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Detection of Nitro-aromatic Explosives with Parametric Regression Modeling Approach

Dipali Ramdasi[1,*] and Rohini Mudhalwadkar[2]

[1]*Department of Instrumentation and Control, Cummins College of Engineering for Women, Pune India*
[2]*Department of Instrumentation and Control, College of Engineering Pune, India*
[1]*dipali.ramdasi@gmail.com*

Abstract

Nitrobenzene and Nitrotoluene are potential explosives and pose a threat to mankind. A data driven modeling approach is developed for detection of nitro-aromatic explosives in this paper. An Arduino based system is developed consisting of four gas sensors along with a temperature and humidity sensor. Data is transferred serially to a computer for model development. As the aroma of nitro-aromatic explosives contains ammonia from few parts per billion (ppb) to hundred parts per million (ppm), a parametric regression model for each sensor is developed for varying concentrations of ammonia from 200 ppb to 200 ppm. The parameters of Area, Slope and Relative Response derived from the response of each sensor are used to develop this model. Using the parametric regression model, a signature print of the sensor array is developed by subjecting it to varying concentrations of Nitrobenzene and Nitrotoluene. A multivariate linear regression model is developed using these signature prints to determine the presence of the explosives. The performance of the model is validated experimentally for mixtures of Nitrobenzene/ nitrotoluene with air. It is observed that the model identified the presence of nitroaromatic explosive correctly in 92 % cases.

Keywords: *metal oxide sensors, parametric regression model, multivariate regression model, nitroaromatic explosive*

1. Introduction

The complete world is under the threat of terrorist activities and use of explosives for destructive purposes is now very common worldwide. India has been suffering due to terrorist organizations being very active in the Indian territory. It has been observed that nitrogen-based explosives are used very commonly for terrorist activities in India. This paper presents a system developed for detection of nitrogen-based explosives, typically Nitrobenzene (PubChem CID 7416) and 4 Nitrotoluene (PubChem CID 7473) using a modeling approach. Literature suggests that these nitro-aromatic compounds possess explosive property and are spark detonable. [1-3]. These explosives when stored exhibit nitro-aroma. Detection of nitrogen compounds like Nitric Oxide, Ammonia and organic solvents like benzene can assist in deciding the presence of explosives. [4-6] The concentration of these Volatile Organic Compounds (VOCs) in the aroma of explosives is in few parts per million (ppm) or parts per billion (ppb). Considering the low concentration of these VOCs, a simple but effective technique for the 'in time' detection of nitroaromatic explosives, typically Nitrobenzene and Nitrotoluene, using the modeling approach is presented in this paper. Some of the commonly used spectroscopic techniques for in-house detection of explosives include Mass Spectrometry, Raman Spectroscopy, Ion

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*Corresponding Author

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Mobility Spectroscopy and Infrared Spectroscopy [7-10]. However, these techniques are confined to laboratories and have limited applications for on-field detection. Numerous scientists have worked upon enhancing the sensing mechanism for explosives and have developed specialized sensors for the target compounds [11-14]. Few researchers have developed sensors for nitrobenzene and nitrotoluene using graphite modified screen-printed carbon electrode [15], nanotubes [16] and NiCu alloy [17]. However, availability of such sensors is stringent to common people. The objective of this work is to make available a system, to common people for detecting presence of explosives, in markets, schools, hospitals and crowded areas. The developed system is simple, low cost and will identify the presence of targeted explosives and indicate its presence by popping up a message on the screen of a computer/ laptop.

The literature survey suggests that limited work has been carried out on metal oxide sensors for detection of Nitrobenzene and Nitrotoluene as potential explosives with nitro aroma. A market survey was done for availability of readymade sensors with high selectivity towards nitrobenzene and nitrotoluene, but such sensors are not available in the Indian market. Also, the study reveals that regression analysis is an effective tool for quantification and identification of compounds. This paper explains the system and procedure adopted to derive the parametric regression model which assists in identifying the presence of nitroaromatic compounds, typically, Nitrobenzene and Nitrotoluene. The system works in three stages, Data Collection, Model Development and Explosive detection. An ATMega2560 based Arduino board is used for interfacing the sensor array to the computer for data collection. The sensor responses are transmitted to a computer, where the parameters for modeling are derived from the sensor response. The parametric regression model developed from the sensor response for varying ammonia concentration is used to develop a signature print of the sensors for varying concentrations of nitrobenzene and nitrotoluene. For explosive detection, a multivariate regression model is developed using the signature prints. Once the model is generated, the system can be used for explosive detection. The system is exposed to gas samples of the suspicious area and the user of the system is notified of the presence of an explosive on a graphical user interface if the explosive is present. Figure 1 shows the schematic of the system used for model development.

