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Article

Sensing Characteristics of Arrayed Flexible Chloride Sensor Based on the XBee Wireless Sensing System

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Abstract: Water quality monitoring was an important objective in the surroundings. In this study, we investigated the sensing characteristics of the arrayed flexible chloride sensor with XBee wireless sensing system. The sensitivity and linearity of the wireless chloride sensing devices were 91.6 mV/pCl and 0.988, respectively. The hysteresis voltages were 50.14 mV and 36.71 mV during the cycles of 1 M→ 10^{-1} M→ 1 M→ 10^{-3} M→ 1 M and 1 M→ 10^{-3} M→ 1 M→ 10^{-1} M→ 1 M, respectively. The selectivity coefficients of the ClO^- ion, ClO_4^- ion, NO_3^- ion and I^- ion for Cl^- ion were 5.0×10^{-2}, 1.0×10^{-1}, 5.9×10^{-3} and 5.6×10^{-1}, respectively. The sensing characteristics of real time measurement were investigated for dynamic microfluidic. The arrayed flexible chloride sensor was integrated with the microfluidic device, syringe pump and wireless sensing system. The sensitivity and linearity were 273.1 mV/pCl and 0.978 at 35 μL/min, respectively.

Keywords: arrayed flexible chloride sensor; wireless sensing system; hysteresis voltage; selectivity coefficient; dynamic microfluidic

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1. Introduction

Industry countries had a common problem that was water pollutions. Water quality monitoring was an important objective in the surroundings. Many researchers [1-4] used the chemical sensors and optical sensors to measure and analyze the water compositions for the water quality. S. F. Gonski et al. [1] used the Honeywell Durafet pH sensor to measure the pH variations of the river. They investigated the water pollution levels by Honeywell Durafet pH sensor. N. Kato et al. [2] investigated the residual chlorine concentrations of the tap water by detecting electrode and compensating electrode.

They investigated the residual chlorine selectivity for their sensors, thus the interferences obtained the hydrogen ion concentration (pH) and dissolved oxygen (DO). A. Aisopou et al. [3] used electrochemical and optical technologies to measure conductivity, pH, free chlorine, total chlorine, DO, oxidative redox potential (ORP) and turbidity. N. Carbó et al. [4] used the voltammetry electronic tongue and pulse voltammetry method to investigate the quality parameters in spring water. The composition of the spring water contained nitrate, sulfate, fluoride, chloride, sodium. The pH ranges of spring water were from 7.3 to 7.8.

The drawbacks of the water quality monitoring were the detection distance limit and operation space limit. Many researchers detected different ions and obtained experiment data easier with wireless sensing system [5-15]. The wireless sensing system had been applied to different fields, such as chloride ion detection [5-16], hydrogen ion detection [17-23], glucose detection [23-25] and lactate detection [25].

S. Zhuiykov et al. [5] investigated the wireless sensing devices of the conductivity, pH, dissolved metal ions, dissolved oxygen (DO) and dissolved organic carbon (DOC). K. G. Ong et al. [6] detected accurately the hypochlorite ion variations with the wireless sensing devices. The hypochlorite ions sensor integrated with wireless transmission system, and investigated the hypochlorite ion concentration variations, and the hypochlorite ion sensor included the layer of polyurethane and alumina on the magnetically-soft ferromagnetic ribbon. S. Zhou et al. [7] used the Ag/AgCl electrode, reference electrode and radio-frequency identification (RFID) communication protocol to measure and investigate the chloride ion concentrations in the concrete.

N. Harris et al. [8] used the screen printed technology to prepare the low cost robust chloride ion sensor. They investigated the sensing characteristic of the chloride ion sensor with IEEE 802. 15. 4 and secure digital (SD) memory card. The chloride sensors were applied to measure the chloride ion concentration for soil column, fluvarium and greenhouse. K. A. Nyni et al. [9] developed the wireless health monitoring system for health monitoring, which contained the devices of the electro cardio gram (ECG), electroencephalogram (EEG) and electromyogram (EMG). They used the Arduino UNO board and Bluetooth module to construct the wireless transmission. J. F. Cheng et al. [10] fabricated the chloride ion sensing devices and Bluetooth wireless measurement system. The wireless sensing devices were used to measure response voltage from 10^{-4} to 1 M NaCl solutions.

