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Economical Gas Chamber for In-situ Gas Measurement and Analysis of Gas Response Characteristics according to Sensor Voltage

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인시투 가스 측정이 가능한 경제적 가스 챔버 구현 및 센서 전압에 따른 가스 응답 특성 분석

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ABSTRACT

Breath analysis using a portable device is better than the classical breath analysis system in terms of installation and operation. There is an increasing need to develop cost-effective equipment for testing gas sensors from the viewpoint of functionalities that can be applied applicable to portable devices. In the present study, an economical gas chamber for in-situ gas measurement is implemented with a single gas chamber without using expensive gas storage and control equipment; the gas response characteristics are analyzed using the above-described chamber. The main features of the implemented gas chamber are simple injection procedure, improved gas diffusion, easy measurement and cleaning, support for low-power mode measurement function for portable devices, and open source platform. Moreover, an analysis of gas response characteristics based on changes in sensor voltage show that the sensitivity and 90% response time are affected by the sensor voltage. Furthermore, the sensitivity graph has an inflection point in a specific range. The gas sensor applied in this study showed fast response speed and high sensitivity for sensor voltages of 3.0-3.5 V, regardless of the concentration of acetone gas, the target gas used in this study.

Keywords : Gas Sensor(가스 센서), Portable Device(휴대형 기기), Respiration Analysis(호흡 분석), Gas Chamber (가스 챔버), Response Characteristics(응답 특성)

1. Introduction

A human's exhalation component consists of more than 200 compounds, including nitrogen, oxygen, carbon dioxide, water vapor, volatile organic compounds (VOC), and sulfur. A few of the compounds present in the exhalation components are related to diseases. Therefore, respiration analysis, a noninvasive diagnosis technique for studying the exhalation component of humans, is increasingly attracting attention. With this diagnostic technique,

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practitioners need not handle complicated biomaterials, as they would with blood or urine analysis. [1,2]

Gas chromatography has been used for breath analysis since the 1960s. It is among the several noninvasive technologies available for clinical diagnosis, disease monitoring, and environmental exposure assessment.[3,4] However, such technologies cannot be applied to portable devices owing to the associated complexity, large size, and high installation and operating costs.

To overcome these disadvantages, researchers have explored the application of metal oxide semiconductor (MOS) sensors, which employ change in electrical conductivity for sensing, to portable devices.[5,6] However, adequate testing equipment for developing new gas sensors specific to portable devices or for applying existing sensors is unavailable. Consequently, installation and operation of dedicated facilities for gas mixing, storage, transportation, and purging are required. In addition, the low-power measurement function for devices with limited power, such as portable devices, is underdeveloped.

In the present study, to achieve in-situ gas measurement without using expensive gas storage and control equipment, we design a device based on a simplified injection process to realize gas injection, sensitivity measurement, and cleaning in the same gas chamber. We investigate the feasibility of an economical gas chamber that can measure gas sensors for portable breath analysis by adding a low-power measurement function to existing portable instruments. In addition, via an analysis of gas response characteristics by considering various combinations of changes in sensor voltage (1.4-4.2 V) and acetone gas concentration (0.8, 1.6, 3.2, 6.4, 12.8 ppm) with fixed heater voltage, sensor material structure, and diffusion rate condition, we examine the factors affecting sensor response characteristics and determine the optimal sensor response characteristics.

2. Materials and Methods

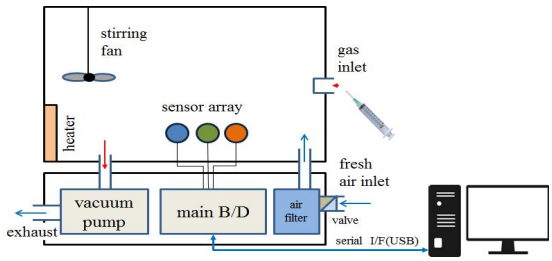


Fig. 1 Schematic diagram of the gas chamber

2.1 Overview of gas measurement system

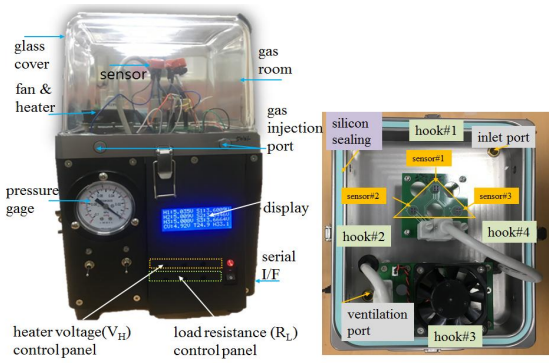
The gas test chamber implemented in this study can measure up to three sensors simultaneously. The gas test chamber has a capacity of 4500 cc, stirring fan for mixing, display, serial interface for communication with an external computer, and vacuum pump (Air pump, DC 24 V, DAP-3675B) for evacuation. The upper cover of the chamber can be opened to facilitate natural ventilation. The overall structure of the gas test chamber, including the components for gas injection, measurement, and purging, is shown in Fig. 1; details of the present study can be founded in sections Mechanical design and Hardware and software.

