

# Characterization of Electrolyte Layer for Dissolved Oxygen Sensor

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**Abstract**— The aim of this study is to optimize the electrolyte recipe of the hydrogel for planar dissolved oxygen sensor. The electrolyte recipe was proposed with chemicals involved such as the PolyHEMA (Poly(2-hydroxyethyl methacrylate)), 99% v/v 1,4-dioxane and potassium chloride (KCl). The electrolytes were tested on the sensitivity towards the chloride ion activity, the thickness and diameter electrolyte and the water uptake rate. The 0.1M KCl electrolyte is a good sensing material with good sensitivity of  $-55.83\text{mV/decade}$  compare to the  $-59\text{mV/decade}$ . The electrolyte gives a low thickness, diameter and a low water uptake rate.

**Keywords**— Ag/AgCl, dissolved oxygen, electrolyte, KCl, PolyHEMA,

## I. INTRODUCTION

**D**ISSOLVED oxygen sensors are essential equipment to be used to measure the oxygen content in liquids such as aquatic water and blood [1],[2]. In dissolved oxygen sensor market, the sensors were very expensive and they are large in size [3]. Smaller devices can be produced in an easier and cheaper method by using the microfabrication technology [4] and also have higher consistency and improved robustness.

In the past few decades, miniaturization is a most common method for the fabrication of the dissolved oxygen sensors [5]. A microelectrode was used to fabricate the dissolved oxygen sensor. Microsensors are devices that only can be assembled manually and fabricated one by one at a time. One of the major types of oxygen sensor is Clark-type sensor. Based on the design of the Clark-type sensor device, the device consists of small electrodes [6]. The electrodes can be

made from silver, gold or even platinum by electroplating. In addition, the electrodes are covered with a layer of hydrogel or aqueous electrolyte [3]. The role of the electrodes and the electrolyte are for electrochemical detection where the electrolytes usually contain chloride ions. The sensor device consists of a thin membrane. This thin membrane is oxygen permeable which allows oxygen to diffuse through it and into the electrolyte [7].

The electrolyte solution may leak out from prolonged use of the dissolved oxygen sensor. The sensing membrane which has a low ability to prevent electrolyte leakage may also be damaged if it is hit with fast moving debris in the water. Hence, it affects the accuracy of the results obtained. Moreover, the sensitivity of the dissolved oxygen sensor is unstable and the sensitivity of the thin film Ag/AgCl element is poor when after a short period of time [8]. The sensor performance must be maintained by developing a better hydrogel system as a medium to hold the electrolyte over a long period of time.

The optimization of silver chloride (AgCl) deposition process on the silver electrode can be conducted to improve the stability of the output currents of the dissolved oxygen sensor and to improve the lifetime of the thin film Ag/AgCl. There are two possible methods to optimize the silver chloride deposition process. One of the methods is the AgCl layer is formed automatically on a pure silver electrode during the chlorination of the electrode. The second method is to pre-form the AgCl layer electrochemically during the fabrication process [9].

The first method is preferred to form the AgCl from a pure silver electrode. However, this method also has its own problem when the anode current density is low. The problem occur usually depends on the partial pressure of the oxygen and the size of the anode area. Based on the size of the anode area, it will consume a longer time for the AgCl layer to deposits on the silver electrode to a certain required thickness when the size of the anode area is wide [10]. On the other hand, the current can be stabilized and AgCl layer can be rapidly formed by making the anode area smaller. However, the lifetime of the AgCl will become short.

In this project, the electrolyte layer is optimized for the dissolved oxygen sensor. The method used to form the AgCl layer on the silver electrodes is using the ferric chloride

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(FeCl<sub>3</sub>) treatment process. AgCl layer was formed by chlorination using ferric chloride as it is a faster process and the coating of AgCl is more uniform. In this chlorination process, the silver electrode is dipped in a ferric chloride solution and is rinsed by using the deionized water [8].

