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Rapid detection of ethanol in beverages using IIUM-fabricated electronic nose

[1]Nurul Asyikeen A.M., [1,2*]Jaswir, I., [3]Akmeliawati, R., [3]Ibrahim, A.M.,[3]Aslam, M. and [4]Octavianti, F.

[1]Department of Biotechnology, Faculty of Engineering, International Islamic University Malaysia, Jalan Gombak, 53100 Kuala Lumpur, MALAYSIA
[2]International Institute for Halal Research and Training (INHART), International Islamic University Malaysia, Jalan Gombak, 53100 Kuala Lumpur, MALAYSIA
[3]Department of Mechatronic, Faculty of Engineering, International Islamic University Malaysia, Jalan Gombak, 53100 Kuala Lumpur, MALAYSIA
[4]Faculty of Dentistry, Universiti Islam Sains Malaysia (USIM), Level 15, Tower B, Persiaran MPAJ, Jalan Pandan Utama, 55100, Kuala Lumpur, MALAYSIA.

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Abstract

This study has been successfully conducted to develop a method for rapid detection of ethanol (EtOH) concentration in beverages using Portable Electronic Nose (E-Nose) developed by International Islamic University Malaysia (IIUM). E-Nose is widely used in food analysis. However, E-Noses used in the food industry are big and not portable. The very recently developed portable device used in this study is very handy and practical for use. Results from this study revealed that the device could be used for rapid detection of ethanol concentration in various beverages such as alcoholic beverages, isotonic drinks, soft drinks and fruit juices from different brands sold in Malaysia. From the result obtained, it was shown that the device has high accuracy and reliability where it could detect ethanol concentration as low as 0.1% (v/v). The analytical condition for the detection was achieved with the lowest voltage output of 0.43V. While for optimization analysis using Response Surface Methodology (RSM), optimum Headspace Generated Time (HGT) and bottle's volume (mL) obtained are 0.66h and 100 mL, respectively.

Introduction

An E-Nose is a device that imitates the principle of human's olfactory mechanism for vapor detection of any volatile compound (Wilson and Baietto, 2009; Raj *et al*., 2012). E-Nose is an important device used in the Food Industry and can give many benefits to the consumers. Numerous studies have been done to analyze the characteristics and quality of beverages such as dairy products (Labreche *et al*., 2005; Raj *et al*., 2012) soft drink (Concina *et al*., 2010), fruit juices (Gobbi *et al*., 2010), instant coffee (Pornpanomchai *et al*., 2010) and the most common research conducted are for alcoholic beverages (Lozano, 2006; Garcia-Martinez *et al*., 2011).

The differences between this project with other existing researched are that the E-Nose developed for the detection of EtOH in this study is the first of its kinds and it was used to detect EtOH content in beverages of different brands sold in Malaysia. Apart from that, the process parameters were optimized using RSM from the Design Expert 6.0 software.

This research is important for the consumer since the IIUM fabricated E-Nose was designed as a portable one that people can bring the device along and test for the EtOH content in their beverages. Moreover, a fully portable device for volatile compounds detection with very sensitive sensors and on-line analysis tools is needed in the market (Özmen and Doğan, 2009). The significance of this research is to calibrate the newly developed portable E-Nose and validate as well as screen the beverages for the presence of EtOH content. One of the major problems in Malaysia's market is that there might be some products consist of EtOH content without being mentioned on the product's label (Azah *et al*., 2008). Apart from that, the objective of this research is to optimize the condition parameters, for how low does the EtOH concentration can be detected by the E-Nose. These voltage outputs will be saved in a database and later analyzed using Design Expert software.

For a clearer overview about the portable E-Nose, it can be summarized by showing its architecture

*Corresponding author.
Email: *irwandi@iium.edu.my*

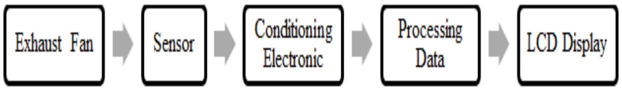

Figure 1. Architecture of electronic nose for alcohol detection

Figure 2. The portable E-Nose prototype (Length=21cm, width=7 cm, thickness= 5 cm)

designed as in Figure 1 which was implemented on the detection of EtOH content and Figure 2 shows the portable E-Nose prototype after it was finished being fabricated.