Our research group [11] investigated the sensing characteristic of the RuO_2 chloride ion sensing devices with voltage-time (V-T) measurement system and wireless remote control platform. Our research group [12] integrated the current-voltage (I-V) measurement system with the three Keithley 236 semiconductor parameter analyzers. They investigated the sensing characteristic of the wireless chloride sensor. V. A. T. Dam et al. [13] prepared sweat sensing device by screen printing system. They integrated the sweat sensing device with high impedance voltmeter (Keithley 617A). They investigated the hysteresis effects and detected chloride ion concentrations of the sweat. K. Smettem et al. [14] prepared the wireless chloride ion sensing devices by the screen printed technology, central microcontroller (MCU), real-time clocks (RTC), GPS module and IEEE 802. 15. 4 standard. Their wireless chloride ion sensing devices applied to monitor the water quality of the stream. Y. Abbas et al. [15] investigated the sensing characteristic of near-field-inductive coupling (NFC) of sensing devices and readout coils with the Ag/AgCl electrode. The capacitance variation were from 180 to 200 pF when the chloride ion concentrations from 0.01 to 0.2 M, respectively. J. Jung et al. [16] used the three-electrode electrochemical amperometric analyzer to investigate the sensing characteristics from 1 mM to 100 mM of ruthenium (III) chloride (Ru III). The three electrodes, current-to-voltage (I/V) measurement, Bluetooth 4.0 module (WT-12, Bluegiga, Espoo, Finland) and notebook computer were used to set up the three-electrode electrochemical amperometric analyzer. They repaired the silver (Ag) counter electrode and silver-silver chloride (Ag/AgCl) reference and indium tin oxide (ITO) working electrode on the ITO glass.

R. Yue et al. [17] integrated solar power supply with wireless sensor system. The wireless sensors were used to detect pH, turbidity and oxygen density. W. Dang et al. [18] used the graphite-polyurethane composite to prepare the pH sensor. The sensitivity of the RFID wireless pH sensor was 11.13 mV/pH. The wireless pH sensor was used to detect sweat pH variation. L. Lu et al. [19] investigated the human gastrointestinal (GI) physiological information by wireless capsule. They repaired the pH micro sensor, pressure micro sensor, temperature micro sensor and radio frequency (RF) transceiver in the wireless capsule. The wireless capsule was used to real test for the animal (pig) and three healthy volunteers. They obtained more data for pH, temperature and pressure by the wireless capsule.

R. Rahimi et al. [20] prepared the transparent pH sensor and near field communication (NFC) wireless measurements for wound infection monitoring. The NFC wireless transparent pH sensor was investigated the hysteresis effect, drift effect from pH 4 to pH 10. R. A. Croce et al. [21] used the winding 125 μm platinum (Pt) and silver wire to fabricate the coil-type electrochemical pH sensing devices. They used the coil-type pH sensing devices, Ag/AgCl reference electrode, saturated calomel electrode (SCE), Bluetooth transceiver and computer to investigate the pH detection. B. Zhou et al. [22] repaired the optical fiber sensor to measure the pH solutions. The pH experiment data was transferred to computer by Zigbee wireless network.

Our research group studied the wireless sensing system of XBee module with sensing devices [23-25]. The wireless sensing system was used to investigate the sensitivities for many kinds of the chemical substances, such as pH [23], glucose [23-25] and lactate [25]. The advantages of the wireless sensing system of XBee module were high stability, lost cost, portable device, easy operation, rapid detection and real-time monitor [23]. P. Abouzar et al. [26] used the Zigbee wireless system and measurement to investigate the sensing characteristics of wireless sensor networks for precision agriculture. N. A. Cloete et al. [27] integrated the flow sensing device, temperature sensing device, conductivity sensing device, pH sensing device with the

XBee wireless module. They used the XBee wireless water quality monitoring system to investigate the sensing characteristic for real time water quality detection.

In order to develop the real-time monitor of chloride ion detection, therefore wireless sensing measurement of XBee integrated with the arrayed flexible RuO_2/GO chloride ion sensor to investigate the selectivity coefficients, hysteresis effect, and dynamic microfluidic measurement of the arrayed flexible RuO_2/GO chloride ion sensor.

2. Materials and Methods

2.1. Materials

The polyethylene terephthalate (PET) was purchased from Zencatec Corporation (Taiwan). PET sheet was flexible and light weight, which was the substrate of the chloride sensing device. Ruthenium target (Ru, 99.95 wt%) was purchased from Ultimate Materials Technology Co., Ltd. (Taiwan), which was used to deposit the membrane of ruthenium dioxide (RuO_2) by the radio frequency (R. F.) sputtering system.

