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Resistivity of SnO2 Gas Sensor to Humidity, CO2 Gas, and Temperature in Food Decomposition Process

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Abstract:

This study aims to determine the sensitivity of a gas sensor based on a Cu substrate coated with SnO_2 through an electroplating process, involving variations in electrolyte solution temperature and thermal oxidation. The deposition parameters were set as follows: electrolyte solution prepared by dissolving $SnCl_2$ in distilled water, applied voltage of 4.5 V, electrode distance of 3 cm, and electroplating duration of 3 minutes. Sensor sensitivity tests were carried out by observing the food decomposition process, placing both the food sample and sensor in a testing chamber. Data acquisition of temperature, humidity, CO_2 concentration, and sensor resistance was conducted using transducers and Logger Pro software. Based on the results, the sensor sample with an electrolyte temperature of 60°C (sample B) exhibited better performance than the sensor sample with an electrolyte temperature of 30°C (sample A). Sample B demonstrated greater responsiveness to temperature changes, with a coefficient of determination R^2 = 0.66943. It also showed better detection of CO_2 concentration changes with R^2 = 0.98225. This improvement is attributed to a more effective electroplating process, as indicated by the mass change and thickness of the SnO_2 layer. The sensitivity of sample B, defined by the equation $S([CO_2]) = 9.42E\text{-}5 - 1.17E\text{-}9[CO_2]$, was superior to that of sample A, which followed the equation $S([CO_2]) = -2.34E\text{-}4 + 4.25E\text{-}9[CO_2]$, as shown in the plotted graphs. Sample B exhibited a negative linear curve with a gentle slope, indicating a stable gas sensor behaviour.

1. Introduction

Food spoilage is a natural process that occurs due to the activity of microorganisms such as bacteria and fungi that break down organic compounds in food. This process is greatly influenced by environmental factors, especially temperature and humidity. High temperatures accelerate the metabolic rate of microorganisms and enzymatic reactions in food, thereby accelerating the decay process. In addition, high air humidity also accelerates decay because it provides ideal conditions for the growth of decay-causing microorganisms [1].

During the decomposition process, physical and chemical changes occur in foodstuffs, including increased production of carbon dioxide (CO_2) due to respiration and decomposition of organic compounds. CO_2 levels in storage rooms can increase several times higher than normal conditions, depending on the type of food and storage temperature [2]. Therefore, managing temperature, humidity, and gas composition in the storage environment is very important to slow down decay and maintain food quality [3].

Food spoilage monitoring technology also involves nanosensors, which utilize inorganic, organic, and carbon-based materials, offering rapid and accurate detection of microbes, toxins, and additives in food products [4]. The implementation of this technology not only helps extend the shelf life of products but also reduces food waste and improves overall food safety [5].

The development of sensors to monitor food quality has made significant progress with the application of various gas sensor technologies, color sensors, and artificial intelligence-based smart systems. Gas sensors are important devices for detecting and monitoring the presence of certain gases in the environment. The basic principle of a gas sensor is to convert gas concentration into an electrical signal that can be analyzed quantitatively [6]. The sensitive element is the part that interacts directly with the target gas. This interaction then causes changes in physical or chemical properties, such as resistance, capacitance, or voltage, which are then converted into electrical signals [6]. With the development of technology, gas sensors can now also be integrated into portable and wireless systems, enabling real-time gas monitoring. This is particularly important in environmental and industrial applications, where the presence of toxic gases such as CO, NO_2, or VOCs must be continuously monitored to ensure workplace safety [7].

Thin films are materials with very small thicknesses, ranging from a few nanometers to a few micrometers, which are applied to the surface of a solid substrate. The physical and chemical properties of materials in the form of thin films often differ from those of bulk materials due to the influence of size, microstructure, and interfacial tension. Therefore, the choice of thin film deposition method greatly determines the final quality of the film and its suitability for the desired application [8].

