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RESEARCH ARTICLE

Design, Simulation and Fabrication of a Fully Solid State Electrochemical NO_2 Gas Sensor Using Nafion/Pt Electrode

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Abstract

A fully solid-state electrochemical NO_2 gas sensor based on Pt/Nafion electrode has been designed, simulated and fabricated. The simulation is performed by 3D model and it provides more correct results than 2D model of previous report. In this paper the sensor with three-electrode configuration (i.e. WE-working electrode, CE-counter electrode, RE-reference electrode) uses platinum nano-powder as electrode material and Nafion membrane as polymer electrolyte. Simulation and experiment are mainly focused on analysis of current characteristics with the bias voltage and ambient temperature, relative humidity, and pretreatment solvent for the sensor. For pretreatment solvents, we use 1M H_2SO_4, 2M KOH and 2M KCl aqueous solution. Simulations carried out by using COMSOL Multi-physic software. We considered the sensing characteristics to the increase of bias voltage in the range of 0.1-1.5V, operating temperature in the range of 20-80℃, relative humidity in the range of 20-100%, and the different pretreatment solvent for the sensor. We also fabricated and made an experiment for NO_2 gas sensor. The results of simulation are compared with that one of experiment, they are almost near the same.

Keywords: Electrochemical NO_2 gas sensor; Pretreatment solvent; Nafion

Introduction

Electrochemical sensors are worked using redox reaction of specific gases at the electrode/electrolyte boundary to detect the gas concentration.

A lot of investigation related with the electrochemical sensors has been conducted for a long time. Now the direction of research is aimed to get a higher selectivity, sensitivity, reproducibility, repeatability and fast response time of the sensor [1, 4].

The most important thing for a design of gas sensor is the option of electrode and electrolyte material. Recently, there are many new types of electrode, including nanopaticle-modified electrodes such as gold or copper nanoparticles modified.

As electrolyte material, there are aqueous solution such as H_2SO_4, KOH, KCl etc, inorganic conducting material such as YSZ (yttria saturated zirconium) or NASICON (Na superionic conductor) [37, 38], room temperature ionic liquid electrolyte such as [C_4mpy][NTf_2][2, 7, 19, 27, 28, 29, 36], and there is the most attractive material Nafion(copolymer of polytetrafluoroethlyene and polyfluorosulphonyl-vinyl-ether) membrane[8, 11, 35, 39].

There are mainly three type of sensor depends on the electrolyte state:

First, liquid-electrolyte based sensor, which uses only aqueous solution;

Second, semisolid electrolyte based sensor, which uses both aqueous and polymer (such as Nafion);

Third, fully solid-state electrolyte based sensor, which uses inorganic electrolyte or only polymer electrolyte.

Liquid-electrolyte sensor has a serious problem such as deterioration of sensitivity due to leakage and decrease of electrolyte solution from the sensor. For these reason, solid-state gas sensors are developed to avoid above disadvantages [14, 21, 22]. Although the sensors with inorganic conducting material use solid-state electrolyte, but they have high operating temperature of a few hundred degrees and bad gas adsorption/desorption on the electrodes. Therefore, much attention is focused on the development of electrochemical sensor using Nafion, a good proton conductor.

Due to Nafion has both branch of hydrophilic and hydrophobic within its molecular chains, it has higher ion conductivity, gas permeability and mechanical strength.

A research group in a country fabricated the amperometric H_2S gas sensor by using Nafion membrane pretreated with H_2SO_4[16].

For this type of sensor Nafion plays a role of electrolyte as a proton conductor, but water content in membrane is significantly affected conductivity of it, so it has an effect by relative humidity of operating condition. It can be operated in the range of 32~96% RH.

However, the research for a fully solid state gas sensor continues to minimize the sensor and to get a sensor without leakage of electrolyte.

NO_2 gas is a hazardous toxic gas. To measure the concentration of NO_2 gas is very important problem in the environmental protection [9, 10].

Therefore, we have designed and fabricated the fully solid-state electrochemical NO_2 gas sensor in this paper.

The mechanism of the NO_2 redox reaction at the three-phase boundary of electrode/electrolyte/gas is known as following.