Materials and methods

Experimental setup

To set up the experiment, first EtOH dilution (0.10%, 1.0%, and 10% (v/v)) was put inside the Schott bottles of the desired volume (100 mL, 250 mL or 500 mL), sealed with aluminum foils and tightly fasten by elastic bands. Then, the E-Nose was put on top of a funnel, through the foils and the oscilloscope was connected to the E-Nose so the voltage response (V) can be recorded. When taking the reading, the first fan was switched on to attract the vapor to the sensor.

Calibration

The way the calibration works is by saving the voltage response in the database in relations to the percentage of the concentration of EtOH being tested, which give the specific voltage. After the reading was constant with time (1-10 s for this particular studies), the data was saved for further reference. Before proceeding to the next test, the second fan was switched on to "wash away" the volatile compound left using ambient air.

Validation and screening

For validation and screening process, experiments were conducted after optimum HGT and bottle's volume were known from the optimization part in RSM. Two types of alcoholic beverages, shandy and beer for validation process; while for screening process: soft drinks, isotonic drinks and fruit juices of different brands were tested. The procedure for validation and screening process is simple. 50mL of those beverages were put into the optimum volume of the Schott bottle and were measured after an optimum HGT. Then, reading was taken after the E-Nose is ready and the concentration of EtOH is displayed on the LCD screen of the device.

Table 1. Experimental Design and Results for Calibration

Run Order	Experimental Values			Voltage (V)	
	HGT (h)	Volume (mL)	Concentration (%v/v)	Actual	Predicted
1	0.5	100	0.1	0.43	0.43
2	0.5	250	0.1	0.40	0.40
3	0.5	500	0.1	0.40	0.40
4	1	100	0.1	0.40	0.42
5	1	250	0.1	0.40	0.39
6	1	500	0.1	0.40	0.40
7	1.5	100	0.1	0.40	0.41
8	1.5	250	0.1	0.40	0.38
9	1.5	500	0.1	0.40	0.39
10	0.5	100	1	0.55	0.54
11	0.5	250	1	0.52	0.50
12	0.5	500	1	0.50	0.50
13	1	100	1	0.52	0.53
14	1	250	1	0.50	0.50
15	1	500	1	0.48	0.50
16	1.5	100	1	0.53	0.53
17	1.5	250	1	0.50	0.49
18	1.5	500	1	0.48	0.50
19	0.5	100	10	0.90	0.87
20	0.5	250	10	0.75	0.82
21	0.5	500	10	0.80	0.78
22	1	100	10	0.93	0.92
23	1	250	10	0.88	0.86
24	1	500	10	0.85	0.83
25	1.5	100	10	0.97	0.97
26	1.5	250	10	0.90	0.91
27	1.5	500	10	0.87	0.88

Results and discussion

Calibration

The result of the response will be in terms of a voltage (V) read from the oscilloscope. After the E-Nose device has reached 0.4v (the zeros), reading can be taken by letting the device to detect the EtOH vapor. From the graph, it was observed that once the device detecting the vapor, the graph started to increase. The higher the EtOH concentration being tested, the more time is needed for the device to reach a steady state. Tables 1 and 2 represent the experimental design and results obtained; with actual and predicted values for voltage response is being highlighted.

Data were best fitted by the following regression model equation:

$$Voltage = 0.81 + 0.019^{*}A - 0.02^{9*}B + 0.23^{*}C + 1.156E\text{-}003^{*}A^{*}B + 0.029^{*}A^{*}C - 0.016^{*}B^{*}C - 1.111E\text{-}003^{*}A^{2} + 0.025^{*}B^{2} - 0.19C^{2} \quad (1)$$

Table 2. Validation and Screening

Types	Brand	EtOH Concentration, % (v/v)	Voltage, V (Checking Purpose)
Alcoholic Beverages	A	1.0-10.0	0.60
	B	>0.1 to <1.0	0.50
Soft Drinks	C	>0.1 to <1.0	0.45
	D	Alcohol Free	0.40
Isotonic Drinks	E	Alcohol Free	0.40
	F	>0.1 to <1.0	0.50
Fruit Juices	G	>0.1 to <1.0	0.45
	H	>0.1 to <1.0	0.45

