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Identification of Acetic Acid-Ethanol Mixture Using Gas Sensor Array and Ensemble Regression

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ABSTRACT

Identification of acetic acid-ethanol mixtures using a commercial gas sensor array equipped with ensemble regression has been carried out. The gas sensor analysis was simple, rapid, and fast since it did not require any sample preparation. A quantitative analysis of the acetic acid-ethanol mixture was carried out to determine the sensitivity and selectivity of the sensor in distinguishing the concentration of the acetic acid and ethanol mixture. This study focuses on the coefficient of determination of 80% of the calibration data set and recovery of 20% of the testing data set. The models showed excellent performance, specifically, the Bagging and Random Forest r^2 for the ethanol calibration data reached 0.91 and 0.94, respectively. The corresponding ethanol test recoveries were 99.95% and 97.84%, indicating the robustness of the model in accurately predicting ethanol concentration. Acetic acid test recoveries were 100.56% and 101.38% with r² of 0.89 and 0.93 for Bagging and Random Forest regression, respectively. Hence, the commercial gas sensor array equipped with ensemble regression can be applied to the quantification of the acetic acid ethanol mixture and demonstrate opportunities for the practical use of this gas sensor array in analyzing real samples, i.e. human breath or environmental monitoring samples.

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1. INTRODUCTION

The odor was produced by volatile organic compounds (VOCs) that can stimulate the sense of smell to produce a sensation through the stimulus of the olfactory system. Smells can interfere with the activities of living beings if they produce an unpleasant odor [1]. Although odors are considered to harm the environment, odors can provide many benefits to human life. The identification of odors is generally performed in agriculture and health. In agriculture, for example, odor identification is used to determine the freshness of fish [2], distinguish types of tea [3], and detect meat adulteration [4]. Odor identification in the medical field is usually carried out for early diagnoses of diseases, such as diabetes [5], pancreatic cancer [6], breast cancer [7], and bowel cancer [8] by identifying the biomarkers of each disease.

Acetic acid can be produced by the human body from foods that contain acetates, such as dairy products, ethanol, and indigestible carbohydrates. [9]. Several studies stated that acetic acid in the human body could be found in the breath and faeces of colon cancer patients. Acetic acid (CH₃COOH) is a colorless and hygroscopic liquid. Acetic acid is one of the most widely used volatile compounds. These compounds are commonly used as food additives and can be found in the environment due to industrial processes [10, 11].

Ethanol is an organic solvent and industrial chemical commonly used in production processes and human life, such as environmentally friendly fuel, cleaning fluids, agriculture, and many other industries. Ethanol is a highly explosive and flammable chemical. Ethanol is explosive if its environmental content reaches 3.3% to 19.0%. Monitoring ethanol in breath is essential for traffic safety, and the ethanol concentration in normal breath is 30-130 ppm. Therefore, it is critical to fabricate sensitive, selective, and real-time ethanol sensors for practical applications [12].

Detection of ethanol and acetic acid mixtures may be relevant in medical diagnostics and research. For example, analysis of breath samples for ethanol content is essential for assessing alcohol consumption. At the same time, the presence of acetic acid can be an indication of specific metabolic processes or diseases. The identification of volatile organic compounds is commonly studied using GC-MS and SIFT-MS [13]. High sensitivity and selectivity were associated with the use of chromatographic methods. However, there were several disadvantages in the analysis using chromatographic methods, i.e. high cost, long analysis time, and the need for sample preparation.

A gas sensor array is a simple method to analyze gas or volatile organic samples with minimal sample preparation. The gas sensor array in this study consists of several commercial gas sensors that can be used as an alternative method to identify volatile compounds in situ and in real-time. Gas sensors generally consist of metal oxide materials such as SnO₂, ZnO, WO₃, CuO, and In₂O₃ as the sensing layer and were doped with other metals such as Au, Pt, Pd, etc., to increase sensitivity and selectivity to the target compounds.

A gas sensor array consists of several sensors with different sensitivities to different VOCs. This allows for increased selectivity because different sensors respond differently to different compounds. Combining the responses of multiple sensors improves the ability to distinguish different VOCs. The array's response to different VOCs creates a unique pattern or "fingerprint" for each compound. Pattern recognition techniques, such as machine learning algorithms, can be applied to analyze these patterns and identify specific VOCs. Individual sensors cannot produce clear patterns. Individual sensors can have cross-sensitivity, in which they respond to multiple compounds. In sensor arrays, the combination of responses helps reduce cross-sensitivity problems. By looking at the overall response pattern, compounds that can trigger similar responses in a single sensor can be distinguished [14].