There is to be a reduction reaction of NO_2 on the working electrode.

$$\mathrm{NO_2 + 2H^+ + 2e^- \rightarrow NO + H_2O} \quad (1)$$

Simultaneously, due to the oxidation of water, two electrons are generated on the counter electrode.

$$\mathrm{H_2O \rightarrow 2H^+ + 2e^- + \tfrac{1}{2}O_2} \quad (2)$$

The total reaction through the sensor is following.

$$\mathrm{NO_2^- \rightarrow NO + \tfrac{1}{2}O_2} \quad (3)$$

As an electrode material of electrochemical gas sensor, novel metals are commonly used, but carbon nanomaterials such as carbon nano-powder, CNT and graphene are also widely used in recent years.

Youqin Altsmid et. al., suggested the $Cu(OH)_2$/MWCNT/carbon nanocomposite electrode to sense the nitrite ion [23].

The results concerned electrochemical NO_2 gas sensor based on Pt electrode and room temperature ionic electrolyte was recorded by Jakub Altsmid et al., [9].

Disposable paper-based analytical device using a copper nanoparticles-modified screen-printed graphene electrode was also used for the electrochemical NO_x gas [24].

It has proved that copper nanoparticles-modified screen-printed graphene electrode can improve the sensitivity rather than use of bare graphene screen-printed electrode through the cyclic voltammetric and differential cyclic voltammetric method.

The example for NO_2 gas sensing with Nafion membrane is reported by Kuo-Chuan Ho.et al., [20]. They designed an amperometric NO_2 gas sensor based on Pt/Nafion electrode and the electrode was manufactured by impregnation-reduction method. They've obtained 0.16μA/ppm sensitivity in 0-100ppm of NO_2 concentration. And many researchers studied electrochemical sensor using Nafion membrane [8, 11, 12, 16, 18, 20, 25, 35].

Recently, in all the scientific field, development of elements is significantly increased on their speed with the simulation using computer. Among them, there are physical field calculation using COMSOL Multi-physics software a lot and chemical reaction are modeled through the software [5, 13, 15, 26, 31-34].

Simulations and experiment results for a electrochemical NO_2 gas sensor with 2-electrode and 3-electrode were obtained and compared each other by Umesh Yadav et. al [15]. They considered the current characteristics with 2-D model in terms of size of cells, electrolyte concentration, and electrode-size. They used H_2SO_4 aqueous solution as an electrolyte. And they also fabricated the sensor with 2-electrode system and had a testing for NO_2 gas.

We've designed and simulated a fully solid-state electrochemical NO_2 gas snesor with 3-electrode system by using COMSOL Multiphysics, engineering analysis software widely used. The model of sensor used in simulation was drawn 3-dimensional. As the simulation results, we've obtained the current density of the electrolyte. And then we've performed an experiment for the testing of suggested sensor.

In other words, we've carried out the simulation concerned with bias voltage, operating temperature and relative humidity, and pre-treated solvents for H_2SO_4, KOH, KCl in the electrochemical NO_2 sensor based on polymer electrolyte, Nafion membrane. And then we've proved simulation results through the testing.

The results in both simulation and experiment are compared each other.

Simulation

We've simulated the current characteristics with electrode structure and geometric changes of electrochemical gas sensor in our previous works [40]. In this paper we deal with steady-state analysis of a NO_2 gas sensor for a detection in the low concentration of the gas. In terms of operating condition for the sensor, we are considered with some changes in the bias voltage, temperature and relative humidity, and the pretreatment solvents. Simulation uses the Electrochemistry Module of COMSOL Multiphysics. 3-D Models are used in the simulation and it improves the accuracy of results than the use of 2-D Models.

Figure 1 Shows the 3-D Model of the sensor that are used in the simulation

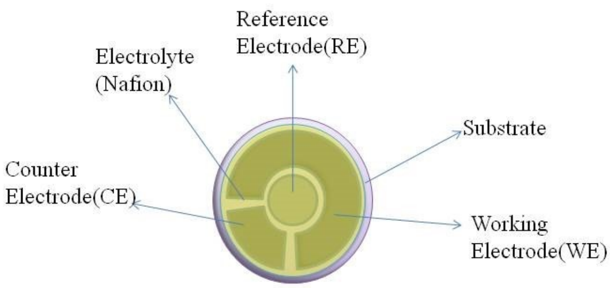


Figure 1: Electrode structure of NO_2 gas sensor using solid-state electrolyte.

The model of sensor in simulation mainly consists of substrate, working electrode, counter electrode, reference electrode and electrolyte-Nafion membrane.