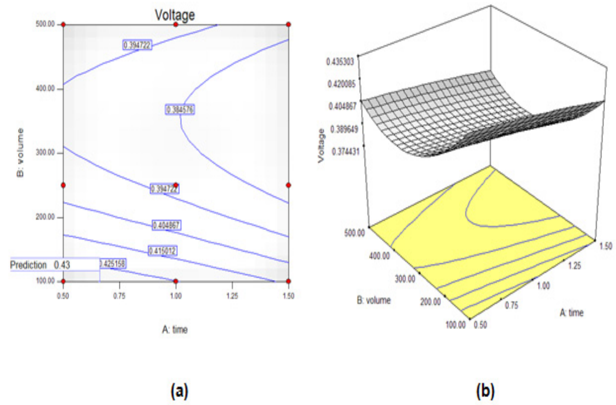

Figure 3. Optimization Graph (MinMax) (a) Contour Plot (b) 3D Surface Plot

The relationship between predicted and actual (experimentally) voltage response was observed, which most of the responses showed a close relationship between actual and predicted values. From the plotted graph, the coefficient of determination (R^2) value is 0.991, which proves that there are strong relations between the predicted and actual values and hence, the equation (1) developed is valid.

While from the design of experiment analysis, the correlation between the voltage output was 0.9959, showed a better correlation. On the other hand, R^2 from the analysis showed a result of 0.9919 which indicated the goodness of fit of the model. While for Coefficient of Variation (CV), the value obtained is considerably low with 3.89%, which signified a better accuracy and reliability of the experiments performed (Lu *et al*., 2009). Apart from that, the measured adequate precision obtained was 41.744 of the signal to noise ratio, which means that the process is desirable since the value is more than 4 (Singh *et al*., 2009).

Optimization analysis

Optimization analysis was done using RSM. Criteria for optimization were set in the beginning of the study. These parameters were set with the specific requirement because optimization process for this particular studies is concerning with the lowest detection of ethanol concentration which can be detected by the portable E-Nose. From 27 runs, the lowest concentration of EtOH detected was at 0.43V.

From all volumes obtained from the RSM, 100 mL bottle is available in the lab so the newly fabricated bottle is not needed. When the factor of HGT was considered, 0.66 hours is desirable since it only takes approximately 40 minutes for the VOC to be optimally generated.

The analysis was also done by analyzing the contour plot and 3D surface plot. The interactions between time, bottle's volume and EtOH concentration are quite significant from the elliptical shape of the plots. The optimum range of the parameters is shown by the present of a circle or elliptical form of the graph (Jamal *et al*., 2011). While for 3D optimization graph, it was well optimized with the maximum of the minimum value (MinMax). However, some of the data were accumulated at one side of the graph that makes the graph slightly slanted (Figure 3).

Validation and screening processes

Validation and screening tests were conducted based on the optimum time and volume analyzes with the help of Design Expert software. The results are shown as in Table 2. Validation test is important to validate whether the result displayed on the LCD of the E-Nose is the same with the one labeled on the beverages claimed by the manufacturer. Here, two samples were tested which were Brand A (5% (v/v) alcohol) and Brand B (<1% (v/v) alcohol) and gives desired results, 1.0 - 10% (v/v) and >0.1 to <1.0% (v/v) respectively. For alcohol that has less than 1% (v/v) alcohol, it had a mild scent and fewer bubbles produced while alcohol of higher concentration gave pungent smell and more bubbles produced when poured out into the beaker.

While for screened beverages, some that have no halal logo or logo claimed by the manufacturer itself (brand C and F to H), has a low level of alcohol detected, which the content was not shown on the label. However, soft drink for brand D has no halal logo on its label and after being tested, no alcohol detected in the beverage. While for an isotonic drink of brand E, it has a halal logo from the manufacturer but it has no alcohol available when being tested using the portable E-Nose. Both fruit juices contain alcohol in the drinks and this might result from the conversion of sugars into EtOH as one of its products.

Conclusion

The finding of this research is that the E-Nose can definitely give many benefits to the consumer, especially if the one design is portable that people can carry around for an on-line detection. The significance of having this kind of device is to check the availability of EtOH presence in beverages sold in Malaysia. The objectives to optimize analytical conditions and to test the accuracy and reliability of the device were successfully achieved, with the lowest voltage output of 0.43V had successfully obtained. Therefore, the lowest concentration which this device can detect is 0.1% (v/v). While for the optimization process, the optimum HGT and volume analyzed by the RSM were 0.66 h and 100 mL respectively. As for validation and screening part, the E-Nose could validate the alcoholic beverages and displayed the concentration in a range formed.

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