental funding support. The authors would also like to thank the NANO ElecTronic Centre (NET) UiTM for their support.

Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

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