The substrate is 4mm in radius, the electrodes are $15mm^2$, $5mm^2$ and $10mm^2$ in area respectively. And electrode thickness is 0.05mm for all the electrodes and the thickness of electrolyte is set in 170μm, that is similar with Nafion 117 membrane

For the simulation, a bias voltage in the range of 0.1-1.5V with an interval 0.1V is applied to working electrode versus reference electrode, containing the redox potential of NO_2(0.9V).

It is set in temperature of 20-80℃ with an interval 10℃ and relative humidity 20-100% RH with an interval 20%.

Simulations depending on the different pretreatment solvents are performed by setting conductivity of pretreated Nafion membrane with that each one respectively.

The results are discussed in Section 4.

Experiments

Fabrication of sensors

To prove the simulation results, the sensor is fabricated and tested for a NO_2 gas sensing in the given condition from the simulation.

As the electrode material, Pt catalyst powder(15H-1805, purchased from www.sryeo.com) is used and Nafion solution(Nafion^{TM} 5 wt.%, 1100EW, Du Pont) is used for the solid state electrolyte. H_2SO_4, KOH, KCl that mentioned above are used as the pretreatment solvents for sulphonation and water absorption of the membrane. Alumina ceramic sheet is used as the substrate, it is cleaned through the acid-washing and ultrasonic washing before it is used. For the fabrication of the sensor, the suspension (Pt powder 90wt.%) of electrode material was prepared from the mixing with and stirring between Pt powder and butyl di-glycol solution.

The pattern mask which is drawn electrodes is made and then electrodes (WE, CE and RE) are coated on the substrate by screen printing using the mask.

Then sintering of it was carried out for an hour in a furnace at 850℃, with the air condition.

After the sintering, about 10μL Nafion solution (5 wt.%) is coated onto the electrodes with drop casting with micropipette. Then it is heat-treated at 80℃ for 30 min. Finally, it is pretreated with 1M H_2SO_4, 2M KOH and 2M KCl solution for 24h.

Measurement Setup

The measurement setup for a gas sensor is shown Figure 2. Standard NO_2 gas(105ppm) and pure air are put into the gas chamber to form a sensing condition. The sensor is placed in the gas chamber.

A bias voltage of 0.1-1.2V is scanned between the working electrode and reference electrode and the scan rate is 0.1V. The redox current signal of NO_2 is measured by a voltammetric device MEC 16.

During the testing of current response to the bias voltage, the operating condition is kept in temperature 25℃ and relative humidity 60% RH.

To consider the current characteristics with the variation of temperature and relative humidity, the temperature is varied from 20℃ to 80℃ and relative humidity from 20% RH to 100% RH respectively. Gas chamber has a thermometer and a hygrometer, humidity in the chamber is maintained by adding water vapor.

The testing results are discussed in the following sections.

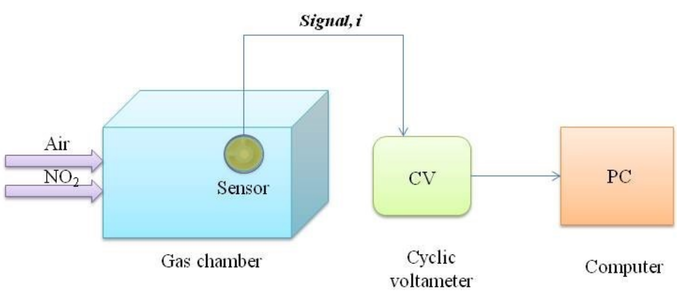


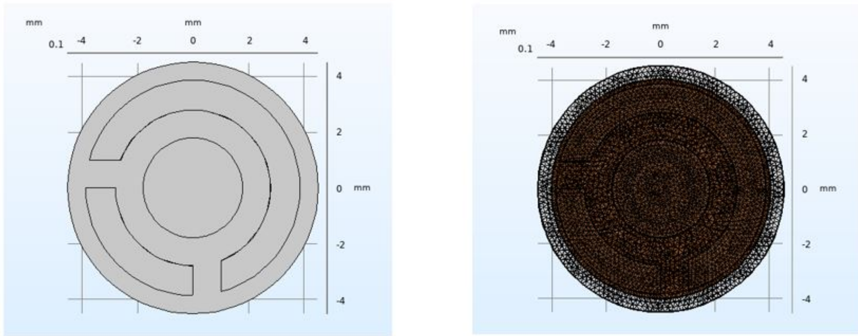
Figure 2: Measurement setup

Results and Discussion

Simulation Results

In this section, there are discussions of results obtained from the simulation using Electrochemistry module of COMSOL Multiphysics.