The silver paste and epoxy thermosetting polymer (product no. JA643) were purchased from Advanced Electronic Material Inc. (Taiwan) and Everwide Chemical Co., Ltd. (Taiwan), respectively. The applications of the silver paste and epoxy thermosetting polymer were conductive wire and insulation layer, respectively.

The poly(vinyl chloride) (PVC), bis (2-ethylhexyl) sebacate (DOS) solution (95%), chloride ionophore III (ETH9033) and tridodecylmethy-lammonium chloride (TDDMACl) were purchased from Sigma-Alorich Co., Ltd. (USA). The tetrahydrofuran (THF) solution were purchased from Nan Ya Plastics Co., Ltd. (Taiwan). THF solution, PVC powder, DOS solution, ETH9033 powder and TDDMACl powder were used to prepare the chloride ion mixture. The sodium cloride (NaCl) was purchased from Avantor Performance Materials, Inc. (USA), it was used to prepare the diffferent chloride ion concentrations from 10^{-5} M to 1 M NaCl solutions.

XBee router and XBee coordinator were purchased from Digi International Inc. (USA). XBee router, XBee coordinator and Arduino Mega 2560 microcontroller board were used to transfer the experiment data to computer.

2.2. Fabrication of the arrayed flexible RuO_2/GO chloride ion sensor

The producing process of the flexible arrayed RuO_2/GO chloride sensor was shown in Figure 1. The preparation method of the flexible arrayed RuO_2/GO chloride sensor was followed the Refs. [28-31]. Then, we dropped 2 μL of the chloride ion mixture on the sensing windows of the GO/RuO_2 sensing devices, respectively. The GO/RuO_2 chloride ion sensors were dried at room temperature (25 ℃) during 4 days.

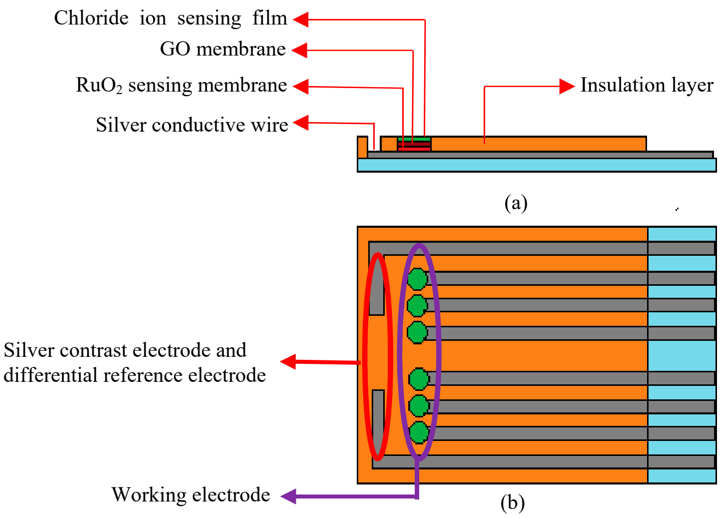

Figure 1. The producing process of the flexible arrayed RuO_2/GO chloride sensor [28-31]. (a) The diagrammatic sketch of the different sensing structures for the flexible arrayed RuO_2/GO chloride sensor (b) The schematic diagram of the silver contrast electrode, differential reference electrode and working electrode.

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2.3. Wireless sensing system

The schematic diagram of wireless sensing system of XBee module was shown in Figure 2. The real picture of the wireless sensing system of XBee module was shown in Figure 3 [23-25]. The wireless sensing system of the XBee module contained computer, XBee coordinator, power supply, wireless sensing measurement and flexible arrayed RuO_2/GO chloride ion sensor, the wireless sensing measurement of the contained 6-channel readout circuit device, XBee router, Arduino Mega 2560 and analysis software (model: LabVIEW 2012). The six instrumentation amplifiers (AD623) were used to construct the 6-channel readout circuit device. We used the wireless sensing system to measure response voltages with different chloride ion concentrations from 10^{-5} M to 1 M NaCl solutions. The sensitivity, selectivity coefficients and hysteresis voltage were investigated by the wireless sensing system. From Figure 4, the wireless sensing system, microfluidic device and syringe pump were used to measure response voltage and investigate real-time monitor of chloride ion detection with different flow rates from 5 μL/min to 40 μL/min.

