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Master's Degree Thesis

Development of Telemetric and Miniaturized Electrochemical Amperometric Analyzer with Indium Tin Oxide Sensor

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Department of IT Fusion Technology
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인듐 주석 산화물 센서를 이용한 전류 적정법 기반의 무선 통신이 가능한 소형화된 전기화학 분석기 개발

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Graduate School of Chosun University

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Development of Telemetric and Miniaturized Electrochemical Amperometric Analyzer with Indium Tin Oxide Sensor

Advisor: Prof. Youn Tae Kim

This thesis is submitted to The Graduate School of Chosun University in partial fulfillment of the requirements for the Master's degree.

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This is to certify that the Master's Thesis
of Jihoon Lee

has been approved by the Examining Committee for the thesis requirement for the Master's degree in IT Fusion Technology.

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Acronyms

ITO	Indium Tin Oxide
I/V	Current-to-Voltage
PCB	Printed Circuit Board
PBS	Phosphate-Buffered Saline
POCT	Point-of-Care Testing
BNP	B-type Natriuretic Peptide
MCU	Microcontroller Unit
USIM	Universal Subscriber Identity Module
PSoC	Programmable System-on-Chip
DAC	Digital-to-Analog Converter
ADC	Analog-to-Digital Converter
Ag	Silver
Ag/AgCl	Silver-Silver Chloride
ACK	Acknowledge message
PHD	Personal Health Device

ABSTRACT

Development of Telemetric and Miniaturized Electrochemical Amperometric Analyzer with Indium Tin Oxide Sensor

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We developed a portable three-electrode electrochemical amperometric analyzer, which can transmit result data to a PC or smart tablet via Bluetooth communication, and we conducted experiments with an indium tin oxide (ITO) glass electrode to confirm the performance reliability. This analyzer uses a current-to-voltage (I/V) converter to convert the micro-current from the reduction and oxidation (redox) reaction of the ITO electrode with a buffer solution into a voltage. This signal is then digitized in the processor. The configuration of the power and ground of the printed circuit board (PCB) layer is divided into digital and analog parts to minimize the noise interference of each part. The analyzer has a size of 5.9 × 3.25 cm^2. A potential of 0~2.1 V can be applied between the working and counter electrodes, and the analyzer has a current resolution of 0.4 nA. This study demonstrated the accuracy of the analyzer by measuring the Ru^{III} concentration in 10 mM phosphate-buffered saline (PBS) with pH 7.4. The measured data could be transmitted to a PC or smart device such as a smart phone or tablet PC using the included Bluetooth module. This device is powered by a 3.7 V, 120 mAh lithium polymer battery and can be operated for 60 mins, including data processing and wireless communication, once fully charged.

요 약

인듐 주석 산화물 센서를 이용한 전류 적정법 기반의 무선 통신이 가능한 소형화된 전기화학 분석기 개발

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본 연구에서는 블루투스 통신이 가능하고 전기화학 신호를 측정할 수 있는 전류 적정 기반의 휴대용 3전극 전기화학 분석기를 개발하였으며 인듐 주석 산화물로 제작된 유리 전극을 이용하여 실험을 진행하였다. 이 전기화학 신호분석기 는 인듐 주석 산화물 센서의 산화-환원 반응으로부터 일어나는 미세 전류를 전류-전압 변환기를 통해 전압으로 변환하여 주 처리장치에 입력되는 구조이다. 아날로그 부분과 디지털 부분으로 분리하고 PCB 층의 전원과 접지의 구조를 용도에 따라 분리함으로써 잡음으로 인한 간섭을 최소화 하였으며 분석기의 크기는 5.9 x 3.25 cm^2 작은 크기로 제작하였다. 전극에 0~2.1 V의 전압을 인가할 수 있으며, 해상도는 0.4 nA를 갖는다. 10 mM의 인산염 버퍼용액 (pH 7.4) 속 루테늄의 농도 별 실험을 통해 전류 적정 기반 분석기의 정확도를 확인하였다. 블루투스가 탑재되어 있어 측정된 결과를 무선으로 데스크탑 또는 스마트폰에 전송이 가능하다. 전원은 3.7 V, 120 mAh 리튬 폴리머 전지를 사용하였으며 한번 충전으로 데이터 처리 및 통신을 포함하여 60분 동안 디바이스의 동작이 가능하다.