One important method is electrodeposition, which is a technique for coating metals using electric current from an electrolyte solution. Its advantages lie in the simplicity of the process and the ability to coat at low temperatures, but it is limited to conductive substrates and is prone to producing irregularities in complex geometries. With these various methods, the choice of deposition technique must be tailored to the application requirements, the type of material, and the properties of the substrate used [9].

In this study, an SnO_2 gas sensor will be made using the electroplating method. Lee et al. (2021) successfully made an SnO_2 sensor coated with a thin layer of reduced graphene oxide (rGO) using the drop-cast electrode method. The results of this study show the sensor's ability to detect changes in CO_2 gas at room temperature with higher humidity.[10]. The novelty of this research lies in the sensor fabrication method used. This method has several advantages, including being relatively simple, efficient, and potentially producing a more uniform, nano-sized layer with controlled thickness and low cost [11]. The electroplating method in SnO_2 sensors is expected to increase sensor sensitivity.

Islamiyati et al. (2024) conducted research on the effect of temperature variations in the electroplating process on the resistivity of temperature sensors. The results showed that the thickness of the thin layer varied in proportion to the variation in electrolyte temperature during the electroplating process, with the optimal temperature being 40°C and 50°C. In addition, the low-temperature sensor produced by electroplating had excellent sensitivity to temperature changes [12]. Another novelty lies in the use of electrolyte temperatures with a wider range, namely 30°C and 60°C. This aims to determine the significance of the effect of electrolyte solution temperature variations in the electroplating process on the resistivity and stability of the sensor.

Most previous studies have been limited to the use of pure gases or simple mixtures, rather than the complex gas compositions that actually occur during decay (such as VOCs, ammonia, high humidity, and temperature fluctuations). This means that the performance of sensors under real conditions has not been fully verified. Therefore, the main focus of this study is to create an SnO_2 gas sensor using the electroplating method with variations in electrolyte solution temperature. The SnO_2 gas sensor was then tested for its thin film characteristics and sensitivity to environmental changes during the food spoilage process.

2. Method

2.1. Substrate Preparation

Substrate preparation begins with creating a sensor lithography on a commercial copper plate (PCB) measuring 108 mm x 2 mm x 70 µm. Copper cleaning is aided by a ferric chloride and acetone solution until a substrate like the one in Figure 1 is formed.

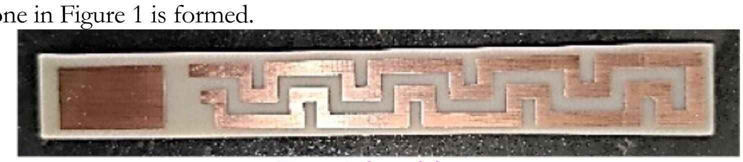

Figure 1. *Sensor Substrate*

Next, the substrate surface is cleaned until it appears shiny by rubbing it with a microfiber cloth coated with Autosol metal polish. The substrate is then cleaned with toothpaste and washed with soapy water. The final step is to clean the substrate using an ultrasonic cleaner with distilled water and , followed by 96% alcohol. After drying with a hair dryer, the Cu plate is weighed using an Ohauss PA214 balance.

2.2. Preparation of SnO_2Samples

SnO_2 samples were prepared using the electroplating method. A carbon electrode as an inert material was placed at the anode and a Cu substrate at the cathode at a distance of 3 cm. Both electrodes were immersed in a 0.016 M $SnCl_2$ electrolyte solution by mixing 1.493 grams of $SnCl_2$ into 500 mL of distilled water. The electroplating process was carried out at a voltage of 4.5 V for 3 minutes with variations in the electrolyte solution temperature of 30°C (sample A) and 60°C (sample B).

At the cathode, tin ions are reduced to tin metal (Sn), which coats the Cu, with the reaction

$$\mathrm{Sn} + 2\mathrm{e} \rightarrow \mathrm{Sn}. \quad (1)$$

Meanwhile, at the anode, the anion Cl^- from the electrolyte solution is oxidized into chlorine gas (Cl_2) with the chemical reaction

$$2\mathrm{Cl} \rightarrow \mathrm{Cl} + 2\mathrm{e}. \quad (2)$$

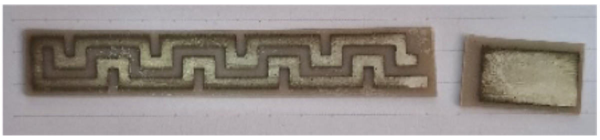

Figure 2. *SnO_2 sample*

After the electroplating process, thermal oxidation is carried out at a temperature of 150°C for 1.5 hours in a Neycraft JFF2000 oven to produce SnO_2 samples [13].