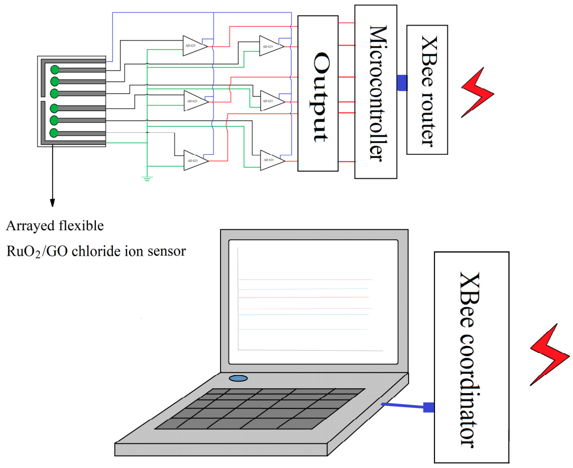

Figure 2. The schematic diagram of wireless sensing system with the flexible arrayed RuO_2/ GO chloride ion sensor [23-25].

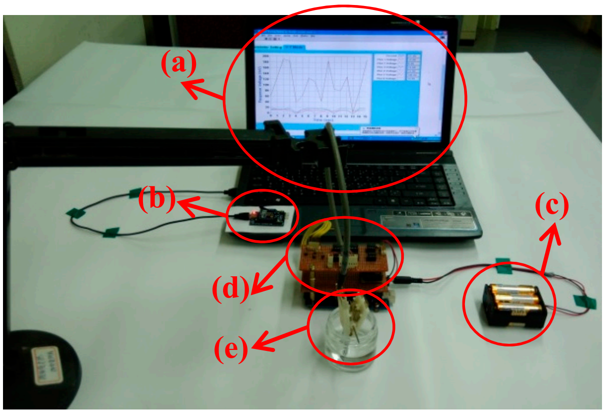

Figure 3. The wireless sensing system with the flexible arrayed RuO_2/ GO chloride ion sensor. (a) Computer, (b) XBee coordinator, (c) power supply (d) wireless sensing measurement (XBee router) and (e) flexible arrayed RuO_2/GO chloride ion sensor [23-25].

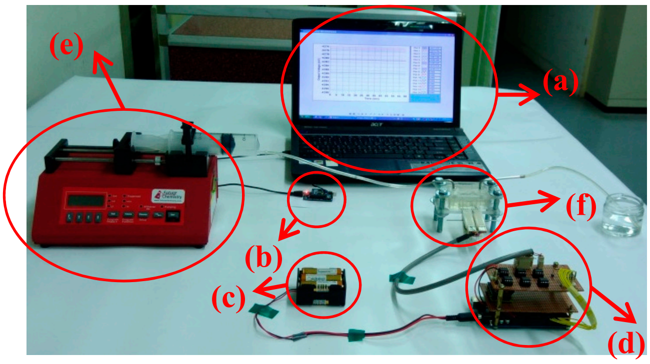

Figure 4. The wireless sensing system with microfluid dymanic system. (a) computer, (b) XBee coordinator, (c) power supply (d) wireless sensing measurement (XBee router), (e) syringe pump and (f) microfluidic device [23-25].

3. Results and Discussion

3.1. Sensitivity in the static state

We investigated the sensitivity and linearity of chloride ion sensing device with wireless sensing system. From Figure 5, the sensitivity and linearity were 91.6 mV/pCl and 0.988, respectively. We used the instrumentation amplifiers (AD623) with 2 times gain and set the parameters in LabVIEW, which amplified the voltage value of the flexible arrayed RuO_2/ GO chloride ion sensor. The sensitivity comparisons of the chloride ion sensing devices were shown in Table 1. The wireless transmission technology was obtained the RFID [7], Bluetooth [10]. S. Zhou et al. [7] prepare the wireless chloride ion sensor. The sensitivity of wireless chloride ion sensor was 47.83 mV/pCl.

Our group used the voltage-time measurement system and the chloride ion devices to measure response voltages and obtained the sensitivity of chloride ion devices [28-30]. The voltage-time measurement system (V-T measurement system) was not wireless data transmission. Therefore, the detection distance of the chloride ion sensing device with voltage-time measurement system was shorter than chloride ion sensing device with wireless sensing system.