I. Introduction

Improvements in technology ensure continual developments in the point-of-care testing (POCT) field [1]. POCT, which can be used to diagnose personal health conditions by extracting a small blood sample, is widely used to manage diseases. For example, a glucose analyzer, which is a representative POCT device, can be used to check the glucose concentration in a patient's blood by converting the chemical reaction generated at a biochemical sensor to an electrical signal [2]. Such a type of device is beneficial to those who desire to manage their health parameters because it not only has a simple design but also is inexpensive and compact [3].

An electrochemical analysis based on amperometry, which is one of the methods used to measure the current induced by a reduction-oxidation (redox) reaction at an electrode, is a suitable analysis method for the previously mentioned devices. A potential is applied between the working and the counter electrodes using a potentiostat, and the current from the redox reaction is measured [4], [5]. A reference electrode is used to maintain a stable potential voltage during the reaction [6], [7].

i-STAT (Abbott) is one of the available electrochemical analyzers [8]. The i-STAT system uses a different cartridge to store a blood sample and can detect various biomarkers, including troponin I, B-type natriuretic peptide (BNP), and glucose, facilitating medical professionals to gauge a patient's health condition. CHI 1040C (CH Instrument) is a multi-potentiostat that can precisely check eight electrochemical signals simultaneously and analyze a wide range of materials using diverse methods such as cyclic voltammetry and differential pulse voltammetry [9]. EmStat (PalmSens) is a portable multichannel analyzer based on three electrodes [10]. It includes four-channel working, counter, and reference electrodes and can detect redox reactions in various analyses. In addition, devices based on electrochemical analysis are applied in an extensive range of fields, such as for lactate, toxin, and gas analyses [11], [12], [13].

However, conventional instruments are too expensive, and their use for study or medical diagnosis requires the skill of an expert [14], [15]. Moreover, the existing devices require complex procedures and an established setting for the test to measure a solution, which can be cumbersome. For these reasons, the existing devices are not only difficult to use in various fields but also unsuitable for use as portable analysis devices.

In this study, we developed a miniaturized amperometric analyzer that can be applied to POCT or a portable detector. We used micropatterned ITO glass as an electrode to confirm the electrochemical activity. For easy operation and user convenience, the proposed analyzer was designed to communicate with a smart phone, a tablet PC, or a desktop using Bluetooth. A user can remotely control the analyzer and confirm the result data using a PC or smart phone. This analyzer is based on an amperometry method.

II. Method

Ⅱ.1 Design of Amperometric Device

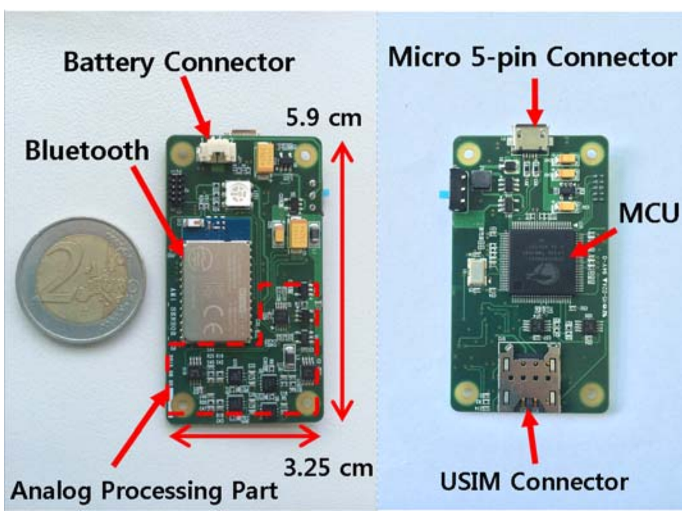


Figure 1. PCB of miniaturized amperometric analyzer

Figure 1 shows the printed circuit board (PCB) configuration of the proposed analyzer. It comprises a data processor that includes a microcontroller unit (MCU), along with analog, power, and communication parts. To accurately measure the microcurrent generated from an electrochemical reaction, a universal subscriber identity module (USIM) connector, which is connected to a piece of ITO glass, is placed near the analog processing circuit. The use of the shortest possible distance for the circuit line minimizes the inflow of external noise.