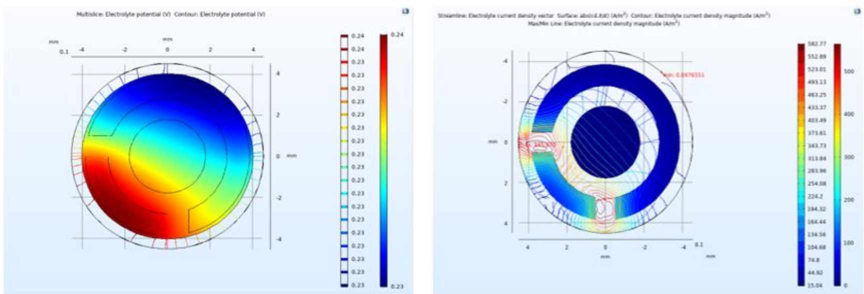
First of all, we've considered the current characteristics to the change of bias voltage. Figure 3 shows the 3D diagram and meshed state of the sensor in the working interface of the simulation software. The number of mesh elements was 33, 651 domains, 16, 629boundary and 1, 165 edge elements.



(a) (b)

Figure 3: The 3D diagram(a) and the meshed state(b) of sensor

The 3D simulation results with the designed sensor are shown Figure 4.



(a) (b)

Figure 4: Simulation results showing the potential and current density of the electrolyte (a) the potential of the electrolyte, (b) the current density of the electrolyte.

Figure 4 (a) is a picture, drawing the potential of the electrolyte in the sensor.

The potential at the side of counter electrode is higher than the others, which is due to the higher concentration of protons, is generated from the oxidation reaction of water on it. Simultaneously the NO_2 gas is reduced by receiving proton and electron at the working electrode. Protons are transmitted from the counter electrode to working electrode through the solid state electrolyte and electrons are conducted through external circuit.

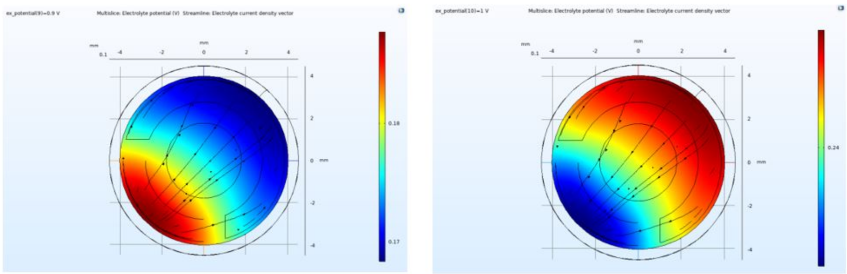
And Figure 4 (b) shows the current direction and the contour plots of current density of the sensor.

From the current density given by the simulation results, we can calculate the current using Eq. (4).

$$I = A * j \quad (4)$$

Where A is an area of the cross section of electrode.

Figure 5 shows the reduction and oxidation state of NO_2 gas on the working electrode under the different bias voltage applying.



(a) (b)

Figure 5: The reduction state and oxidation state of NO2 gas on the working electrode (a)- reduction reaction state, (b)- oxidation reaction state.

As shown in Figure5, the color of the electrolyte potential for the side of working electrode changed from blue to red at 0.9V and 1.0V applying.

The standard redox potential of NO_2 gas is 0.99V, therefore we can understand it will be oxidized below 0.99V (bias voltage), but it will be reduced above that point. Alternating color in the picture describes the redox mechanism. In other words, during the oxidation reaction on the working electrode, there will be reduction reaction on the counter electrode at the same time.

The Current-Voltage characteristics of the sensor is shown figure 6.