2.3. Determination of Layer Thickness

The SnO_2 sample was weighed using an Ohauss PA214 balance. The weight data for Cu and SnO_2 were recorded as m_{Cu} and m_{SnO2}. The data m_{Cu} and m_{SnO2} as well as ρ_{Sn} the density of Sn (7.3 g/cm³), can be used to indirectly determine the thickness of the thin layer using equation (3).

$$t_{Sn} = \frac{m_{SnO2} - m_{Cu}}{\rho_{Sn} A}. \tag{3}$$

2.4. Determination of Sheet Resistivity

The sheet resistivity (R_s) of the SnO_2 sample was measured using a four-point probe JG-ST2258C. The electroplating process will be successful if the value of R_{sSnO2} is greater than R_{sCu}.

2.5. Sensor Measurement

Sensor testing was conducted during the food spoilage process by observing the environmental conditions using voltage, temperature, current, humidity, and CO_2. The sensors used in the voltage measurement were Vernier VP-BTA; temperature was measured with a Vernier TCA-BTA temperature sensor; current with a Vernier DCP-BTA; humidity with a Vernier RH-BTA; and CO_2 gas with a Vernier CO_2-BTA. These sensors were then connected to a transducer and Logger Pro software with the help of a LabQuest Mini for 22 hours, with a sampling rate of 60 samples per hour.

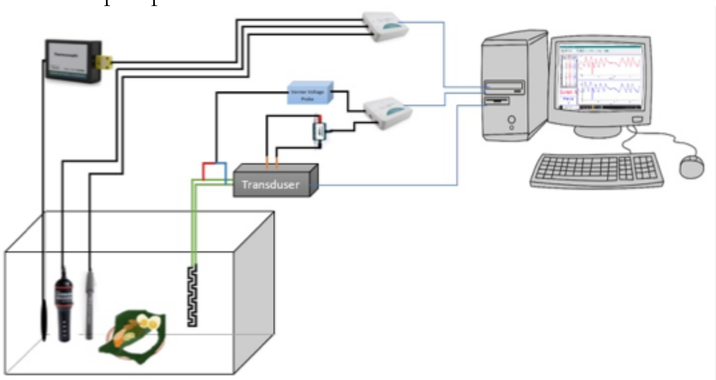

Figure 3. *Sample Resistivity Test Schematic*

The output voltage of the SnO_2 sensor is used to determine the sensor's response to environmental changes [14]. If the sensor voltage has a good correlation with environmental conditions, then the sensor is working properly.

2.6. Determination of Sensor Sensitivity

To obtain the absolute sensitivity of the sensor, a resistance vs. temperature curve, resistance vs. CO_2 curve, and resistance vs. humidity curve were first created according to the polynomial equation

$$\mathrm{R(X) = AX + BX + C} \tag{4}$$

where R is resistance, X is temperature/CO_2/humidity, and A, B, and C are constants. Absolute sensitivity is obtained from the slope of the curve in equation (4), namely:

$$\mathrm{S(X)} = \frac{\mathrm{dR(X)}}{\mathrm{dT}} = 2AX + B \tag{5}$$

where S is the absolute sensitivity, and A and B are constants.

3. Result and Discussion

3.1. Physical Data of the Sensor

Table 1 and Table 2 show the results of measuring the mass of Cu and SnO_2 , the thickness of the layer calculated using equation (3), and the resistivity of Cu and SnO_2 wafers in samples A and B. These results indicate an increase in layer mass and resistivity on the substrate before electroplating and on the samples after electroplating, which is an indicator of the success of the electroplating process.