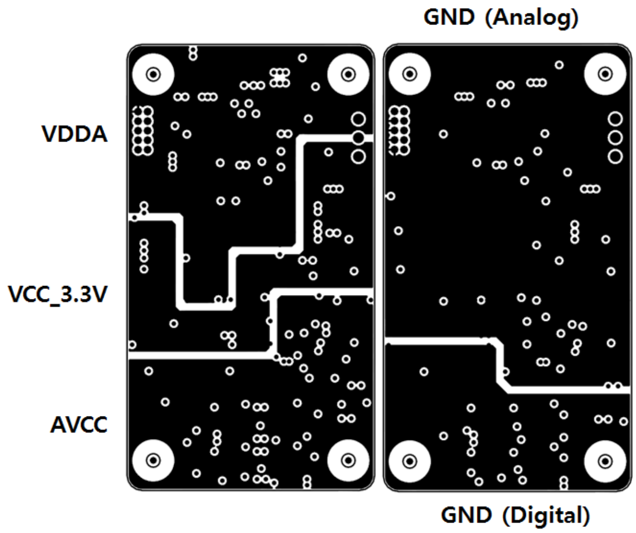


Figure 2. Structure of PCB for eliminating interference

In addition, Figure 2 shows that the power and ground layers of the PCB are divided by a functional part to prevent interference. This division of the two layers enables the accurate detection of a signal without effects from each part. In this study, we used a 3.7 V, 120 mAh lithium polymer battery, which made it possible to perform operations, including data processing and Bluetooth communication, for 60 mins. This enabled the analyzer to be operated fifty times when the measurement time was set at 50 s. The battery could be recharged through a micro 5-pin USB cable. To transmit the result data processed by the MCU, a Bluetooth 4.0 module (WT-12, Bluegiga) was used. CY8C5888-LP based on Cortex-M3 (Cypress) was used as the MCU, exhibiting high performance and low power consumption. CY8C5888-LP is a programmable system-on-chip (PSoC) series, the use of which made it possible to generate an analog block in the MCU via programming. This enables 12-bit digital-to-analog convertor (DAC) in the MCU, along with the easy editing of device functions. The PCB occupies an area of 5.9 × 3.25 cm^2.