## II. MATERIALS AND METHODOLOGY

### A. Materials

Potassium chloride (KCl), isopropyl alcohol (IPA) and ferric chloride (FeCl<sub>3</sub>) were from Merck. PolyHema (Poly(2-hydroxyethyl methacrylate)) and 1,4-dioxane 99% v/v which are both purchased from Sigma-Aldrich. Deionized (DI) water was from a Milipore Mili-Q water purification system (USA).

### B. Equipment

For the equipment used, Ion Meter and Communication Reference Electrode are used as the testing equipment. The ORION Reference Electrode and the Ion Selective Electrode (ISE) Meter are used for characterization study. A thickness gauge was used to measure the thickness of the electrolyte. Air gun was used to dry the Ag/AgCl electrode.

### C. Electrolyte Layer for Dissolved Oxygen Sensor

The electrolyte layer recipe was prepared using polyHema and 1,4-dioxane [11]. Nine samples of 0.1 g polyhema were prepared with different concentration of 500  $\mu$ L KCl solution (0.1 - 1.0 M) and 500  $\mu$ L of 1, 4-dioxane. The solutions were shaken to dissolve and mix it thoroughly. The solutions were left for 30 minutes to allow the gel formation before deposited onto the surface of the Ag/AgCl electrode to form the electrolyte layer. The electrolyte layer was characterized by conducting the sensitivity of electrolyte to chloride ion activity analysis [3]. The adhesion and the water uptake test were conducted by using an adhesive tape and measuring the weight difference, respectively.

For the sensitivity of Ag/AgCl electrode to chloride ion activity, the ORION pH and Ion Selective Electrode meter is used as the equipment to measure the pH and the electrical potential of the chloride. The chloride sensitivity of Ag/AgCl wire potential and Ag/AgCl wire potential in electrolyte layer is measured against ORION reference electrode in 0.0001M, 0.001M, 0.01M and 0.1M KCl solution using the ISE meter. The graphs of response against log KCl concentration were plotted. The values of slopes obtained were compared to the theoretical potential developed which was -59mV/decade [12]. The optimization studies of AgCl deposition process on Ag electrode were done in FeCl<sub>3</sub> with covering half of the electrode with adhesive tape. Half of the electrode was immersed in IPA solution for cleaning before dip it into 0.1 M FeCl<sub>3</sub> for 1 min, rinsed with DI and blow dry with nitrogen (N<sub>2</sub>) gun. The adhesive tape was peeled off carefully after a day.

### D. Water Uptake Rate Analysis

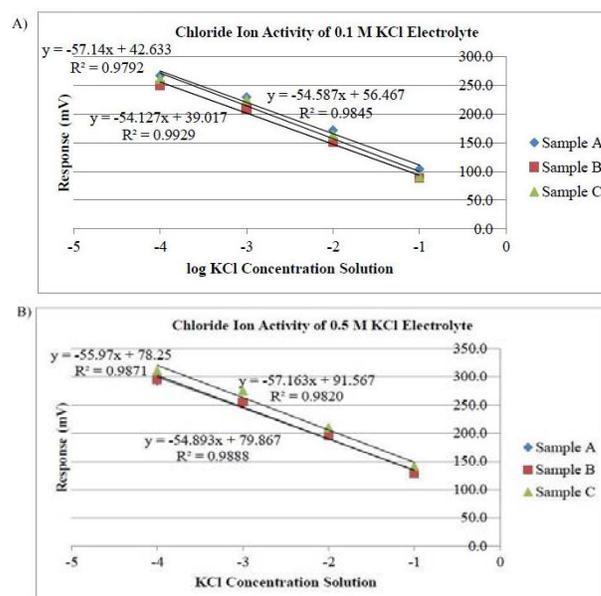
The electrode plate samples were then immersed into water to measure the water uptake rate. Before the samples are immersed in the water, they were weighed. After immersing for 2 hours, the samples were taken out from the water and the whole surface except the electrolyte was dried carefully. The process is repeated for each of the next 6 and 30 hours. This is to check the swelling of the electrolyte by measuring the weight difference.