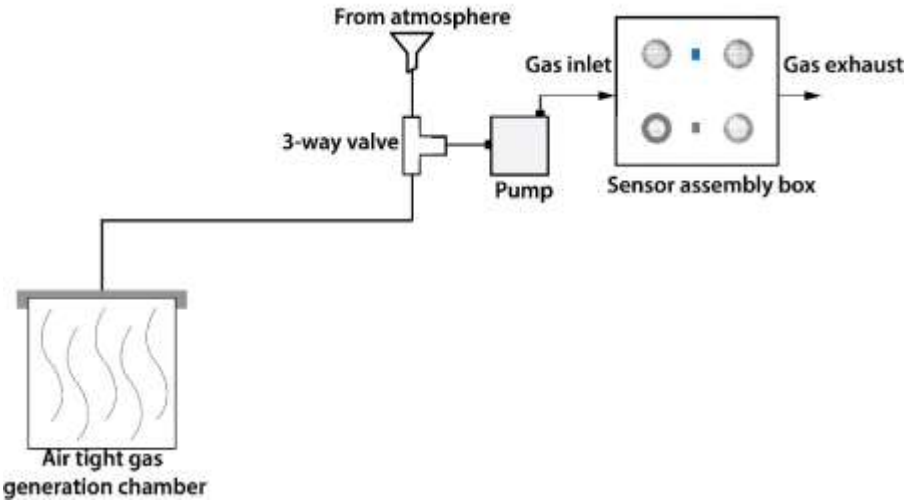

Figure 1. Schematic Diagram of the System for Model Development [23]

2. System Developed for Modeling

For the detection of Nitrobenzene (NB) and Nitrotoluene (NT), a system based on the ATMega2560 based Arduino board at operating frequency of 16MHz is developed [23]. A

sensor array consisting of four gas sensors along with a temperature and humidity sensor is interfaced to the system. The decomposition of Nitrobenzene and Nitrotoluene result into Ammonia, Methane, Nitrogen Dioxide, Isobutane and Hexane, where ammonia is the dominant element in both the reactions, with concentration ranging from a few ppb to hundred ppm. Hence the sensors sensitive towards the above elements are selected in the sensor array and are illustrated in Table 1.

Table 1. Sensor Array Configuration

Sensor Details	Specificity
TGS 826	Ammonia (Figaro)
TGS 2201	Gasoline, diesel exhaust gas (Figaro)
TGS 822	Organic Solvent vapors (Figaro)
MQ 135	Ammonia (Hanwei)
LM 35	Temperature
DHT 11	Humidity

The sensors are connected to the microcontroller through the on-chip ADC. The supply voltage and heater voltage for the gas sensors is set to 5 V. The general block diagram is illustrated in Figure 2. A 20 X 4 LCD display is used to display messages, the current temperature and humidity values. A 4 X 4 keyboard matrix is used to set parameters of temperature, cycle time and speed of motor driving the pump. The programming is done in the open source Arduino Software, which allows writing code in C and upload it on compiling to the board. Though the system is powered by 230V AC mains, provision is made for the system to operate on a 12V battery for portability purpose.