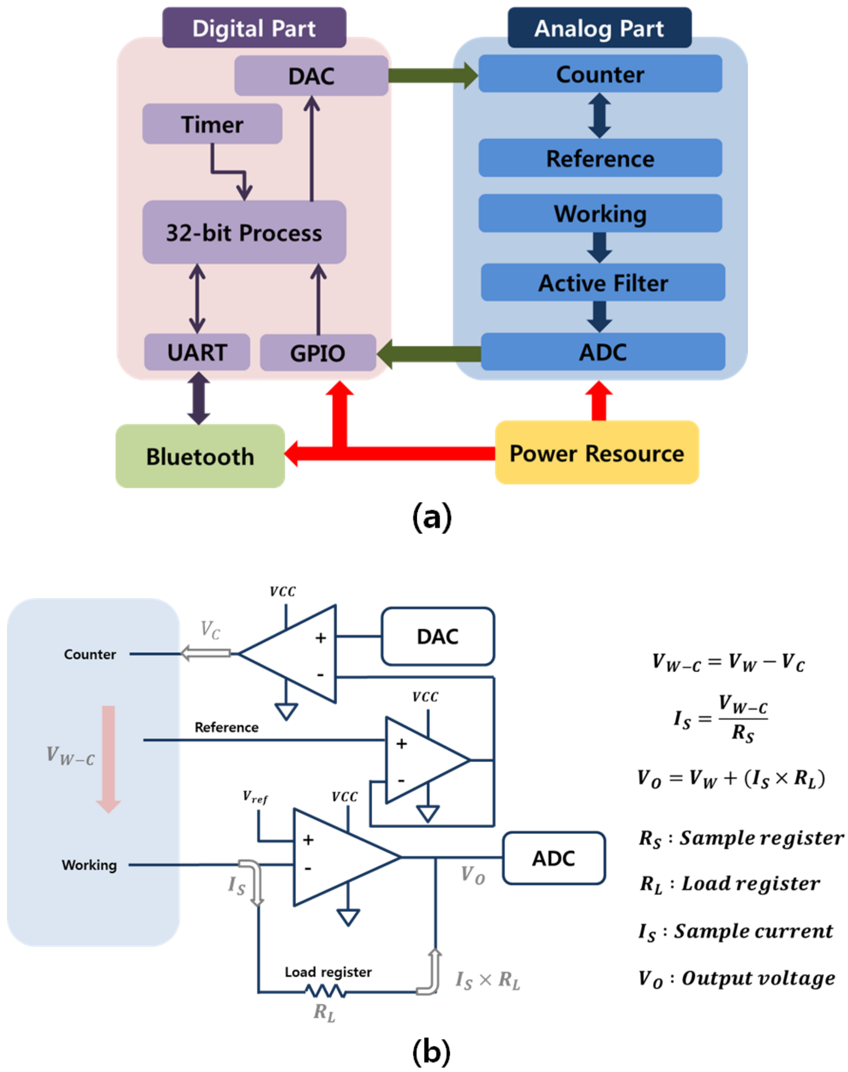


Figure 3. (a) Device block diagram, (b) Configuration of sensing part

The power source of the analyzer supplies its various functions, including the Bluetooth, digital part, and analog part, as shown in Figure 3 (a). The analog part consists of the potentiostat, including the counter, reference, and working circuits, as well as an active filter and a 16-bit analog-to-digital converter (ADC). An OPA2376 (Texas Instruments) op-amp is used for the reference, counter, and active filter circuits, whereas the working circuit uses an OPA381 (Texas Instruments), which is a high-resolution current-to-voltage (I/V) converter. When a potential is applied to a solution, the reference circuit stabilizes the output voltage of the counter circuit using a feedback loop. The electrochemical signal from the analog part is converted into a digital value via the ADC, which is then transmitted to the MCU. The data are processed into packets for wireless communication.

Figure 3 (b) shows the current flow generated by the redox reaction, V_{ref}, which is the basis for working voltage V_W, has a value of 1.2 V, and V_C is controlled by the DAC. The potential V_P is the differential voltage between the working and counter electrodes ($V_P = V_W - V_C$), and R_S is the solution or sample resistance. The electrochemical reaction of the solution represented by the potential produces I_S, which is equal to $V_P \div R_S$ based on ohm's law. This current flows in load register R_L, which has a value of 1 MΩ, and is then converted to the load voltage $V_L = I_S \times R_L$. Finally, a readable voltage V_O is produced by the sum of V_W and V_L before being input to the ADC. We experimented with a sample resistance of 10 MΩ and potential of 500 mV to check the device performance. The measurements of V_W and V_L were 1200 and 50 mV, respectively, which meant a current of 1 nA was equivalent to 1 mV.

The proposed analyzer device adopts Bluetooth communication. A manager device embedded Bluetooth such as a PC, smart phone, or tablet PC provides the analyzer with settings for the potential, maximum number of samples, and sampling interval. Electrochemical analysis starts immediately after the setting information established by the manager is transferred and continues for the entire runtime, which is the sampling count multiplied by the interval. When the operation is completed, the result data are transmitted to the manager, where they can be represented graphically and stored in a text file format.

Ⅱ.2 System Operation Flow

The data process between the analyzer and the manager is shown in Figure 4. When the Bluetooth pairing between the analyzer and the manager is successful, the analyzer is ready to activate the analysis and start the timer. When the analyzer receives information about the requirements for a run, it starts analyzing the solution during the allocated time. All the results are converted into packets for Bluetooth communication after the completion of the measurement. The packets are numbered and transmitted to the manager in a numerical order through the Bluetooth module. Typically, when a packet arrives at the manager, the manager sends the analyzer an acknowledgement message (ACK) corresponding to the sequence. When the analyzer receives an ACK, the next packet is transferred. When the last packet is transmitted by these steps, the entire operation is completed after the manager sends a termination message to the analyzer.