## III. RESULT AND DISCUSSION

### A. Sensitivity of Electrolyte to Chloride Ion Activity Analysis

The hydroxyl ions are constantly being replaced with chloride ions during dissolved oxygen measurement. KCl can be used as an electrolyte for the dissolved oxygen sensor to provide the chloride ions. However, the electrolyte will slowly experience shortage of Cl<sup>-</sup> ions and this leads to insufficient chloride ions and the need to replace it. Some improvements have been made to overcome the difficulties. The electrolyte layer can be sealed by the oxygen permeable membrane and can be screen-printed on the electrode surface. The charge particles of the electrolyte are able to move freely for measurement by rebuilding the electrolyte by heat or water due to its low melting point [13].

The electrolyte with three different KCl concentrations such as 0.1M, 0.5M and 1.0M and the potential developed of the electrolyte was examined using the ISE meter. The reference electrode casing which contains the electrolyte sample was connected with the Ag/AgCl wire and both are immersed in 0.1, 0.01, 0.001, and 0.0001M KCl solutions. A graph of potential response to chloride ion activity of the electrolyte was then plotted against the log KCl concentrations in Fig. 1.



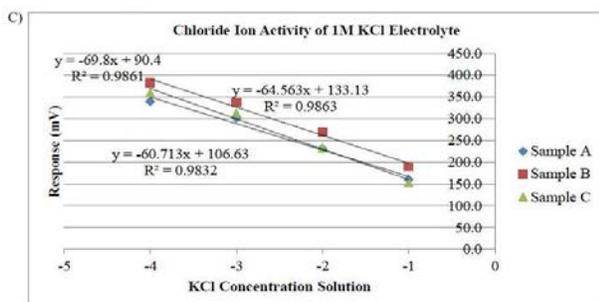


Fig. 1 Chloride ion activity of A) 0.1M KCl, B) 0.5M KCl and C) 1.0M KCl electrolyte.

Based on Fig.1, the graph shows that all of the electrolyte samples with different concentration of KCl give a linear response over the different KCl solutions but with some small difference in the  $R^2$  value. Results show that the stability of the electrolyte with 0.1M KCl concentration was high as an excellent linearity was obtained. A good linearity was also obtained from each of the samples with 0.5 and 1.0 M KCl concentration.

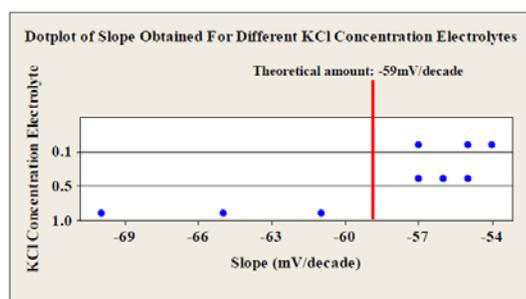


Fig. 2 Dot-plot of slope for each KCl concentration of electrolyte.

A dot-plot of slope for each of the electrolyte samples was plotted in Fig. 2 where the KCl concentrations of the electrolyte were plotted against the slopes obtained from Fig.1. The average slope obtained is  $-55.29\text{mV/decade}$ . Based on the Nernst Equation, the theoretical potential slope developed will be  $-59\text{mV/decade}$  when the chloride ion concentration increases to 10x difference compare to the previous concentration [12]. The values obtained for the 0.1M KCl electrolyte approaches the theoretical slope of  $-59\text{mV/decade}$ , having a relative error of 6.29%. The 0.5M KCl electrolyte with an average of  $-56.01\text{mV/decade}$  gives a relative error of 5.07%. The slopes obtained for 1.0 KCl electrolyte with an average of  $-65.02\text{mV/decade}$  deviates further from the theoretical Nernstian value and give a high relative error of 10.21%. The Nernstian response highly depends on the concentrations and compositions of the electrolyte [14]. The high relative error may be due to the higher concentration of KCl in the electrolyte which causes the higher chloride ion activity. The formation of hydroxides or oxides will have a significant effect on the stoichiometric value. In the electrode reaction, the number of electrons transferred will be changed to some fractional value. The chloride ion activities of the electrolytes were highly active and stable in overall. 0.1M and 0.5M KCl of electrolyte can

be chosen to be used as the electrolyte because the concentration is low, and it also improves the adhesion between the membrane and the silicon electrode. Moreover, it also helps in the reference electrode functionality.