Tabel 1. Quantities on the Substrate and Sample A

Measured Quantities	Value
Mass of Cu plate	(2928,84 ± 0,02) mg
Mass of SnO2 sample	(2933,14 ± 0,02) mg
Mass of SnO2 layer	(4,30 ± 0,03) µg
Thickness of SnO2 layer	(44,76 ± 0,62) µm
Resistivity of Cu plate	$(1,35 \pm 0,01) \times 10^{-3}$ Ω/sq
Resistivity of SnO_2 plate	$(1,39 \pm 0,03) \times 10^{-3}$ Ω/sq

Tabel 2. Quantities on Substrate and Sample B

Measured Quantities	Value
Mass of Cu plate	(2836,54 ± 0,02) mg
Mass of SnO2 sample	(2849,32 ± 0,02) mg
Mass of SnO2 layer	(4,62 ± 0,03) µg
Thickness of SnO2 layer	(48,09 ± 0,62) µm
Resistivity of Cu plate	$(1,36 \pm 0,01) \times 10^{-3}$ Ω/sq
Resistivity of SnO_2 plate	$(1,40 \pm 0,02) \times 10^{-3}$ Ω/sq

The table shows that the increase in SnO_2 layer mass in sample A with an electrolyte solution temperature of 30°C during electroplating was (4.30 ± 0.03) µg, lower than sample B with an electrolyte solution temperature of 60°C during electroplating, which was (4.62 ± 0.03) µg. The same can be seen from the thickness of the SnO_2 layer formed. Sample A has a layer thickness of (44.76 ± 0.62) µm, which is smaller than sample B, with a layer thickness of (48.09 ± 0.62) µm.

This condition occurs because at an electrolyte solution temperature of 30°C, the solution viscosity tends to be higher. This causes low ion mobility, resulting in a slow ion deposition rate [15]. At a solution temperature of 60°C, the solution viscosity decreases, causing mobility and conductivity to increase, which makes the electroplating process more efficient [16].

The resistivity of the wafers before and after the electroplating process also increased, with the resistivity of sample A being $(1.39 \pm 0.03) \times 10^{-3}$ Ω/sq and sample B being $(1.40 \pm 0.02) \times 10^{-3}$ Ω/sq. The increase in both samples was the same and very small. This was because the Sn coating had more uniform and adhesive characteristics [17].

3.2. Environmental Conditions and Sensor Voltage

Figures 4 and 5 show the response of the temperature, humidity, and CO_2 sensors over time as a description of the environmental conditions during the food decay process, as well as the response of the voltage and current sensors, which is shown by the resistivity graph over time.

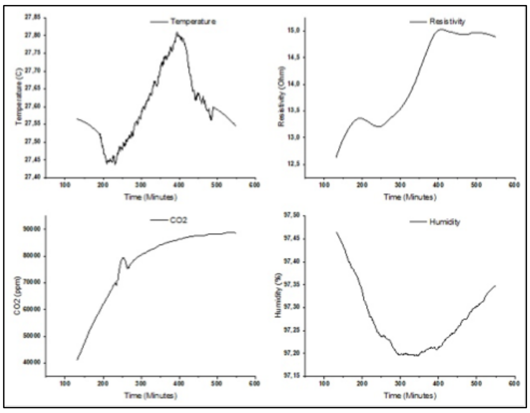

Figure 4. *Sensor Response to the Environment in Sample A*

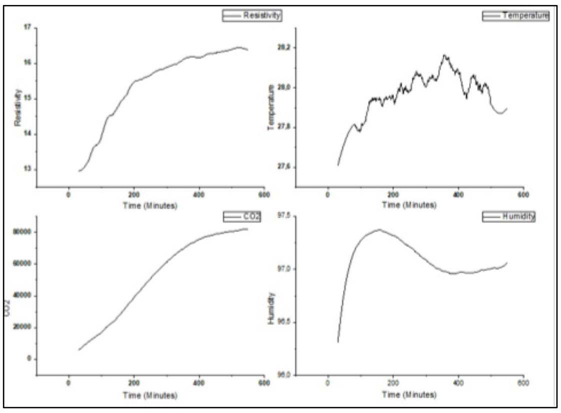

Figure 5. *Sensor Response to the Environment in Sample B*

In the food decay process, there are very complex interactions between the gas conditions—such as ambient temperature, CO_2 levels, and air humidity—and the sensor resistivity. It takes time for the environment to reach equilibrium after the addition of the test food [18].