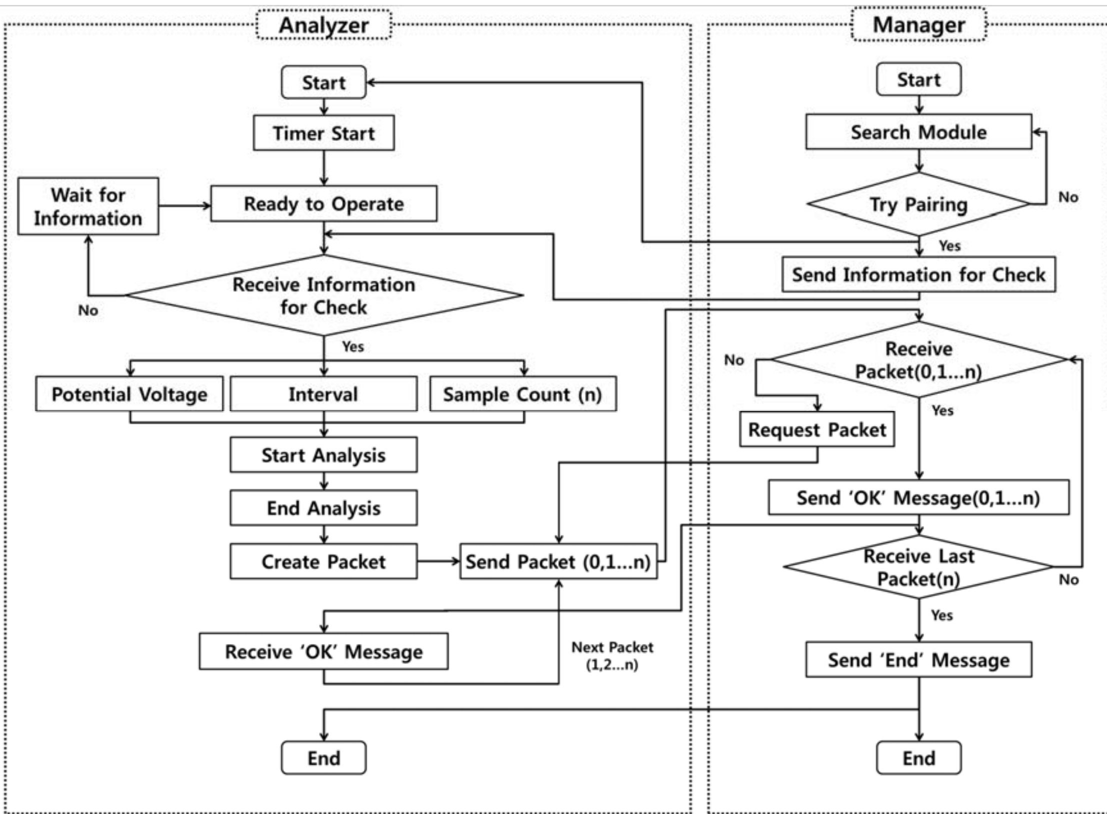


Figure 4. System operation flow via Bluetooth communication

Ⅱ.3 ITO Glass Sensor

In this study, an electrochemical sensor manufactured by micro-patterned ITO glass was used for signal analysis. This consisted of working, reference, and counter electrode, with the reference and counter electrode treated with silver (Ag) and silver-silver chloride (Ag/AgCl) paste, respectively, for the optimized detection of an electrochemical signal. Using ITO glass for electrochemical electrodes has many advantages, including a wide potential window, low capacitive current, and highly practical fabrication of micro-electrodes [16].

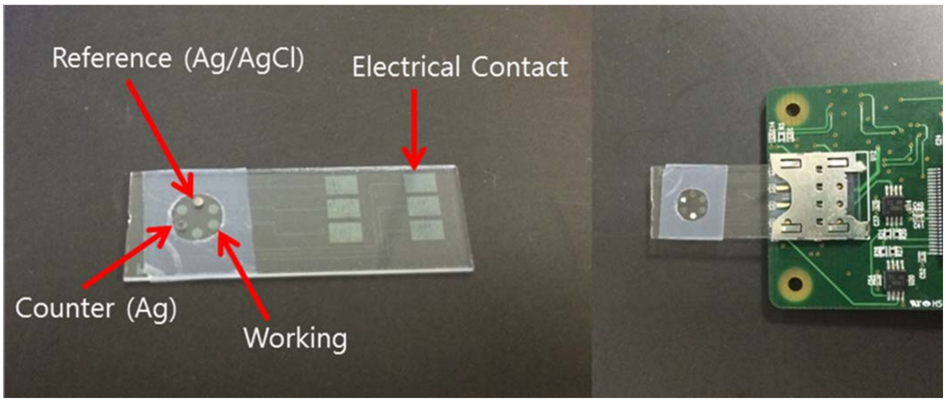
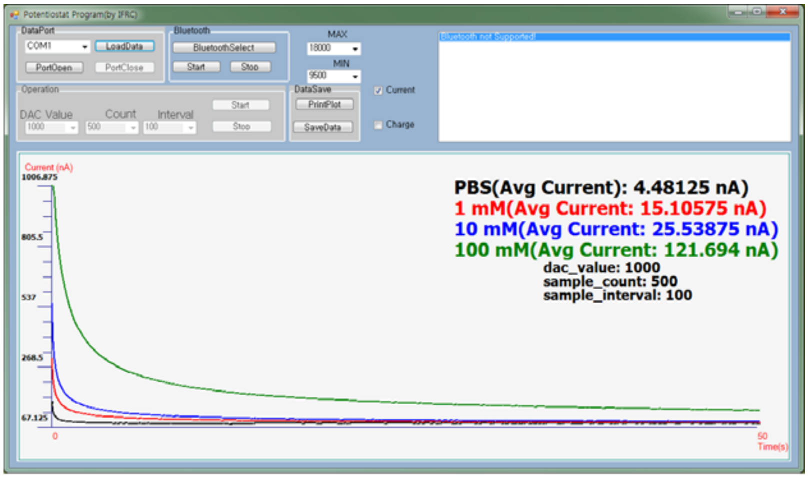