3.2. Sensitivity in the dynamic microfluid

The sensitivity variations of the arrayed flexible chloride sensor were integrated by the microfluidic device, syringe pump and wireless sensing system. From Figure 6, the sensitivities of the wireless chloride ion sensor were increased when the flow rates increasing from 5 μL/min to 40 μL/min. The boundary layer was produced by friction and viscous force, and the boundary layer was between chloride membrane and molecular electrolyte [28-30]. The boundary layer thickness was decreased when flow rate increasing. Therefore, the sensitivities of the wireless chloride ion sensor were increased when the flow rates increasing. The best average sensitivity and linearity were 273.1 mV/pCl and 0.978 at 35 μL/min, respectively. When the flow rate was over 35 μL/min, the sensitivities were decreased. The flexible arrayed RuO_2/GO chloride ion sensor with the wireless sensing system was quick response and convenient operation, which could be applied to monitor chloride ion concentrations in real time.

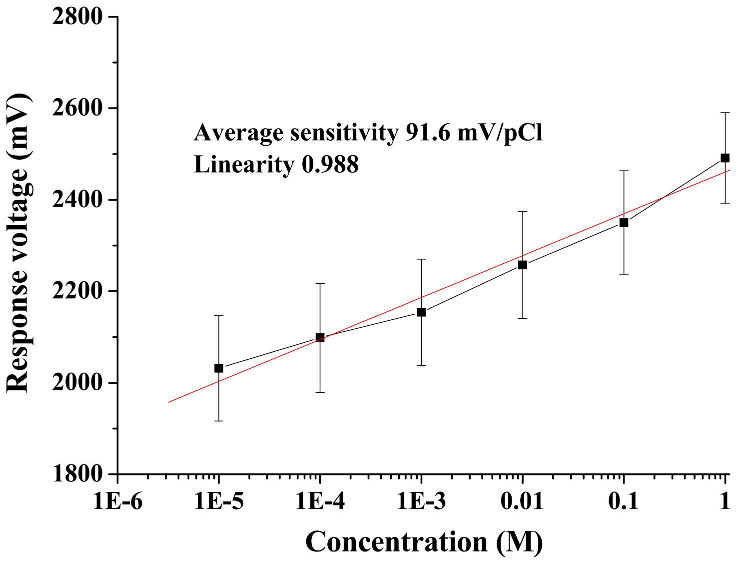

Figure 5. The curve of the response voltage versus concentration for the flexible arrayed RuO_2/GO chloride ion sensor integrated in the wireless sensing system between 10^{-5} M and 1 M NaCl solutions.

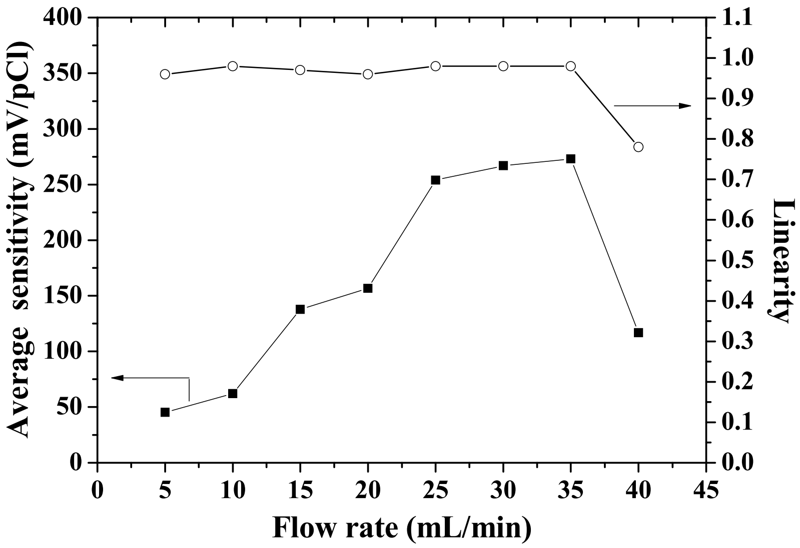

Figure 6. The average sensitivity and linearity of the flexible arrayed RuO_2/GO chloride ion sensor with the wireless sensing system at different flow rates from 5 µL/min to 40 µL/min.

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Table 1. The comparisons of the chloride ion sensing devices with other literatures in different chloride ion concentrations.