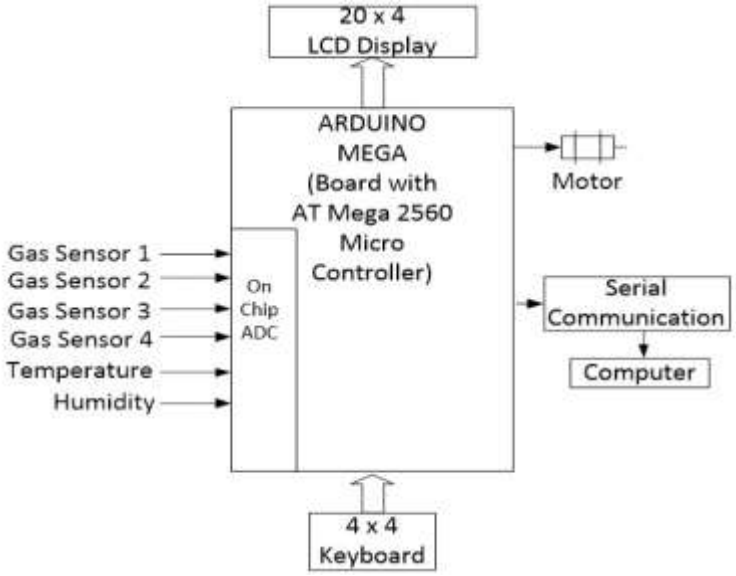

Figure 2. Block Diagram of the Data Acquisition System

The time for which the sensor array is exposed to a sample is programmable and can be set from the keyboard matrix. For modeling the sensor behavior, calculated amount of nitroaromatic compound in liquid form is used to generate the required concentration of VOC in the gas chamber. The motor and pump assembly enhances the flow of target gas on the sensor array. The motor for the pump is driven using an H-bridge drive. The measurement and control of physical parameters is described in detail in reference [23]. The sensor response is measured in terms of voltage, digitized and transmitted to a computer at a baud rate of 9600. The parameters of relative change in sensor response

(RR), area (A) and Slope of response are derived from the sensor response to develop the model. Figure 3 shows an example of real time sensor response and equations (1), (2) and (3) illustrates the derivation of these parameters. The Relative Response and Slope of the sensor response are calculated using the maximum value, minimum value and time taken to reach the maximum value (t), using equation (1) and (3). An average of 25 values is considered for calculating the minimum value.

$$RR(i) = (\max(i) - \min(i))/\min(i) \quad (1)$$

Area under the curve of the sensor response is calculated using the trapezoidal rule. An approximate numerical solution was used for generating the program using equation (2).

$$A(i) = \sum_{t=0}^{n-1} \frac{1}{2} [f_i(t) + f_i(t+1)]\Delta t \quad (2)$$

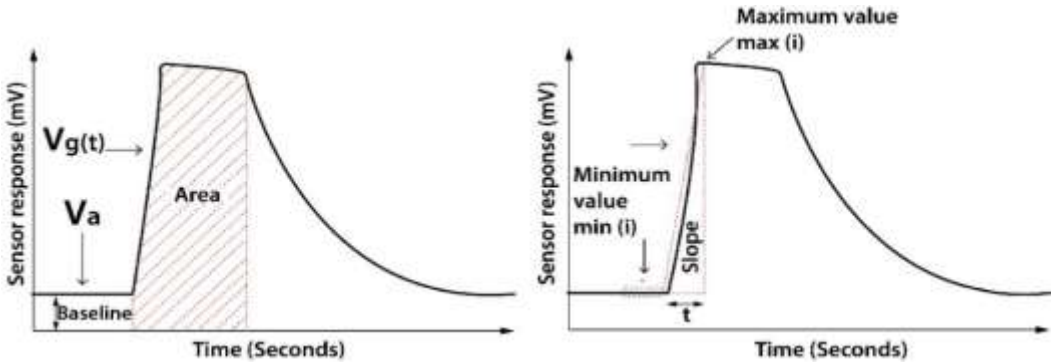

Figure 3. Derivation of Parameters from Sensor Response

A is the area, $f_i(t)$ is the response of sensor i at time t, $f_i(t + 1)$ is the response of sensor i at time t + 1, t=current time, n= total time for which sensor response is taken, Δt= time interval, preset to 110 millisecond.

$$Slope(i) = (\max(i) - \min(i))/t \quad (3)$$

A three-way valve is used to subject the sensors to ambient air or the gas. After every cycle, the sensor array is subjected to ambient air for flushing the earlier traces of test gas.

3. Experimentation

Rigorous experimentation is carried out to develop a system for explosive detection with a modeling approach. The various tasks carried out under experimentation are discussed in this section.

3.1 Sample Preparation

To generate the regression model for explosive detection, samples of volatile ammonia with concentrations varying from 200 ppb to 200 ppm are generated. For this purpose, liquor Ammonia of 30% concentration and a specific gravity of 0.89 is used. A high precision liquid chromatography syringe is used to inject diluted liquor ammonia in the air tight glass chamber with a volume of 2155cm^3 to achieve the desired concentration in volatile form. As the temperature, pressure and volume are known, the amount of diluted liquid ammonia needed to generate the desired concentration is calculated using ideal gas theory and explained below.

For 1 ppm ammonia concentration in a volume of 2.15 liters, 2.15µL of ammonia must be present, that is 1.492µg must be present according to standard temperature and pressure law. As 1µL of diluted ammonia contains 0.3µg, 4.972µL diluted ammonia is required to

be injected in the airtight vessel to obtain the concentration of 1 ppm. Table 2 shows the different concentrations of Ammonia and the amount of diluted liquid ammonia to be injected. Though 46 samples were generated, only 3 samples are illustrated as a representation of the concentration band.