Figure 5. Patterned electrodes fabricated using ITO glass

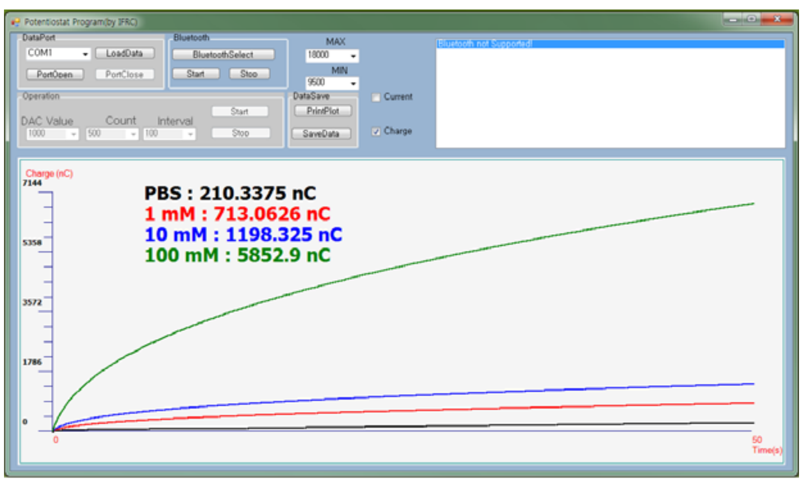
Pattern of ITO sensor fabricated using the same specifications as the USIM connector are shown in Figure 5. This consists of counter, reference, and three working electrodes. In this study, the unmarked electrodes were not used in the test. Their circuits were disconnected from the system to prevent them from having any effect during the electrochemical reaction. Because the ITO pattern connected to the electrode could have an adverse effect on the result, the unnecessary area of the face was blocked, except for the ITO electrode face that is in direct contact with the solution.

III. Results and Discussion

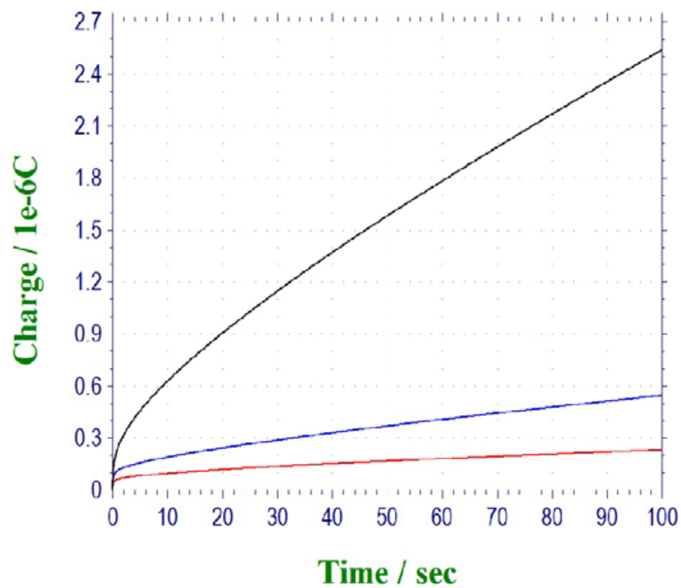
We established settings for the experiment after connecting the Bluetooth dongle to a PC and after enabling the pairing of the dongle with the analyzer. Each experiment used 10 μL of solution at the ITO sensor. The experiment was performed at a potential of 0.2 V with a measurement time of 50 s. The measurement result was displayed in terms of current and an integrated charge with respect to time. We conducted experiments to identify the analyzer performance using 1 mM, 10 mM, and 100 mM of Ru^{III}. The solution for testing is produced using 10 mM PBS at 7.4 pH. Figure 6 shows the graphs of the results of this research, where the Ru^{III} concentrations are represented in the PC program.