2.2 Mechanical design

Mechanically, the chamber must be able to measure gas concentration in the gas diffusion space. Only minor changes occur in the external environment when the target gas is injected into the chamber, and chamber must be capable of inducing fresh air into the chamber to effectively remove residual gases or odors before a new experiment.

The design and implementation schemes that meet the abovementioned requirements are shown in Fig. 2. The implementation details are as follows:

To minimize internal contamination owing to residual gases formed by repeated gas concentration



(a) front view **(b) inner side**

Fig. 2 Customized gas chamber

measurements, the upper cover of the chamber is made of a heat-resistant tempered glass with a soda lime component, and the lower part of the chamber is processed with aluminum oxide.

The chamber is equipped with a silicone ring and coupling hooks on four sides to facilitate target gas measurement without dilution due to the induction of external air. To remove any residual odors before and after gas measurement, the top cover is designed to separate from the chamber to allow through-passage of the natural ventilation structure, and vacuum pumps are installed to force air intake and exhaust. In addition, a pressure gauge for measuring the degree of vacuum is mounted on the front surface of the chamber.

The required circuit configurations for testing various type of sensors in the chamber may differ, which means installation and replacement may be difficult. In the present study, we standardized the sensor interface by combining a 2 X 2 socket with a voltage regulation circuit to facilitate convenient installation and replacement.

We applied a three step measurement process herein. First, (1) after baseline purging (opening top cover or forcing intake/exhaust), (2) we injected the target gas through at syringe into the space inside the closed chamber. (3) After measurement, we purged the chamber (by opening top cover or forcing intake/exhaust) for the next experiment.

Table 1. MOS gas sensor

MOS sensor	Sensing material	Specificity
TGS 822	SnO_2	organic solvent vapors (ethanol, acetone, alcohol, etc) heater resistance: 38.0 ± 3.0 Ω

2.3 Hardware and Software

2.3.1 Metal oxide semiconductor sensor

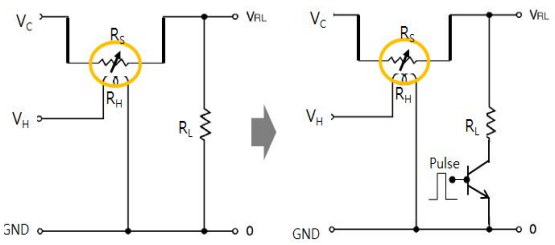
Metal oxide semiconductor gas sensors, invented by Taguchi in the 1960s, are among the most widely used commercially avaliable gas sensors. They provide gas selectivity by sensing material composition and heater temperature control.

Acetone, a bio-marker for diabetes measurement was used as the target gas, and the characteristic test was performed with the TGS 822 sensor, described in Table 1, among the MOS sensors that are reactive with the target gas.[7]

The surface temperature of the sensor material, which is well reacted with the gas sorption behavior of the MOS sensor, is known to be 200-400 ℃, and the heater voltage recommended for TGS 822 is 5 V. [7] In this study, measurement deviations due to different surface temperatures were minimized by using the manufacturer-recommended heater voltage.

2.3.2 Sensor measurement

Unlike the general gas sensor circuit shown in Fig. 3 (a), in this study, we designed and implemented the circuit shown in Fig. 3 (b) to individually control the supply times of the heater and the sensor voltages.



(a) (b)

Fig. 3 Control circuit for interdigitated electrodes (IDE) duty period

The heater voltage was supplied continuously to maintain the surface of the sensor material at a constant temperature, but the sensor supply voltage (V_c) was supplied only during the gas reaction measurement. This was achieved by controlling the duty period pulse signal applied to the transistor base. The circuit shown in Fig. 3 (b) can minimize power consumption in portable devices that use MOS sensors and be used to measure gas sensors using microelectomechanical systems.[8]

Based on the load resistance, by turning the transistor on, one can measure the sensor load voltage (V_{RL}), and adjust the pulse period applied to the transistor base in millisecond. Figure 4 shows the pulse period controlled to 1.58 ms.