Sensing electrode	Measurement system	Test solutons (M)	Average sensitivity (mV/pCl)	Ref.
RuO_2/0.01 wt% GO/Chloride ion selective membrane	XBee wireless V-T sensing system	10^{-5} to 1 NaCl solutions	91.6	In this study
Ag/AgCl electrode and reference electrode	RFID communication protocol	10^{-4} to 1 NaCl solutions	47.8	[7] 2017
ITO glass /SnO_2/ Chloride ion selective membrane	Bluetooth wireless measurement system	10^{-4} to 1 NaCl solutions	51.4	[10] 2012
Silicon substrate/ RuO_2/ chlorine ion selective membrane	Voltage-time (V-T) measurement system and wireless remote control platform	0 to 1.34×10^{-4} NaClO solutions	1.8 mV/ppm	[11] 2012
Silicon wafer /RuO_2/ chlorine ion selective membrane	Current-voltage (I-V) measurement system with three Keithley 236 semiconductor parameter analyzers	10^{-4} to 1 NaCl solutions	59.0	[12] 2012
Polyethylene terephthalate /dupont 5876 AgCl conducting paste/ dupont 8153 insulating paste/ pHEMA hydrogel layer	High impedance voltmeter (Keithley 617A)	10^{-3} to 3 NaCl solutions	58.0	[13] 2016
Polyethylene terephthalate /RuO_2/Chloride ion selective membrane	V-T measurement system	10^{-5} to 1 NaCl solutions	25.1	[28] 2016
Polyethylene terephthalate /RuO_2/0.01 wt% GO/Chloride ion selective membrane	V-T measurement system	10^{-5} to 1 NaCl solutions	44.5	[29] 2018
Polyethylene terephthalate /RuO_2/Chloride ion selective membrane	V-T measurement system	10^{-4} to 1 NaCl solutions	26.2	[31] 2012

3.3. Investigation of the hysteresis voltage

The response voltages of the flexible arrayed RuO_2/GO sensors were measured by the V-T measuring system with different measurement cycles of chloride ion concentrations, which from 10^{-3} M to 1 M NaCl solutions by XBee wireless modules. The experimential results were shown in Figure 7 and Figure 8. The hysteresis voltages were 50.14 mV and 36.71 mV during the cycles of 1 M→ 10^{-1} M→ 1 M→ 10^{-3} M→ 1 M and 1 M→ 10^{-3} M→ 1 M→ 10^{-1} M→ 1 M, respectively.

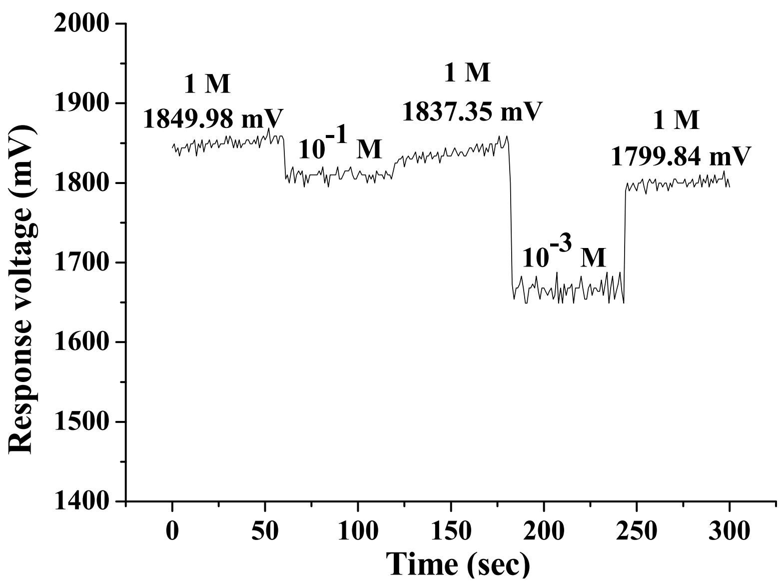

Figure 7. The response voltages of the flexible arrayed RuO_2/ GO chloride ion sensor in the NaCl solutions during the cycle of 1 M→ 10^{-1} M→ 1 M→ 10^{-3} M→ 1 M.

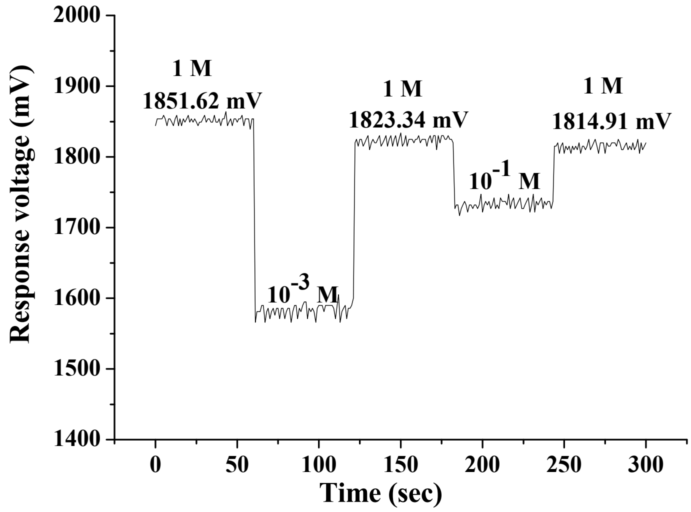

Figure 8. The response voltages of the flexible arrayed RuO_2/ GO chloride ion sensor in the NaCl solutions during the cycle of 1 M→ 10^{-3} M→ 1 M→ 10^{-1} M→ 1 M.