Afterward, the temperature slowly increases along with the metabolic activity of the microbes present. CO_2 gas also increases sharply in the early stages because microbes begin to break down carbohydrates, proteins, and fats into gaseous compounds. CO_2 levels may then decrease as the food begins to dry out. The same applies to air humidity: humidity initially increases due to evaporation and microbial respiration, and begins to decrease when the food starts to dry out [19].

Figures 4 and 5 show that the SnO_2 sensors in samples A and B were able to respond to changes in environmental conditions, as indicated by the temperature, humidity, and CO_2 sensor readings. Based on these results, further analysis can be conducted on the sensor sensitivity.

3.3. Resistance Curves Against Temperature, CO_2, and Humidity

Figures 6, 7, and 8 show the resistance curves against temperature (R vs. T), CO_2 (R vs. CO_2), and humidity (R vs. RH) according to equation (4).

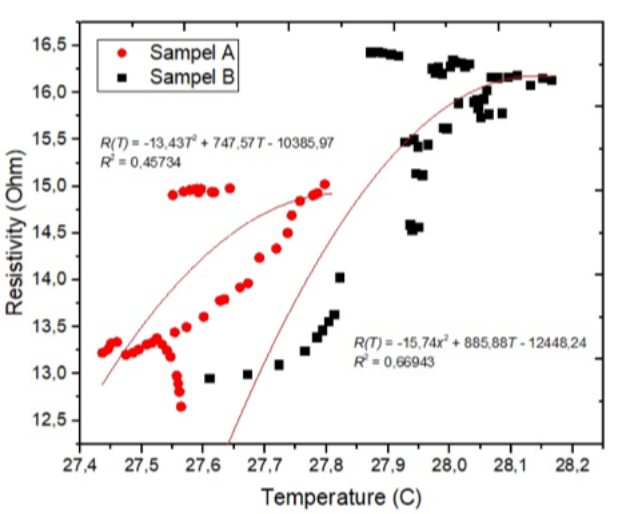

Figure 6. *Analysis of Resistance Data Against Temperature*

Figure 6 shows a significant difference between the results of samples A and B. Sample A has a coefficient of determination of $R^2 = 0.4573$, while sample B has $R^2 = 0.66943$. Both sensor samples are not yet able to accurately detect changes in ambient temperature. In addition, other variables such as temperature and humidity may influence the output. However, sample B shows a higher coefficient of determination value. This finding aligns with the electroplating results obtained at an electrolyte solution temperature of 60°C, which were better than those at 30°C.

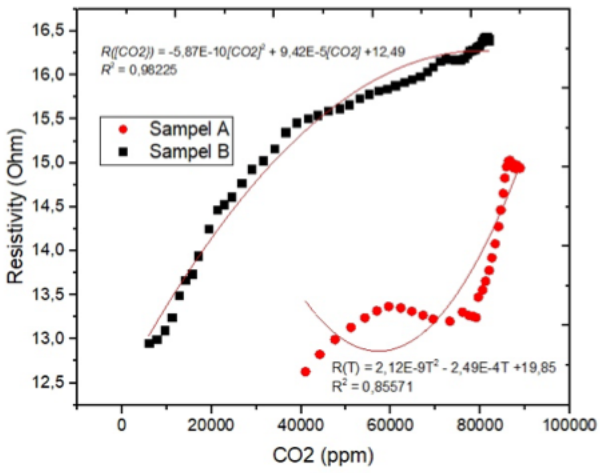

Figure 7. *Analysis of Resistance Data to CO*

Figure 7 also shows a significant difference in results. Sample A has $R^2 = 0.88571$, while sample B has $R^2 = 0.98225$. These results indicate that the SnO_2 sensor is capable of accurately detecting changes in CO_2 levels. Furthermore, sample A, fabricated at an electrolyte solution temperature of 30°C, exhibits lower sensitivity compared to sample B, which was prepared at 60°C.