The output signal of the gas sensor array was usually processed using multivariate statistical analysis. The statistical analysis applied to distinguish quantitative data was based on regression analysis. Sensor performance was optimized using ensemble regression analysis. The purpose of ensemble regression is to combine multiple models to improve prediction accuracy in learning problems with quantitative target variables. The ensemble learning process can be divided into three phases: the generation phase, the pruning phase, and the integration phase. The ensemble method has higher prediction accuracy compared to single models. The ensemble method is beneficial when the dataset contains linear and nonlinear data types; different models can be combined to deal with this type of data [15].

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This research uses a gas sensor array to evaluate a mixture of volatile compounds, acetic acid and ethanol, at a specific ratio. The gas sensor array consists of commercial sensors (MQ-3, MQ-4, MQ-6, and MQ-8) [16]. The ensemble regression method determines the acetic acid and ethanol mixture based on the sensor array response pattern. The effectiveness of these commercial gas sensor arrays in identifying mixtures of acetic acid and ethanol depends on the inherent crosssensitivity properties of the sensors.

2. EXPERIMENTAL METHODS

2.1. Materials and Instruments

The tools used in this study include a 100 mL three-necked boiling flask, 50 mL and 100 mL beakers, 10 mL graduated pipettes, digital thermometers, 100 L micropipettes (Socorex), and 500 ml hexagonal sensor chambers, N_2 gas, 5/8-inch diameter silicone tube.

The materials used in this study were distilled water, acetic acid pro-analysis Merck CAS No.1.00063.2500, ethanol pro-analysis RCI Labscan CAS No.64-17-5, Gas sensors MQ-3, MQ-4, MQ-6, MQ-8, sample chamber, breadboard, jumper cable, Arduino UNO R3 and solderless breadboard MB – 102.

2.2. Methods

This research was conducted by injecting volatile compounds into the sample chamber at a temperature of \pm 65 °C. The sensor chamber was made using a container with a 500 mL hexagon base where an MQ gas sensor was installed on each side. A PET plastic container was provided in the center of the sensor chamber so that the sample gas entry could be spread evenly to all sensor modules. At the top of the tube, a hole was made to connect to a silicone tube connected to the sensor chamber. Sample injection was divided into three timeframes, i.e., 1 minute for N₂ gas baseline measurement, 10 minutes of sample injection, and 15 minutes of mixing chamber cleaning using N₂ gas. The flow rate of N₂ gas was 0.1 L/min or 100 cm³/min. Measurements were carried out with five replications for each test sample. Sensor array data were processed to determine the selectivity and sensitivity of each sensor in discriminating different volatile compounds and different ratios of acetic acid - ethanol.



Figure 1. Schematic of the gas sensor array setup.

2.2.1 Gas Sensor Configuration

The gas sensor array is assembled by attaching MQ-3, MQ-4, MQ-6, and MQ-8 sensors in the perforated sample chamber. The sample chamber was perforated in ± 2 cm diameter to attach the sensor. The sample injector hole was perforated in the cap of the sample chamber. Each sensor has four pins: AO, DO, GND, and VCC. The sensor's pins connect to the Arduino UNO board as follows: *i*) the VCC and GND pins are connected to the Arduino UNO's 5V and GND pins using a

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breadboard, *ii*) the VCC and 5V pins are on the negative side of the breadboard while the GND pin is connected through the positive side, *iii*) the AO pins are connected to analog pins A0, A1, A2, and A3, and *iv*) pin A0 is connected to MQ-3, pin A1 to MQ-8, pin A2 to MQ-6, and pin A3 to MQ-4. The Arduino UNO board connected to the sensor is then connected to a laptop with the Arduino IDE software installed to program the microcontroller. The data obtained was recorded with Microsoft 365 Data Streamer.