Potentiostat Program(by IFRC)
Current (nA)
1006.875
805.5
537
268.5
67.125
0
50
Time(s)
PBS(Avg Current): 4.48125 nA)
1 mM(Avg Current: 15.10575 nA)
10 mM(Avg Current: 25.53875 nA)
100 mM(Avg Current: 121.694 nA)
dac_value: 1000
sample_count: 500
sample_interval: 100

(a)


Potentiostat Program(by IFRC)
Charge (nC)
7144
5358
3572
1786
0
0
50
Time(s)
PBS : 210.3375 nC
1 mM : 713.0626 nC
10 mM : 1198.325 nC
100 mM : 5852.9 nC

(b)



(c)

Figure 6. (a) Current showing PBS solution and as the concentration level of Ru^{III} (Black: PBS, Red: 1mM, Blue: 10mM, Green: 100mM), (b) Charge as the concentration level of Ru^{III}, (c) Result of Ru^{III} test using CHI-1040C (Red: 1 mM, Blue: 10mM, Black: 100mM)

Figure 6 (a) shows a graph of the current over time based on amperometry. However, because it was difficult to visually classify the amount of current per concentration of the solution in the graph, we made it possible to identify differences in the reaction of the sample and concentration by displaying a graph of the charge over time, as shown in figure 6 (b).

Table 1. Results of experiment with Ru^{III}

	PBS	1 mM	10 mM	100 mM
Current	4.5 nA	15.1 nA	25.5 nA	121.7 nA
Charge	0.2 μC	0.7 μC	1.2 μC	5.8 μC

In the Table 1, average currents of 4.5 nA, 15.1 nA, 25.5 nA, and 121.7 nA, and electric charges of 0.2 μC, 0.7 μC, 1.2 μC, and 5.8 μC, were measured about PBS and solution as the concentrations of Ru^{III}, which are 1 mM, 10 mM, and 100 mM respectively. We could identify the linear increase in the signal by augmenting the solution concentration using a tenfold increase, and these results were compared to result by CHI-1040C. Although, current level differed from both results, change as concentration of solution was similar. The PC program was developed using Visual Studio 2010, C# based on Windows 7 32-bit.

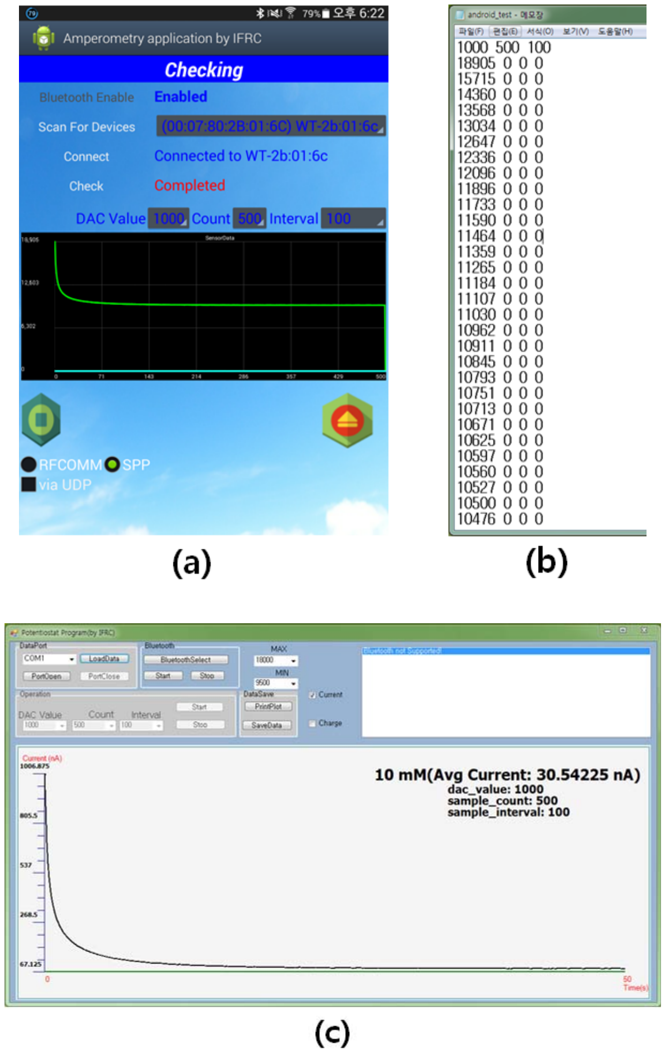


Figure 7. (a) Results for 10 mM Ru^{III} shown on smart tablet, (b) Result data in text file format, (c) Graph of current in PC program