3.4. Investigation of the selectivity coefficients

In this study, we checked the chloride ion selectivity of chloride ion sensing devices with wireless sensing system. We prepared the 30 mL different chloride concentrations from 10^{-5} M to 1 M NaCl solutions as the major ion A, respectively. The other interfering ions B contained 10^{-3} M ClO^- solution, 10^{-3} M ClO_4^- solution, 10^{-3} M NO_3^- solution and 10^{-3} M I^- solution. The 10^{-5} M to 1 M NaCl solutions were based on a fixed major ion concentration. The selectivity coefficient was obtained from Equations (1) and (2):

$$\Delta\, a_A = a_A' - a_A \qquad (1)$$

$$\mathrm{K}_{A,B}^{pot} = \frac{\Delta a_A}{a_B} \qquad (2)$$

Where $\Delta\, a_A$ was the increasing quantity of the major ion; a_A' was the equal concentration when the interfering ion reaches its maximum value; a_A was the concentration of the background solution; a_B was the maximum concentration of the interfering ion; and $\mathrm{K}_{A,B}^{pot}$ was the selectivity coefficient of matched potential method (MPM) [32]. J. F. Cheng et al. [33] calculated the $K_{Cl^-/j}^{pot}$ values of the HPO_4^{2-}, F^-, SO_4^-, Br^-, I^- ions for chloride ion selectivity by MPM. They found the $K_{Cl^-/j}^{pot}$ value sequences were $I^- > Br^- > SO_4^- > F^- > (HPO_4^{2-})$. K. G. Kumar et al. [34] investigated the $K_{Cl^-/j}^{pot}$ values of the Zn^{2+}, Fe^{3+}, Cu^{2+}, Na^+, Mn^{2+}, Co^{2+}, K^+, Ca^{2+}, NO_3^-, Br^-, NO_2^-, CH_3COO^- and SO_4^{2-} ions for chloride ion selectivity. We used the MPM to investigate the chloride selectivity coefficients in this study. From Table 2, the selectivity coefficients of the ClO^- ion, ClO_4^- ion, NO_3^- ion and I^- ion with Cl^- ion were 5.0×10^{-2}, 1.0×10^{-1}, 5.9×10^{-3} and 5.6×10^{-1}, respectively. We found the $\mathrm{K}_{Cl^-/j}^{pot}$ value sequences were $I^- > ClO_4^- > ClO^- > NO_3^-$. According to the experimental results, the chloride ion sensor with the wireless sensing system had good chloride ion selectivity.

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Table 2. The selectivity coefficients of the chloride ion sensor with the wireless sensing system under four different interfering ions.

Interfering ions	Selectivity coefficients log $K^{pot}_{Cl^-/j}$
ClO^-	5.0×10^{-2}
ClO_4^-	1.0×10^{-1}
NO_3^-	5.9×10^{-3}
I^-	5.6×10^{-1}

3.5. Chloride ion detection of the tap water

Water chlorination of the water treatment plant was an important process for drinking water. The sterilization capabilities were from free chlorine. The free chlorine were contained the hypochlorous acid (HClO) and hypochlorite ion (ClO^-) concentrations [7, 34]. We used the wireless sensing system to investigate the sensitivity and linearity for different NaClO solutions from 10^{-6} M to 1 M. From Figure 9, the average sensitivity and linearity were 28.92 mV/pCl and 0.982, respectively, and it could be found the flexible arrayed RuO_2/GO chloride ion sensor integrated in wireless sensing system had good stability for chloride ion concentration. From Figure 10, the average response voltage was 1978.34 ± 9.89 mV in tap water with XBee wireless measurement system. The chloride concentration ranges of the tap water was between were between 10^{-5} M and 10^{-4} M.