TABLE I. List	of sensors	studied
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Channel	Sensor	Compounds
S1	MQ-3	Alcohol
S2	MQ-8	H_2
S3	MQ-6	Isobutane, propane, LPG
S4	MQ-4	S4

2.2.2 Quantitative Analysis

The compounds for the quantitative analysis were a mixture of acetic acid and ethanol. The acetic acid and ethanol ratios were 25%, 50%, and 75% v/v. Five replications were applied for each measurement. The injection for quantitative analysis was carried out at programmed time intervals. During the qualitative test, the sensor measured the ambient air for 1 minute, then the sample mixture was injected, and the sample chamber was closed for 90 minutes. The output signals at the 29th minute were extracted. The extracted data were analyzed using ordinary least squares (OLS), AdaBoost, Bagging, GradientBoosting, and Random Forest regression. The data obtained was split into 80% training and 20% test data to validate the regression models. The results of this research are expected to provide the possibility of practical application of gas sensor arrays to detect mixtures of ethanol and acetic acid as a basis for quality control, safety measures, medical diagnostics, environmental protection, and various industrial applications. Accurate and timely detection helps improve processes, implement safety protocols, and improve overall well-being [17, 18].

3. RESULTS AND DISCUSSION

The sensing mechanism was based on the reaction between metal oxides with oxidizing or reducing volatile organic compounds that caused changes in their conductivity. The change in the conductivity was measured by a pair of electrodes attached to a metal oxide sensor. The sensor acts as an electron donor, providing electrons to the conduction band. The sensor temperature was adjusted using a heater [19]. Acetic acid and ethanol react with oxygen ions on the sensor surface to form CO_2 gas. The equation for the reaction of the four compounds is shown in Eq 1-2:

Acetic acid:			
$CH_3COOH_{(g)(ads)} + 4O^{-}_{(g)(ads)}$		$2CO_{2(g)} + 2H_2O_{(g)} + 4e^{-1}$	(1)
Ethanol:	·		
$CH_3CH_2OH_{(g)(ads)} + 6O^{-}_{(g)(ads)}$	->	$2CO_{2(g)} + 3H_2O_{(g)} + 6e^{-1}$	(2)

The quantitative analysis was carried out by injecting ethanol and acetic acid at a certain composition into the sample chamber. The signal was recorded for 90 minutes. The output signal at 29 minutes was extracted and tabulated as a function of the ethanol: acetic acid ratio. The sensor voltage output is summarized in Table 2. The MQ-3, MQ-4, MQ-6, and MQ-8 sensors were identified by Arduino as S1, S2, S3, and S4, respectively, as shown in Table 2. A radar plot of the output data for each sensor indicated that the magnitude of sensor output depends on the ethanol-acetic acid ratio, as shown in Figure 3. The mixture of 25% acetic acid and 25% ethanol produces a response in a rhombic shape. On the other hand, in the variations of 50% acetic acid, 50% ethanol,

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and 75% acetic acid: 25% ethanol, the plot was shaped like a kite. The shape of each plot shows a change in size, indicating that the sensor can discriminate between the compounds [20].

The pair plot of each sensor response to the acetic acid and ethanol ratio indicated that the responses were not well correlated for individual responses. The pair plot of S1 with S2 and S3 shows some correlation. The S4 pair plot did not show some correlation to sensor responses, as shown in Figure 3. The density plot shows the distribution of each mixture in the sensor. The first row was the responses of each sensor to ethanol (Figure 3 (a)) and acetic acid (Figure 3(b)). The pattern of sensors S1, S2, and S3 increases as the compound's ratio increases. However, S4 did not show such a pattern. Thus, the MQ8 sensor could be emitted for the following study.

Sensor performance was optimized using ensemble regression analysis. The purpose of ensemble regression was to combine multiple models in learning problems to improve prediction accuracy for numerical target variables. The ensemble learning process can be divided into three phases: the generation phase, the pruning phase, and the integration phase. The ensemble method has higher prediction accuracy compared to single models. The ensemble method is particularly useful when the dataset contains linear and nonlinear data types, different models can be combined to deal with this type of data. The regression curves of sensor data toward acetic acid ratio using ordinary least square, AdaBoost, Bagging, GradientBoosting, and Random Forest regression for 80% of the data were shown in Figure 4.

Acetic acid ratio	S1	S2	S3	S4
25%	3.63	4.37	3.89	4.24
25%	3.56	4.38	3.73	3.97
25%	3.65	4.47	3.4	3.87
25%	3.57	4.46	3.43	3.84
25%	3.5	4.49	3.38	3.83
50%	3.4	3.49	2.9	0.37
50%	3.3	3.13	2.66	0.35
50%	3.28	3.22	2.29	0.44
50%	3.34	3.61	2.47	0.57
50%	3.38	3.5	2.25	0.48
75%	2.69	2.38	1.59	0.65
75%	2.96	2.48	1.76	0.48
75%	3.02	2.56	1.94	0.46
75%	2.89	2.46	1.93	1.06
75%	3.01	2.66	2.1	0.83

TABLE II. Sensor output at minute 29th measurement.