Sensor supply voltage and heater voltage (V_H) were varied from 0V to 5 V by using a low drop output linear regulator (LDO) (LT3080, Analog Devices) and a variable resistor. The sensor load resistance R_L was set to 0.1-100 KΩ by using a variable resistor.

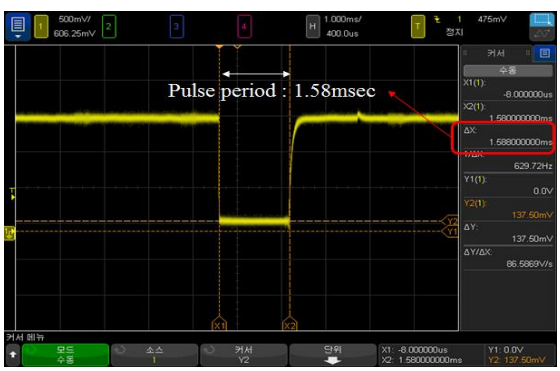


Fig. 4 Pulse period control signal measurement

The sampling periods of the values collected using an analog-to-digital converter and digital-to-analog converter (ADC-DAC) (ADC: 12 bit, DAC: 8 bit, AD 5593R, Analog Devices) installed in the measurement circuit were adjusted using a program. The collected value of the sensor load voltage V_{RL}, heater voltage V_H, and temperature and humidity inside the chamber were transmitted to computer via the UART serial interface (115200 bps, n, 8, 1), stored, and processed subsequently.

In measurements conducted after the implementation of heater voltage and load resistance control (Fig. 4 (4)), voltage fluctuation occurred owing to duty control of the sensor voltage. The fluctuation was ascribed to the fact that measurements conducted immediately after tuning the transistor turn on included variations in the load resistance voltage. Therefore, a certain stabilization time after application of the duty control pulse is required for stable gas concentration measurement in duty mode operation. This problem was solved by measuring V_{RL} with a time delay of 100 μs.

The micro control unit (MCU) applied in this study is ESP32 (32 bit, dual core, Flash 4 MB, ESP32-DOWD), which is equipped with Wi-Fi, Bluetooth, three serial interfaces, and two I2C interfaces. A block diagram of the main circuit of ESP32 is shown in Fig. 5.

2.3.3 Control components

1) Measurement of chamber environment: To measure temperature and humidity changes before and after gas measurement, the temperature and humidity sensor was configured to use I2C signal instead of analog signals when gathering sensor data via a digital interface.

2) Fans and heaters: A fan and a heater were used to control the temperature and humidity in the gas chamber, and the gas mixture in the chamber was

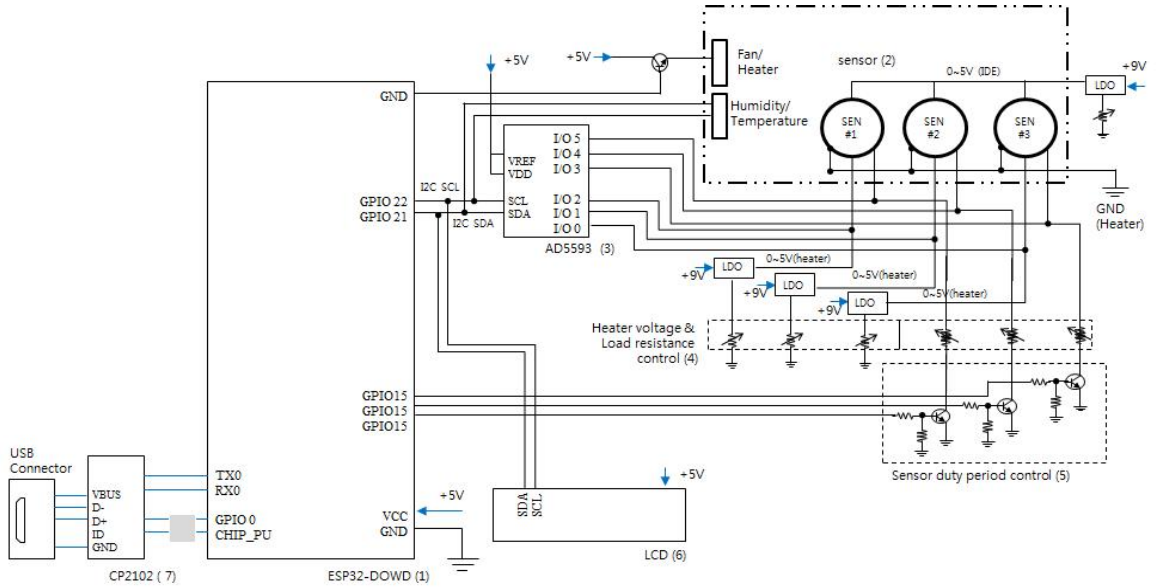


Fig. 5 Electronic circuit designed for gas chamber

controlled. The program facilitates, logic control and manual control. In addition, the motor noise generated at fan start was eliminated by separating the measurement circuit from the power supply circuit, and applying a LC filter and a counter electromotive force diode.