Table 2. Ammonia Concentration and Volume of Ammonia in Volatile Form

Ammonia Concentration (ppm)	Volume of pure ammonia cm^3	Liquid ammonia to be injected µL
0.5	0.001076	2.485
10	0.02155	49.72
100	0.2155	497.2

3.2 Static Characterization of the System

The characterization of the system in terms of their sensitivity and response time due to pump speed and their positioning inside the test chamber is important to minimize the operational errors of the detection system. The pump delivering the gas sample to the detection system is operated for two speeds, 450 mL/ min and 250 mL /min, providing a pulse width modulated output of 100% and 40% for change of speed. A students t-test is conducted to study the comparison of response of each sensor. The air tight gas chamber is injected with liquor ammonia to generate 45 ppm concentration. After the standing time of 25 minutes, the sample inlet tube is placed into the jar, just before the pump starts. The maximum response and the time taken by the sensors to reach the maximum response are presented in Figure 4 and Figure 5.

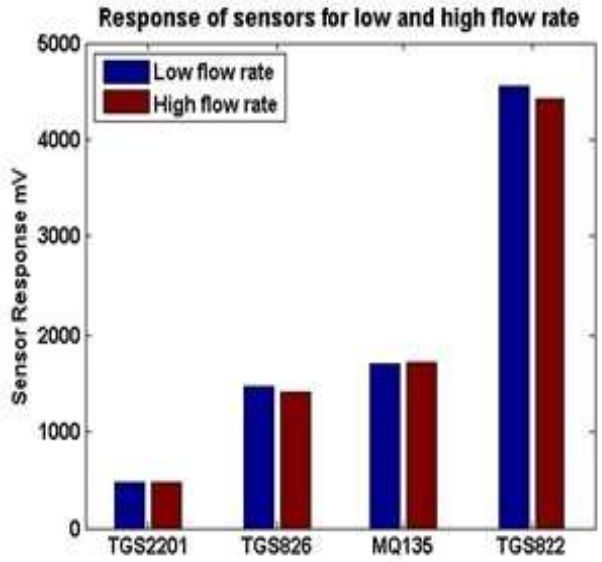

Figure 4. Effect of Flow Rate on Magnitude of Maximum Sensor Response

It is observed that except for TGS 822, the sensors did not exhibit a considerable difference in the maximum sensor response for both speeds. However, when the response time is considered, all the sensors exhibited a faster response time at higher pump speed. Thus, the maximum rated speed is used for further experimentation.

Also, the effect of positioning of sensors in the sensor assembly is studied. As the gas travels from a pipe and then enters the sensor assembly box, it may be subjected to fluid dynamic effects. It is observed that all the sensors showed no significant difference and the sensors were not sensitive to their position in the sensor assembly box.

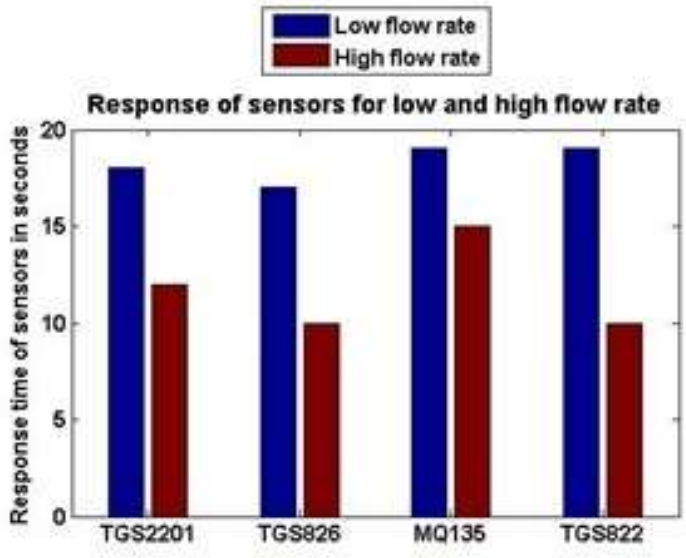

Figure 5. Effect of Flow Rate on Sensor Response Time

3.3 Derivation of Parameters from Sensor Response

After setting the operating temperature, pump speed and exposition time on the data acquisition system, the sensor array is either subjected to a known concentration of gas in the modeling stage or to an unknown suspicious sample of gas in the testing stage. The feature parameters of the sensor array response derived using equations (1-3) are further used for modeling or explosive detection.