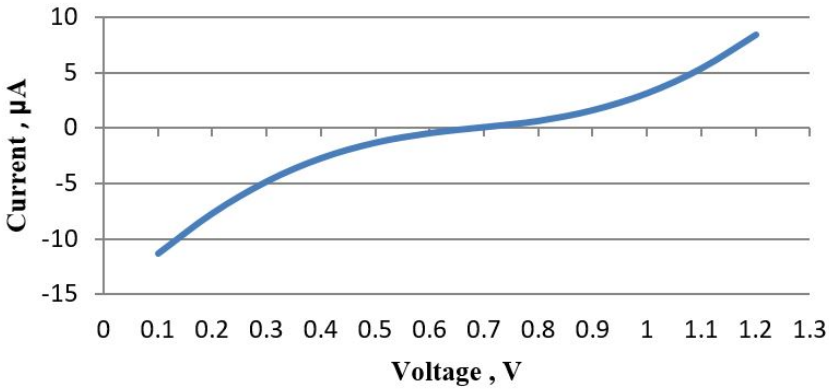


Figure 6: Current-Voltage characteristics of the sensor(simulation)

As shown figure 6, sensing current increases as the bias voltage increases.

I-V characteristics reflect the stability of the sensor with the increase or decrease of the bias voltage during it operates.

As shown figure 6, the current plot presented a gentle slope from 0.6V to 1.3V, therefore we can get a stable current with applying voltage within above-mentioned range.

Simulation was also performed to consider the current with the change of operating temperature. The temperature(T) was increased by 5℃ at a time in the interval 20~80℃.

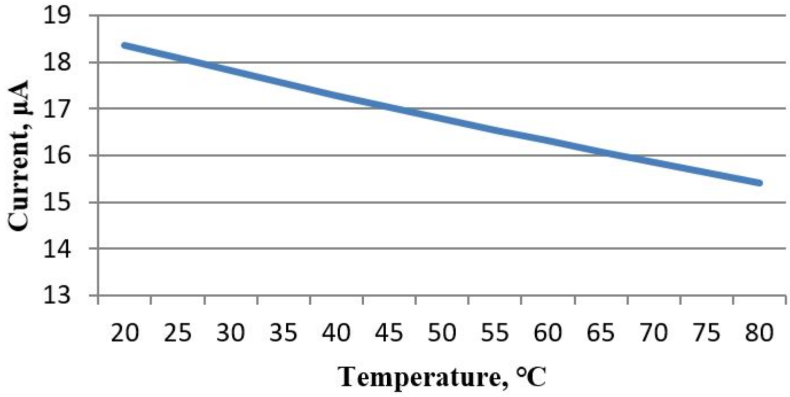


Figure 7: Simulation curve of the current characteristics of the sensor (NO_2 concentration 20 ppm, bias voltage 0.9 V) with temperature variation.

The plot shows that the sensing current reduces against the increase of the temperature.

It is well known that the proton conductivity in the Nafion membrane is varied depending water content within itself. For the application, it is very important to consider the humidity-dependence of the sensor.

From the previous work [16], Nafion membrane absorbs the water up to 25 wt.% under the 100% RH condition and shows variation in the proton conductivity.

Therefore, simulation was carried out in terms of consideration for the humidity from 20%RH to 100%RH.

Table 1 lists the water content and conductivity of Nafion membrane depending on the relative humidity.

Table 1: Water content and conductivity of Nafion membrane depending on the relative humidity [17].

Relative Humidity (RH, %)	Water content (wt, %)	Conductivity (S/m)
20	2.5	0.05
40	5	0.1
60	7	0.7
80	12	1.2
100	22	3.1

Figure 8 shows the simulation results with the humidity variation.

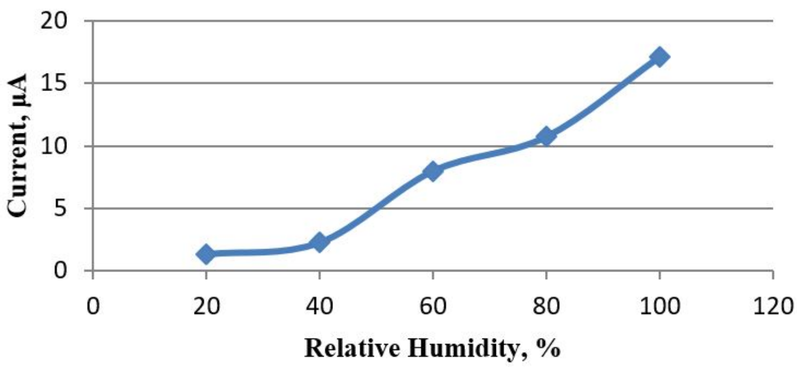


Figure 8: Current-Voltage characteristics for the humidity variation (NO_2 gas concentration 20 ppm, bias voltage=0.9V)

We can see the upward tendency with higher humidity condition from the figure. It is because the conductivity increases depending on the water content in the membrane. Higher water content means the more hydrophilic region of the membrane and it also means protons can be more easily conducted through the membrane. But it exceeds the specific limit for the water content it can cause the inverse effect against the proton conductivity. It can be explained by limitation of gas permeation due to the decrease of the hydrophobic region in the membrane.