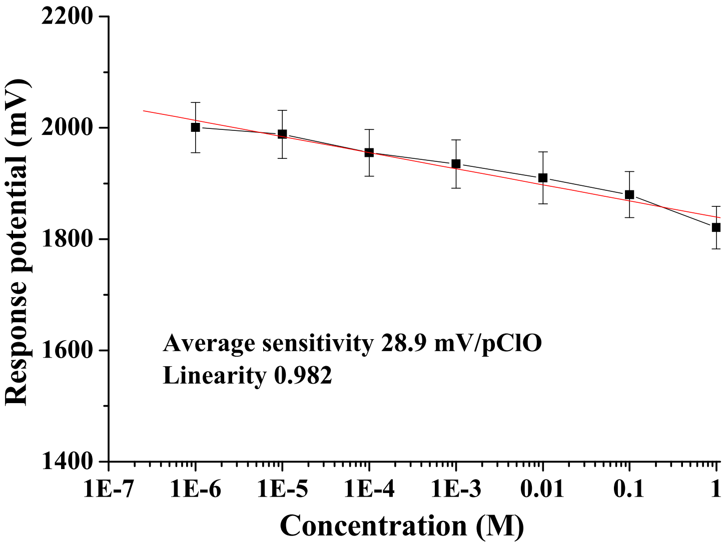

Figure 9. The curve of the response voltages versus concentration for the flexible arrayed RuO_2/GO chloridne ion sensor with the wireless sensing system in the 10^{-6} M and 1 M NaClO solutions.

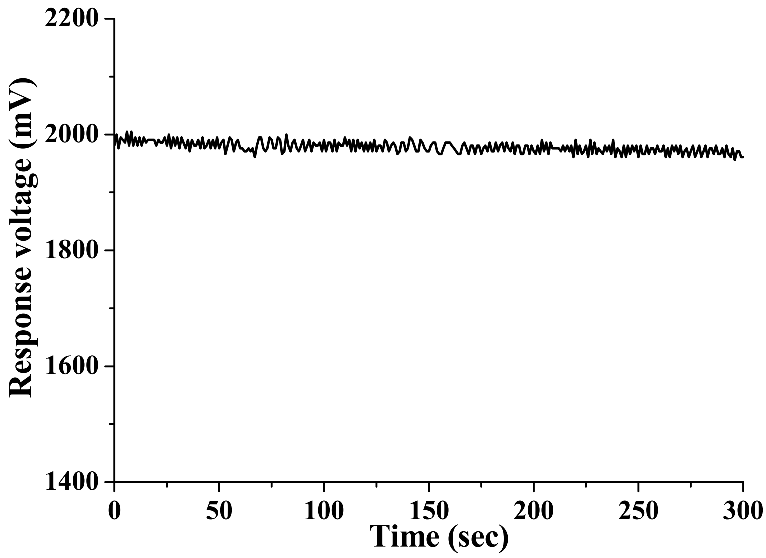

Figure 10. The response voltage curve of flexible arrayed RuO_2/GO chloride ion sensor with the wireless sensing system in the tap water.

4. Conclusions

The sensitivity and linearity of the chloride ion sensor with the wireless sensing system were 91.6 mV/pCl and 0.988, respectively. The sensitivity and linearity of the flexible arrayed RuO_2/GO chloride ion sensor with the XBee wireless sensing system and dynamic microfluidic were 273.1 mV/pCl and 0.978 at 35 μL/min, respectively. The hysteresis voltages were 50.14 mV and 36.71 mV during the cycles of 1 M→ 10^{-1} M→ 1 M→ 10^{-3} M→ 1 M and 1 M→ 10^{-3} M→ 1 M→ 10^{-1} M→ 1 M, respectively. The selectivity coefficients of the ClO^- ion, ClO_4^- ion, NO_3^- ion and I^- ion with Cl^- ion were 5.0×10^{-2}, 1.0×10^{-1}, 5.9×10^{-3} and 5.6×10^{-1}, respectively. The above experimental results had proved that the wireless sensing system with the flexible arrayed RuO_2/GO chloride ion sensor had good selectivity and good stability for chloride ion detection.

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Author Contributions: Shi-Chang Tseng, Jung-Chuan Chou and Tong-Yu Wu conceived and designed the experiments; Tong-Yu Wu and Si-Hong Lin performed the experiments; Cian-Yi Wu and Yi-Hung Liao analyzed the data; You-Xiang Wu and Chih-Hsien Lai checked the analyzed data; Yu-Hsun Nien and Jung-Chuan Chou contributed reagents/materials/analysis tools; Tong-Yu Wu wrote the paper; Jung-Chuan Chou revised the paper.

Conflicts of Interest: The authors declare no conflict of interest.

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