4. Explosive Detection

In the developed system, presence of the nitroaromatic explosive, typically nitrobenzene or nitrotoluene is detected using the modeling approach. The general flow diagram of the system is illustrated in Figure 6.

4.1 Parametric Regression Model

Every sensor in the sensor array is subjected to 46 concentrations of ammonia varying from 200 ppb to 200 ppm and 552 data points were taken for Area, Relative response and Slope. A parametric regression model is generated for the system as explained in the book of Draper [24]. This model predicts the response of the sensors for any given Area, Relative response and slope.

Table 3. Performance Parameters of Parametric Regression Models for Sensors

	TGS 826	TGS 2201	MQ 135	TGS 822
RMSE	9.47	5.28	8.75	4.61
R^2	0.976	0.993	0.98	0.994

The model performance is verified using the values of coefficient of determination (R^2) and the histogram plot of residuals. The performance parameters of the model are presented in Table 3. The model performs with a Root Mean Square error less than 10 and a coefficient of determination greater than 0.97. The model performance is validated experimentally as well for 15 ammonia concentrations not used for calibrating the model.

It is observed that the model performs with a percentage absolute variation of approximately 10 %. The program flow for model development is illustrated in Figure 7.

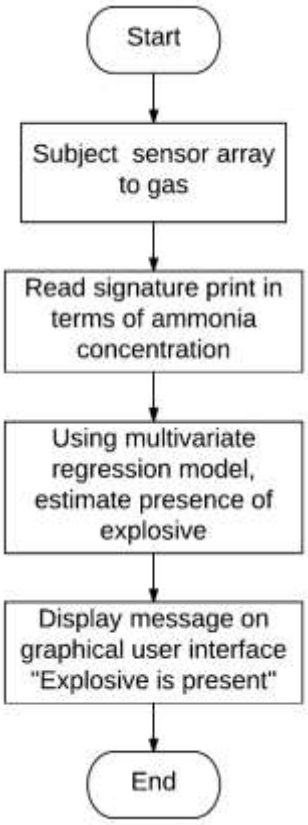

Figure 6. Flow Diagram for Explosive Detection

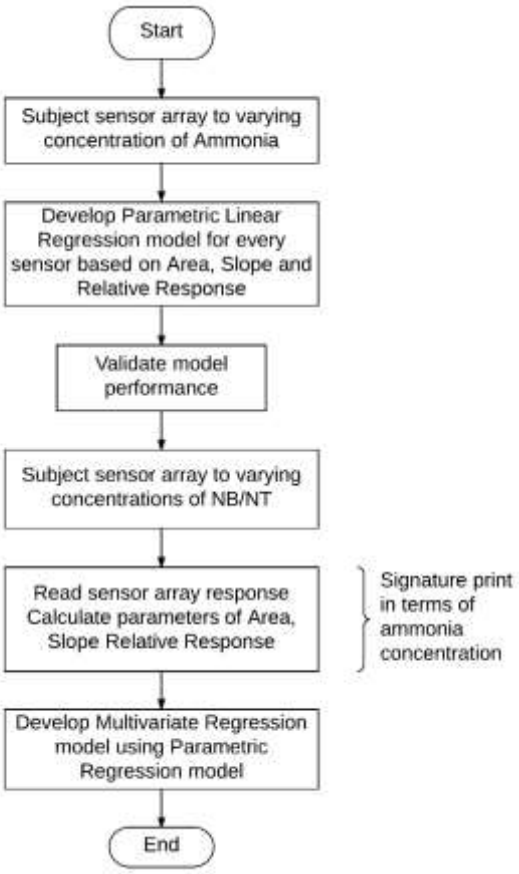

Figure 7. Flow Diagram for Model Development

4.2 Multivariate Regression Model

The parametric linear regression model for each sensor predicts the concentration of ammonia for any given sensor response parameters. For the detection of NB and NT, the response of all the sensors of the sensor array for varying concentration of Nitrobenzene (LOBA Chemie Pvt. Ltd. Mumbai, India) and Nitrotoluene (Research Lab Fine Chem Industries, Mumbai, India) are acquired. Using the parametric regression model, the signature print of the sensor array in terms of ammonia concentration is derived. This signature print (x1, x2, x3 and x4) is used to formulate a multivariate regression model for the dependent parameter of concentration of NB/NT (c). The signature print x1 is a function of Area, Slope and Relative Response calculated from response of sensor 1. To form the multivariate regression model, a database is created by acquiring the signature prints of the sensor array for 7 concentrations each of NB and NT, from 1 mL to 30 mL as volumes greater than this are dangerous to handle. The nitroaromatic explosive was injected in the air tight gas chamber (0.5 liters) and allowed to stand for 25 minutes. The compound is allowed to mix with normal air and not with a pattern gas. The aroma of the compound is allowed to pass over the sensor array for 20 seconds and sensors response parameters are calculated over three days for different time zones. This process is performed in triplicate to ensure repeatability of the observations and performance of the