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Figure 2 Radar plot of acetic acid-ethanol mixture.



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Figure 3. Pair plot of sensor signal based on the (a) acetic acid ratio and (b) ethanol ratio.



Figure 4. Regression curve of sensor responses as acetic acid ratio function using (a) ordinary least square, (b) AdaBoost, (c) Bagging, (d) GradientBoosting, and (e) Random Forest regression.



Figure 5. Regression curve of sensor responses as ethanol ratio function using (a) ordinary least square, (b) AdaBoost, (c) Bagging, (d) GradientBoosting, and (e) Random Forest regression.

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The determination coefficients of AdaBoost and GradientBoosting regression for training data were 1. This means the sensor data entirely correlates with the acetic acid ratio. The ordinary least square (OLS) regression has the lowest determination coefficient. The low determination coefficient was because OLS involves all the data in regression models. The presence of outliers in the input or output data decreases the determination coefficient significantly. On the other hand, ensemble regression prunes insignificance estimators to obtain better regression models [21]. A similar phenomenon was observed for ethanol regression models, as shown in Figure 5. The trade-off between accuracy and variance must be considered in regression analysis.



Figure 6. (a) Ethanol determination coefficient of training data and (b) mean recovery of test data.

The balance between the determination coefficient and the accuracy of test data was studied by comparing the recovery of predicted test data. The recoveries of 20% of the test data are shown in Figure 6. The ethanol recovery of AdaBoost was 111.38%, Bagging was 99.95%, Gradient Boosting was 154.16% and Random Forest was 97.84%. Thus, in terms of recovery, AdaBoost and Gradient Boosting were not reliable in predicting ethanol test data. The suitable regression models for ethanol determination in this gas sensor array were Bagging and Random Forest with 0.91 and 0.94 determination coefficients, and 99.95% and 97.84% ethanol test recovery, respectively.



Figure 7. (a) Acetic acid determination coefficient of training data and (b) mean recovery of test data.

Acetic acid test recoveries were 112.41%, 93.87%, 100.56%, 82.27%, and 101.38% for ordinary least square, AdaBoost, Bagging, GradientBoosting, and Random Forest regression, respectively. The determination coefficients of acetic acid training datasets were 0.65, 1.00, 0.89, 1.00, and 0.93 for ordinary least squares, AdaBoost, Bagging, GradientBoosting, and Random

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Forest regression, respectively. Thus, the same conclusion can be drawn, Bagging and Random Forest regression models have a good balance in training data determination coefficients and test data recovery [22].

The sensor responses differed depending on the type of doping ion used. The presence of doping ions in the metal oxides reduces the depletion layer of semiconductors [23]. Cheng et al., 2014 used doping in the form of a rare earth metal, yttrium, for the detection of acetic acid. The study proved that 5 wt% Y-SnO₂ had a higher response than pure SnO₂. The increase in electron density may occur due to a decrease in adsorbed oxygen ions when the temperature is more than 300° C. This can reduce the gas reaction [24].

4. CONCLUSION

The quantification of the acetic acid-ethanol mixture using a commercial gas sensor array has been conducted. Four gas sensors, MQ-3, MQ-4, MQ-6, and MQ-8, were optimized to obtain signal patterns to quantify the ratio of the acetic acid-ethanol mixture. Ensemble regression methods, i.e., AdaBoost, Bagging, GradientBoosting, and Random Forest regression performance in predicting test data, were optimized and compared with linear regression or ordinary least square regression. The determination coefficients of 80% of the training datasets and the recoveries of the 20% test datasets were studied. Regarding the determination coefficient, AdaBoost and Gradient Boosting regression correlated very well between sensor outputs and acetic acid-ethanol ratio. Regarding test recovery, Bagging and Random Forest regression have the best recovery, almost 100%. Based on their accuracy and variance trade-off, Bagging and Random Forest regression performance that the other regression models.

In summary, the results of this research demonstrate the possibility of practical applications of the gas sensor array for the detection of ethanol and acetic acid mixtures in quality control, safety measures, medical diagnostics, environmental protection and various industrial applications. Accurate and timely detection helps improve processes, implement safety protocols, and promote overall well-being in various areas.

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