Next, these characteristics like water content and sulphonation of the membrane are also affected by pretreatment solvent. For that reason, depending on the kind of pretreatment solvent against Nafion membrane, the characteristics of the sensor is affected in its operation [16, 17, 18].

In the simulation, we use the conductivity for 1M H_2SO_4, 2M KOH, and 2M KCl as the pretreatment solvent based on the previous work [40].

The values are shown Table 2.

Table 2: Proton conductivity of Nafion membrane to the kinds and concentration of electrolyte

Pretreated solution	Concentration	Conductivity (S/m)
H_2SO_4	1M	2
KOH	2M	0.4
KCl	3M	0.25

And the results are plotted on figure 8. We calculated the sensitivity of the sensor from the results, that is, 0.2897μA/ppm in H_2SO_4, 0.0821μA/ppm in KOH, 0.0836μA/ppm in KCl respectively. As shown the results, the sensitivity with H_2SO_4 pretreated is the highest among three them, this proves the affection of electrolyte conductivity to the sensitivity.

As shown figure 9, when we used 1M H_2SO_4 as a pretreatment solvent, the sensor presents the high state in terms of not only current intensity but also sensitivity than the other cases for NO_2 gas, 1-20ppm level.

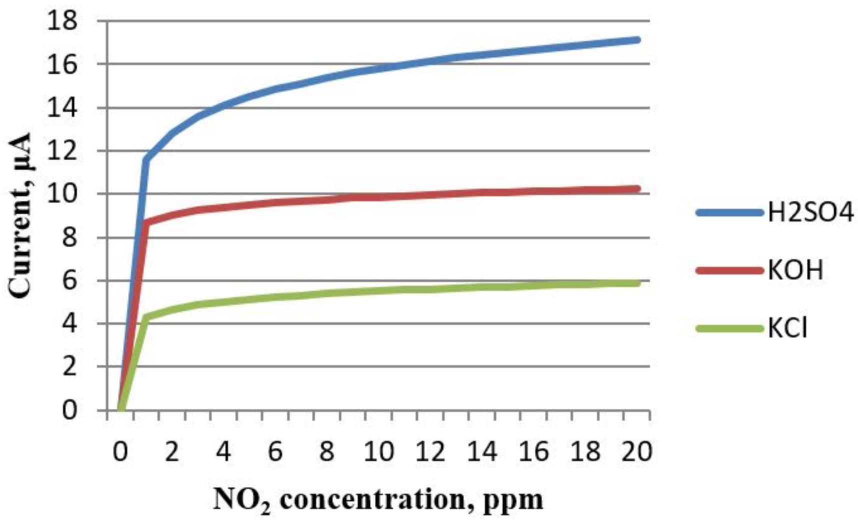


Figure 9: The current characteristics of three sensors with different pretreatment solvent versus NO_2 gas concentration.

Experimental results

An experiment has conducted to confirm the simulation results.

The fabrication process of the sensor followed mentioned above. First of all, a sensor which is pretreated with H_2SO_4 is used to analyze the I-V characteristics in terms of bias voltage, operating temperature and relative humidity.

Figure 10 shows the experimental results to the I-V characteristics by using cyclic voltammetry. We use the Multi-functional Electro-Chemistry analyzer MEC-16 as a measuring equipment.