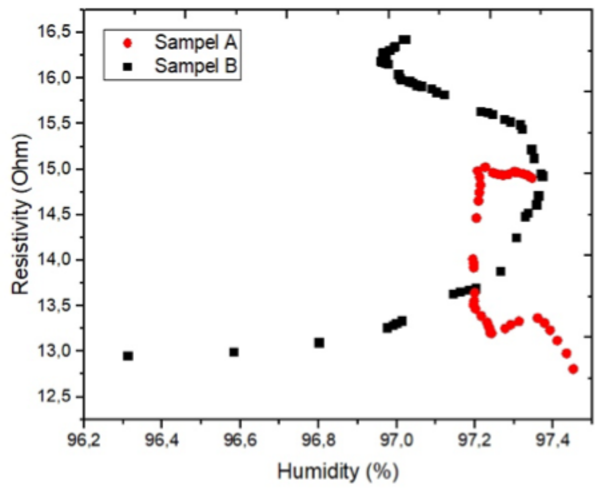

Figure 8. *Analysis of Resistance Data to Humidity*

Figure 8 shows that the SnO_2 sensor is unable to detect changes in air humidity during the food decay process. This occurs because the SnO_2 sensor primarily detects gases through the chemisorption of oxygen on its surface. Ions such as O_2^- or O^- capture electrons from the SnO_2 surface, causing changes in resistivity when the gas reacts. Water molecules, however, interact weakly and have minimal direct effect on resistance [20].

3.4. Sensor Sensitivity

Figure 9 shows the absolute sensitivity curve of the SnO_2 sensor at various CO_2 concentrations. It shows a significant difference in the sensitivity of the SnO_2 sensor between sample A and sample B. Sample A shows a positive linear curve. This curve shows sensitivity increasing as the CO_2 concentration increases. However, the change in sensor sensitivity from negative to positive indicates that the sensor sample is unstable [21]. The steep slope of the curve indicates that the sensor is highly responsive to changes in CO_2 concentration [22].

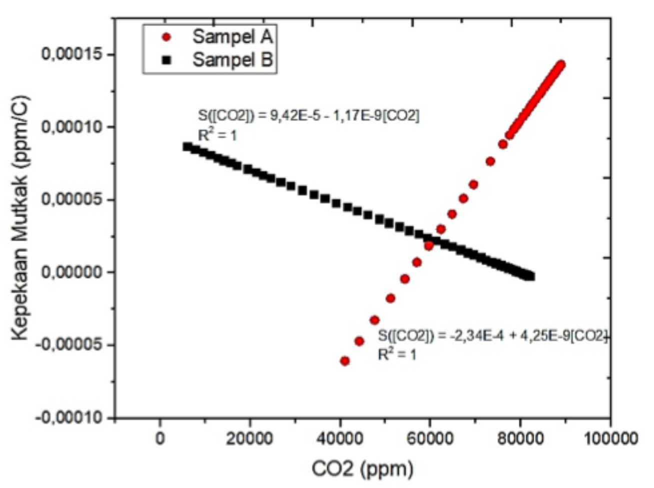

Figure 9. *Absolute sensitivity of the SnO_2 sensor*

In contrast, sample B shows a negative linear curve with a gentle slope. This condition indicates a more stable gas sensor. However, the sensitivity of the sensor decreases as the CO_2 concentration increases. Nevertheless, the sensitivity of the sensor in sample B is more stable than that in sample A.

4. Conclusion

Thus, it can be concluded that increasing the electrolyte temperature in the SnO_2 coating process as a gas sensor has a positive effect on the sensitivity of the sensor to changes in temperature and CO_2 levels in the food decay process. More optimal coating at an electrolyte solution temperature of 60°C results in sensor sensitivity that is more stable to temperature at high CO_2 levels.

Acknowledgments

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