3) Heater voltage and load resistance control: The front panel was configured to allow the user to adjust the heater voltage and load resistance required by the gas sensor, and the applied values were displayed in real time on the display shown in Fig. 2.

2.3.4 Software

The Arduino IDE development environment, an open source development platform, was used in this study. We developed dual ADC and dual I2C libraries by considering the scalability of multiple channels and I2C line configurations. ESP32 can be operated using two types of programs: sensor characteristic experiment logic, which is the logic for repeated sensitivity measurement and sensor change experiments in real time, and portable measurement logic, which simulates portable devices. In this section, we describe our application of the sensor characteristic experiment logic.

By applying this logic, we realized sensor data collection and data analysis in real time in Excel (Microsoft), and one record information per second was set to store and display graph in real time. In addition, to support batch processing of data, the measurement data were collected using an open-source terminal program capable of text-based display and storage. The collected data were processed in CSV format to facilitate offline extraction and analysis of characteristics from Excel.

2.4 Gas concentration test

The target gas was acetone (C3H6O), and the target concentrations were 0.8, 1.6, 3.2, 6.4, and 12.8 ppm. The volume of acetone gas that must be injected into the gas chamber for the target concentration measurement was determined by substituting the concentration of the first dilution gas according to the Eq. (1) into Eq. (2),

$$G_{1st\,dilution}(ppm) = \frac{\left(\frac{m_{gas}(cc) \times D_{gas}(g/cc)}{M_{gas}(g/mol)}\right)}{n_{air}(mol)} \times 10^6 \quad (1)$$

where

$G_{1st\ dilution}$ = first dilution gas,

m_{gas} = mass of gas (acetone),

D_{gas} = density of gas (acetone),

M_{gas} = molecular mass of gas (acetone),

n_{air} = number of air mol.

$$V_{inlet}(cc) = \frac{G_{target}(ppm)}{G_{1st\,dilution}(ppm)} \times V_{chamber}(cc) \quad (2)$$

where

G_{target} = target concentration of gas(acetone),

V_{inlet} = volume of inlet/injected gas,

$V_{chamber}$ = volume of gas chamber.

The calculated values of gas concentration used in the acetone concentration test are given in Table 2. The procedure for injecting the target gas at the desired concentration into the gas chamber is shown in Fig. 6, and the details are as follows. 0.154 ml of acetone solution (molecular weight: 58.08 g/mol, density: 0.785 g/ml, purity: 99.9%) was mixed with air in a 25 L vessel to produce a first dilution gas with a concentration of 2,000 ppm. The pressure was 1.0 atm, and the temperature was 293.15 K. A volume of the first dilution gas corresponding to the target concentration was input into the 4.5 L gas chamber by using a syringe according to Table 2.

The target gas was injected using the syringe at an average flow rate of 220 sccm. To minimize the effect of diffusion speed of the injected gas, uniform gas diffusion in the chamber was achieved by mixing air in the chamber with fan for a certain period.

Moreover, to reduce the effect of ambient temperature on the sensor, the heater mounted inside the gas chamber was controlled to maintain a temperature of 293.15 K.

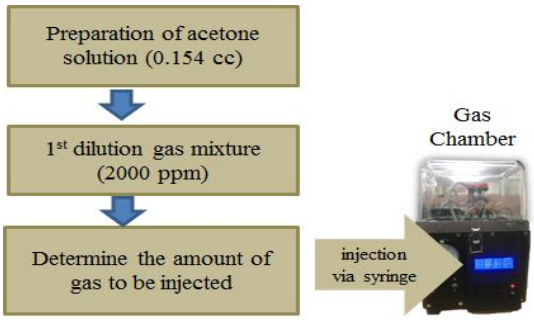


Fig. 6 Gas injection procedure

Table 2 Inlet gas volume list

Target concentration of gas (ppm)	Volume of injected gas (cc)
0.8	1.8
1.6	3.6
3.2	7.2
6.4	14.4
12.8	28.8

3. Results and Discussion

3.1 Sensitivity calculation

Using the sensor load voltage (V_{RL}) obtained using the gas sensor measurement circuit (Fig. 3) and Ohm's law, the gas sensor resistance (Rs) was computed according to Eq. (3).

$$R_s = \left(\frac{V_c}{V_{RL}} - 1\right) \times R_L \quad (3)$$

where

V_c = sensor supply voltage,

R_L = sensor load resistance.