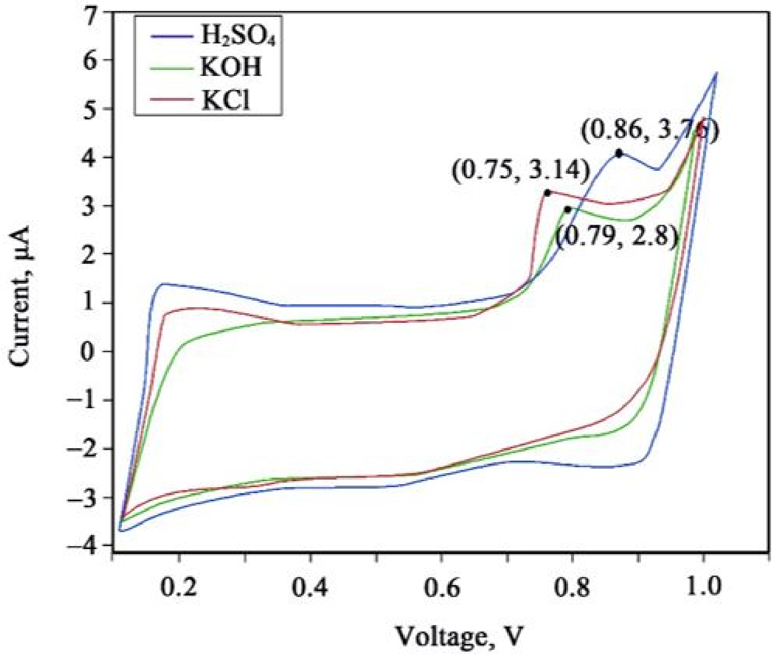


Figure 10: Current-Voltage curve of the sensor by using cyclic voltammetry.

As well as simulation results, for the sensor with H_2SO_4-pretreated, the current peak is the highest as 3.76mA at bias voltage 0.86V. And the current signal is higher than the others.

Figure 11 and Figure12 shows the experimental results for the current characteristics of the sensor with operating temperature and relative humidity to consider the effect of the ambient condition.

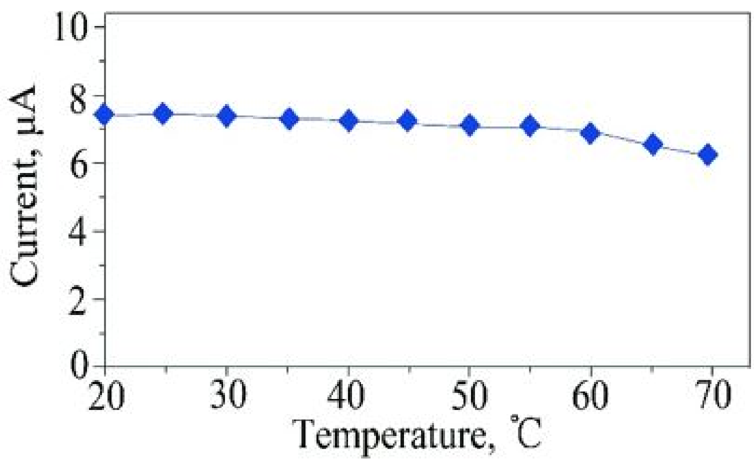


Figure 11: Experimental results for the current characteristics versus operating temperature (NO_2 gas concentration-20ppm, bias voltage-0.9V)

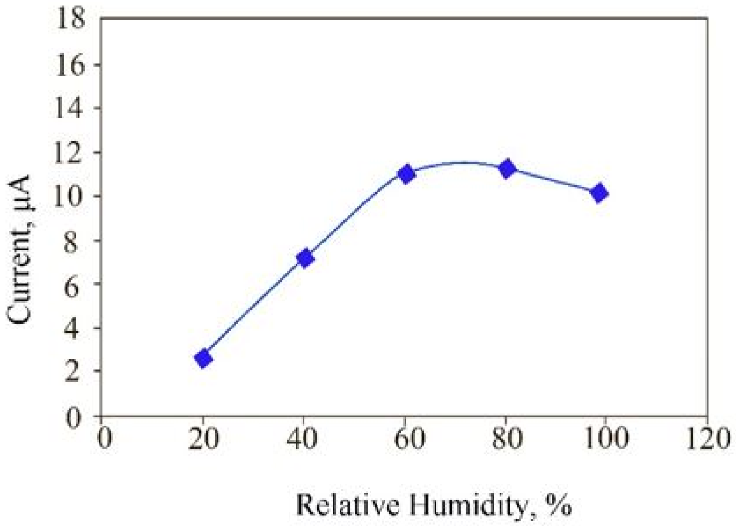


Figure 12: Experimental results for the current characteristics versus relative humidity.

(NO_2 gas concentration-20ppm, bias voltage-0.9V)

The temperature rises up to the higher, the sensing current gradually reduces. It can be explained by the deterioration in the adsorption and dis-adsorption of the gas onto the electrodes with the increase of temperature. This agrees with the simulation.