### B. Thickness of Electrolyte Layer Analysis

The electrolyte was dispensed on to the AgCl electrode plates using the dispenser by the same pressure of 0.1MPa and a 0.1s shot time. One set of samples were dried using nitrogen gas and another set were dried under room temperature. In the nitrogen drying, the samples were put into a closed chamber while nitrogen gas was supplied into the chamber continuously for around 30 minutes. The other set of samples were dried overnight under room temperature. The results obtained were tabulated and graphs were plotted. Based on the graph in Fig. 3, it shows that the thickness of electrolytes dried under nitrogen gas was thicker compare to the ones that were dried under room temperature. In room temperature, the electrolyte will dry slower where the liquid is allow to evaporate slowly and will give a thinner thickness compare to the faster drying rate using nitrogen gas. However, there was a slight different result in 0.5M electrolyte where the electrolyte dried under room temperature was found to be thicker compare to the ones dried using nitrogen gas. This may be largely due to some leftover of electrolyte at the tip of the syringe before it was dispensed on the electrode plate. Therefore, extra amount of electrolyte had been dispensed and this will result a thicker electrolyte on the electrode plate.

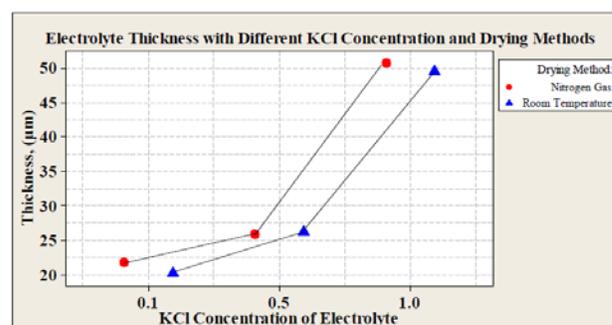


Fig. 3 Multi-variation chart for the thickness of electrolytes under two drying methods.

Fig. 3 also shows that 0.1M KCl electrolyte dried under both methods gives a thinner thickness compare to the other KCl concentration electrolytes by giving an average of  $20.167\mu\text{m}$  and  $21.667\mu\text{m}$ . This result was near to the theoretical result which range between 10-15 $\mu\text{m}$  [15] due to the less concentration of KCl in the electrolyte and the amount of water that can be evaporated more easily compare to the other KCl concentration. The dispersion of 0.1M KCl gave a thinner film allows the oxygen to reach the cathode faster and results in lower response time compare to the thicker layers such as the 0.5M and 1.0M KCl electrolyte. However, thinner electrolytes are less sturdy and do not hold

up better to process conditions that have abrasive properties compare to thicker electrolyte layers. Furthermore, a dot-plot of thickness of each KCl concentration electrolyte under 2 drying methods was plotted in Fig. 4.

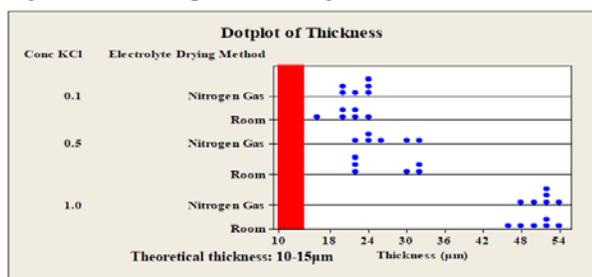


Fig. 4 Dot-plot of each KCl concentration electrolyte and their thickness under two drying methods.

Based on Fig. 4, it was shown that the thickness of 0.1M KCl electrolytes fall in the range of 15.5 to 24.5µm. This results was near to the theoretical result which range between 10-15µm. The properties of the electrolyte were too thick and sticky so it were difficult to be sucked up and dispensed using micropipette. Therefore, pneumatic dispenser will dispense the electrolyte in a larger amount compare to the micropipette. Hence, the thickness obtained will be higher.