Figure 7 (a) shows the measurement results for 10 mM of ruthenium in an environment identical to the previous one, as displayed using a smart tablet. An analyzer for measurement is operated by application program. When completing an analysis, the results, which are a type of raw data, are saved in the form of a text file, as shown in Figure 7 (b). This data from the

MCU can be converted to a current value through a simple modification. In addition, the data can be displayed using a PC, similar to that shown in Figure 7 (c), after being transmitted to a web server via mobile communication (Wifi, 3G, or 4G). In this study, a Samsung tablet (SHW-M500W) equipped with a Bluetooth 4.0 module was used. Moreover, an application was developed based on an Android 4.1 (Jelly Bean) and web server was conFigured by APMSETUP 7 for Win-32.

In this study, we developed an accurate and inexpensive electrochemical analyzer based on amperometry. The proposed analyzer can communicate with various other devices through Bluetooth communication and has an intuitive interface that is user friendly. But, the capacity of packet storage of the MCU can be exceeded by an increase of the sample amount. A real-time processing communication, which is that a packet of processing data is transmitted to terminal immediately, without waiting construction of whole packet, is appropriate to alleviate this situation. Therefore, it is necessary to synchronize the data-processing rate and the Bluetooth communication rate when data is transmitted immediately after generating each packet, because if both rates are different, the system will operate abnormally or stop. To solve this problem, an additional study is needed about the synchronization of MCU and Bluetooth transmitting rates. From the results of this research, we expect a faster data process, as well as low battery consumption by reduction of operating time from analysis to termination of communication.

In the main study, the developed analyzer is operated on the basis of Bluetooth communication. Bluetooth communication based version 4.0 takes approximately 45 ms to transmit a data packet and receive a confirmation response at baud rate of 115200 bps. The amount of time spent in transmitting all of the data increases as an increasing number of samples. If abnormal packet is sent to analyzer during the communication, measurement is interrupted. To prevent the cessation of all operations in the event of lost and damaged packets during the communication process, the analyzer was designed to retransfer these packets from the device to the manager using a stop-and-wait protocol. We secured the operational stability of the proposed analyzer using this design. Subsequently, the proposed analyzer can apply the ISO/IEEE 11073 standard protocol because the WT-12 attached to the PCB supports a personal health device (PHD) with Bluetooth 4.0. By developing a wireless health device with the application of this protocol, we estimate to enhance the data transmission efficiency and compatibility with other communication devices.

An ITO glass sensor with the pattern shown in Figure 5 was used to measure a single signal. An optimal signal was detected in a processing experiment with identical settings for the distances of the counter, reference, and working electrodes. However, additional research is required to change the magnitude of the electrode and the pattern to eliminate the oxygen-bubble phenomenon, which can be caused by a redox reaction, depending on multichannel signal detection, and the solution or experimental circumstances [17], [18]. Research on the development of an electrochemical immunosensor that can detect specific biomarkers by applying an antibody fixation technology to ITO is currently underway [19]. Such a sensor is expected to be applied to a new field of POCT, as well as U-healthcare thereby enabling the diagnosis of diseases, such as through the use of cardiac markers, by combining this type of sensor with the analyzer developed in this research.

IV. Conclusion

A portable three-electrode electrochemical analyzer with wireless communication capability was developed through this research. The proposed analyzer measures the current signal generated by an electrochemical sensor based on the amperometry and telecommunicates the result. Further, the proposed analyzer was designed such that the potential and measurement time can be set through a convenient interface, which is used to save and display the results using a graph and numerical values. Because CY8C5888, which is one of the PSoC series, can support analog functions through programming, functions can be actively changed or developed on the basis of its use, without changing circuits. The WT-12 MCU and Bluetooth module consume a low amount of power and exhibit high performances. Thus, the system had no difficulty in operating with a low-capacity battery (120 mAh), which can be replaced with one having a higher capacity. We measured samples using micropatterned ITO glass and identified the difference in the concentrations of solutions in experiments. Thus, we proved that it was possible to accurately perform electrochemical analysis using a device with a low production cost.

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