Meanwhile, when the sensor operates under the 20~80& RH, the current intensity increases as relative humidity rises up. But it reaches around 100% RH, the signal shows significant reduction in their operations. This explains that too much water content in the Nafion membrane is unfavorable to make the path for proton conduction. The difference between simulation and experiment can be explained it comes from the use of theoretical proton conductivity in the simulation without practical experience.

As shown the experimental results, the sensor should be used under the 40~80% RH condition.

Figure 13 shows the results for the current characteristics of sensors with different pretreatment solvent versus NO_2 concentration.

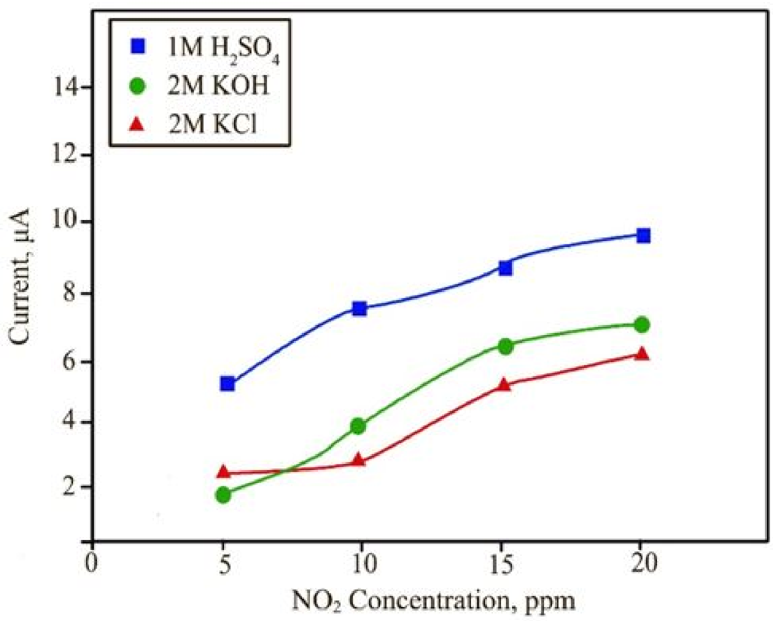


Figure 13: Experimental results for the current characteristics of sensors versus NO_2 concentration.

Although there are some numerical differences from the simulation, the sensor with H_2SO_4-pretreated shows the highest sensitivity than the others. It ranges from 4.5 to 8.9µA in the signal size and has 0.22µA/ppm in sensitivity.

This proves that the use of H_2SO_4 as a pretreatment solvent is the best for the water absorption and sulphornation to the membrane, continuously for the current intensity and sensitivity of NO_2 gas sensor.

Above all the experimental results show the numerical differences, but it is nearly similar with the simulation results in terms of reaction tendency or sensitivity.

Because it is calculated in the simulation for the results based on the finite element analysis, the number of element influences results significantly. The more elements take longer time for simulation, but we can more correct results. In this paper, to reduce the simulation time we had a rough meshing and we considered the reaction tendency and sensitivity. Also, we cannot ensure the material parameters used in the simulation are perfectly same. And there can be several factors which contribute to differences simulation and experiment including applying equation, considering the complex phenomenon of electrochemical reaction etc.

As a result, experimental results are nearly similar with the simulation results in terms of reaction tendency or sensitivity and they are also similar with the previous works.

Conclusion

We have performed the simulation for the electrochemical NO_2 gas sensor based on the Nafion membrane using the COMSOL Multiphysics. And then we've fabricated and tested the suggested sensor.

The sensitivity from the simulation is 0.2897µA/ppm to H_2SO_4, 0.0821µA/ppm to KOH, and 0.0836µA/ppm to KCl. It shows the highest sensitivity in the case of H_2SO_4. We can understand that the pretreatment solvent affects significantly to the characteristics of the sensor, so we should choose the solution in the right.

Experimental results were obtained by 0.2897µA/ppm in sensitivity with the H_2SO_4-pretreated sensor. And all the results for operating temperature, relative humidity, and the kind of pretreatment solvent are nearly similar with the simulation and previous works.

In our work we focused on the combination of simulation and experiment, we don't care about the selectivity and long-term stability of the sensor. These studies will be continuous by us.

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