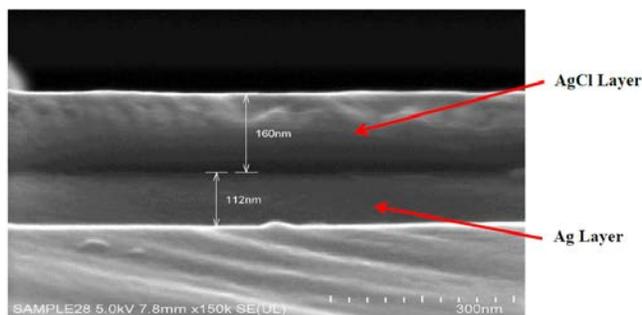


Fig. 5 Images of thickness of AgCl and Ag layer by FESEM.

From Fig. 5, the thickness of the AgCl was 160nm and the Ag layer on the electrode was thin which is approximate 112nm based on the Ag electrode supplied. The Ag was easily consumed off when it is chlorinated in FeCl<sub>3</sub> solution and easily scratched off due to the thin thickness of the Ag layer. The thickness of Ag layer should be improved to about 250 nm [16]. Moreover, another difficulty was it was hard to handle the Ag electrode due to its small size during chlorination.

### C. Electrolyte Water Uptake Rate Analysis

From the graph in Fig. 6, it can be seen that at the first 2 hours, the water uptake is high for every concentration of electrolyte, the water uptake rate for each of the electrolyte of 0.1, 0.5 and 1.0M KCl concentration electrolyte dried using nitrogen gas were 0.197, 0.175 and 0.2% g/hour. The water uptake rates for three different concentrations of electrolytes were almost the same because the dry electrolyte absorbs more water in the early stage. The water entered the

electrolytes by simple diffusion in response to the osmotic gradient established by the electrolyte. The water uptake rates for each of the 0.1, 0.5 and 1.0M KCl concentration electrolytes dried under room temperature were found to be 0.3, 0.2 and 0.4% g/hour based on Fig. 6. There was a high water uptake rate in 1.0M KCl electrolyte due to the higher thickness of the electrolyte which was 49.5µm compare to the 0.1 and 0.5M KCl electrolyte. More water absorbed by a thicker electrolyte.

After some time at 6 hours, the water uptake rate was then calculated based on the weight measured. It was found that the water uptake rate of each of the 0.1, 0.5 and 1.0M KCl concentration electrolyte dried using both methods had decreased. After 30 hours, the water uptake rate of each of the electrolyte is almost same due to the amount of water taken into the electrolyte had become maximum. Therefore, less amount of water will be taken up after 30 hours. Based on Fig. 6, the 0.1M KCl electrolyte can be chosen to be use in the dissolved oxygen sensor. This is because the water uptake of the 0.1M KCl electrolyte is lower and does not cause the electrolyte itself to swell and eventually burst. When the electrolyte swelled, it tends to be more easily peeled off from the surface of the electrode when high water pressure is exerted from in to out of the electrolyte.

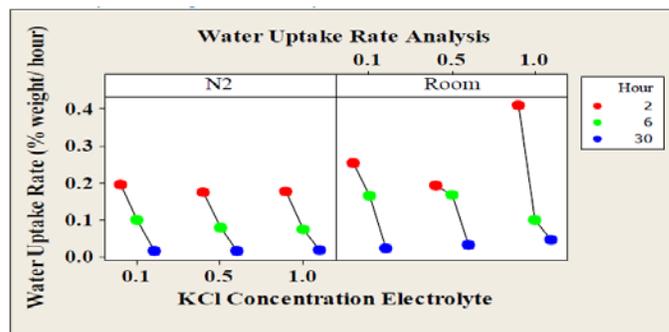


Fig. 6 Multi-varied chart for diameter of each KCl concentration electrolyte dried with two different methods.

### D. Sensitivity of Ag/AgCl Electrode to Chloride Ion Activity Analysis

Before the chlorination process, the samples of silver (Ag) electrode were cleaned using the chemical cleaning and IPA solutions. After that, the silver electrodes were then chlorinated by immersing them in the ferric chloride FeCl<sub>3</sub>. According to the Nernst Equation, the potential developed by an Ag/AgCl half-cell would be a theoretical amount of -59mV/decade for every 10x difference in the concentration of Cl<sup>-</sup> [12].