The sensitivity of the sensor (S), which indicates the degree of reaction according to the gas input, is expressed by Eq. (4),

$$\text{Sensitivity(S)} = \frac{R_o}{R_s} - 1 \quad (4)$$

where

R_o = sensor resistance in fresh air.

In Eq. (4), when fresh air is introduced into the chamber without the target gas (acetone), Rs is approximately the same as Ro, and sensitivity converges to zero. This equation shows that sensitivity is independent of R_L and V_c.

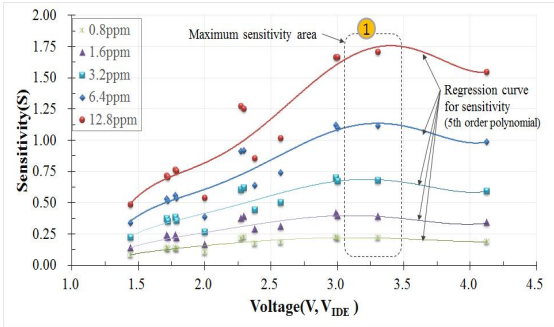
3.2 Gas response characteristics

By observing the change characteristics, it is possible to derive the elements necessary for sensitivity control.

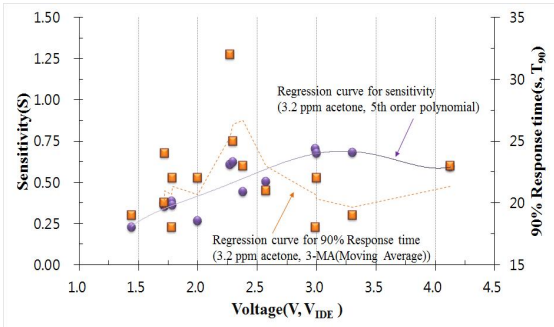
Figure 7 shows two interesting phenomena. One phenomenon varies with changes in sensor voltage (V_{IDE}) obtained in the pre-measurement step for the same acetone concentration. The sensitivity value increases as the target gas concentration increases. However, when V_{IDE} is varied while maintaining the same target gas concentration, the sensitivity value varies with V_{IDE}. Based on this feature, we inferred that different sensitivity values can be obtained if different voltages are applied to the interdigitated electrodes (IDE) of the sensors for the same target gas.

The other phenomenon is the generation of an inflection point in the sensitivity curve in region (1), as in Fig. 7 (a). This means if V_{IDE} is changed for the same target gas concentration, the sensitivity value changes, and a polynomial trend with a maximum value at 3.25 V appears. Moreover, Fig. 7 (b) shows the T_{90} (response time to reach 90% of the maximum variation in sensor load voltage) with the maximum value near 2.25 V and the minimum value near 3.25 V according to V_{IDE}.

The MOS sensor used herein offers fast response and high sensitivity when V_{IDE} is between 3.0 V and 3.5 V.



(a) Sensitivity according to acetone concentration and sensor voltage (V_{IDE})



(b) Sensitivity and response time according to sensor voltage (V_{IDE})

Fig. 7 Characteristics of sensitivity and response curve according to sensor voltage (V_{IDE})

4. Conclusion

In this paper, we presented the results of the design and implementation of a gas chamber that facilitates economic testing, allows user to freely select the target gas and the target concentration, and is equipped with the elements necessary for the development of a portable respiration analyzer. A customized gas chamber was combined with a commercial MOS sensor to archive optimal sensitivity and response speed. This characteristic is the sensor voltage V_{IDE} applied to the sensor. It is shown that the result of the 3rd section is a key factor to consider when comparing sensor sensitivity

measurement results.

Application of the results of this study to a portable respiration analyzer would yield a system that provides optimum sensitivity by considering the response speed characteristic and the sensitivity change specificity according to changes in $V_{IDE.}$ In addition, the system would offer a low-power measurement function by using power only when necessary.

In the future, we will expand the number of sensors from three to a minimum of five and a maximum of 16 to support the e-nose study. We will conduct a sensitivity study by considering gas injection position, gas sensor location, other structures, and moisture. In addition, we will conduct studies on the classification and regression of gas selectivity through machine learning and study element technologies that are essential for portable respiration analysis equipment.

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