Table I shows a data of sensitivity of Ag/AgCl electrode to chloride ion activity analysis for two different cleaning method, chemical cleaning and IPA cleaning. The electrodes were tested in solutions of KCl with different concentrations. Excellent linearity of the 3 samples were obtained with the correlation coefficient of >0.96.

TABLE I  
SENSITIVITY OF Ag/AgCL ELECTRODE TO CHLORIDE ION ACTIVITY

KCl, M	Log [KCl]	Electrode Cleaned With Chemical Cleaning Solution Response (mV)			Electrode Cleaned With IPA Cleaning Solution Response (mV)		
		Sample A	Sample B	Sample C	Sample A	Sample B	Sample C
0.001	-3	199.6	198.3	208.4	192.6	195.9	189.8
0.01	-2	149.6	145.3	146.5	143.1	146.8	143.6
0.1	-1	101.2	103.8	116.9	96.6	99.6	101.0
Slope (mV/decade)		-49.17	-47.27	-45.75	-48.00	-48.18	-44.42
R2 value		0.9999	0.9951	0.9600	0.9996	0.9999	0.9994

Based on response to chloride ion activity of Ag/AgCl electrode, the results were recorded and a multi-varied chart was plotted as shown in Fig.7. It can be seen that the mean slope obtained was found to be -47.40mV/decade when the Ag electrode was cleaned using chemical cleaning solution and the mean slope obtained was -46.87mV/decade when the Ag electrode was cleaned using IPA cleaning solution, but with a high relative errors of 19.7% and 20.5%. The response to the activity of the chloride may be affected by some elements, such as the uniformity of AgCl layer on the Ag electrode after chlorination, purity of the Ag electrode and the type of chemical used for cleaning of the Ag electrode.

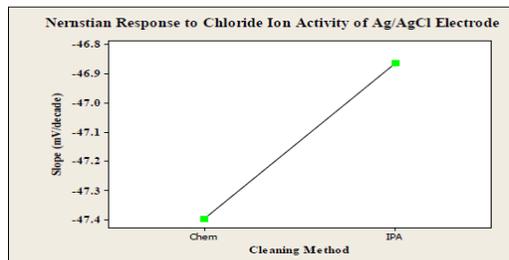


Fig. 7 Average slope obtained of two different cleaning methods

To produce a stable electrode, 10-25% [17] of Ag on the electrode was needed for the formation of AgCl layer. In this experiment however, the ferric chloride may consume off the thin layer of Ag easily and there were no Ag/AgCl layer left on the surface. Therefore, the idea of increasing the thickness of the Ag layer to 250nm [16] should be propose to improve the AgCl deposition. The type of cleaning solution used to clean the Ag electrode in this experiment may also affect the accuracy of the results obtained. The IPA solution was used to remove dirt particles such as residues left on the Ag electrode during the sawing process. Chemical cleaning solution was used to remove silver oxide or grease. The chemical cleaning solution formula was undisclosed due to the company regulations. Therefore, there's a need to improve and optimize the chemical cleaning solution to make sure the Ag layer is free from dirt, contaminant and oxides. This is because will result in poor adhesion with the present of contaminant in between the AgCl and Ag layers and the uniformity of the AgCl layer will be poor. More improvements shall be made to minimize the high relative errors by optimizing the chlorination of the Ag electrode.

#### IV. CONCLUSION

In general, the proposed recipe of the electrolyte was used which involves the PolyHEMA, 1,4-dioxane and KCl. The effect of the concentration of the KCl in the electrolyte on the sensitivity to the chloride ion activity, thickness and diameter and the water uptake rate was determined. It was found that the electrolytes give a good stability according to the excellent linearity of graphs obtained. Besides that, the 0.1M KCl electrolyte shows to be a good sensing material within a sensitive range, gives a low thickness and diameter and has a low water uptake rate and the chances of the electrolyte to swell are low. Therefore, the 0.1M KCl electrolyte can be chosen as the best electrolyte for the hydrogel in the dissolved oxygen sensor.

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