system. Figure 8 and 9 illustrates the parameter of Area for Nitrobenzene and Nitrotoluene for 10mL.

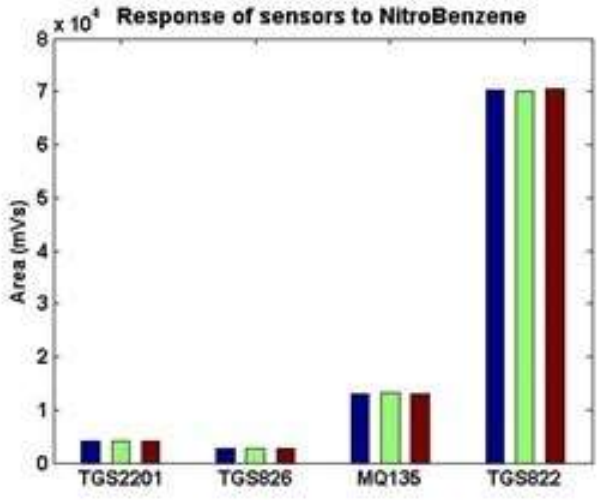

Figure 8. Response of Sensors to Nitrobenzene

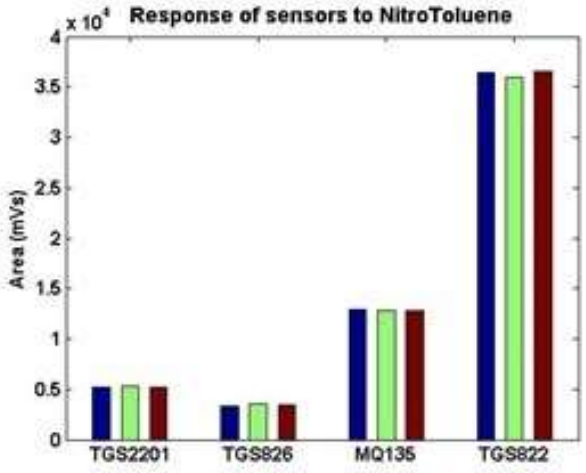

Figure 9. Response of Sensors to Nitrotoluene

After confirming satisfactory performance of the system with approximately equal area under the curve for all three sets, the sensor array is subjected to concentrations (in milliliters) of 1, 5, 10, 15, 20, 25, 30. The signature prints (x1, x2, x3, x4) in terms of concentration of ammonia are predicted using the parametric regression model. A multivariate model is formulated to estimate the concentration of nitroaromatic compound. This model is validated by injecting the same volumes of the nitroaromatic compound and observing the actual response of the model. The multivariate model is represented in equation (4), where x1 to x4 are the signature prints derived from the linear regression model, c is the concentration of the nitroaromatic compound, α and β are the regression coefficients and ε is the error term. [25]

$$c_{ij} = \alpha_j + \beta_j x_{ij} + \varepsilon_{ij} \quad (4)$$

For i=1,…,7; j=1,…,4

Each sensor thus predicts the concentration of Nitrobenzene and Nitrotoluene. Average of each sensor is calculated to decide the presence of nitroaromatic explosive. If the average concentration is a nonzero positive number, the nitroaromatic explosive is considered to be present.

Table 4. Results of the Multivariate Regression Model

Actual concentration in mL	Estimated concentration of Nitrobenzene by sensors				Mean predicted Concentration NB mL	Estimated concentration of Nitrotoluene by sensors				Mean predicted concentration NT mL
	TGS 826	TGS 2201	TGS 822	MQ 135		TGS 826	TGS 2201	TGS 822	MQ 135	
1	0.29	-0.72	4.78	1.53	1.47	-1.64	-2.78	2.1	-0.39	-0.68
5	6.06	5.12	6.16	7.49	6.21	3.18	6.39	4.36	4.78	4.68
10	8.95	10.96	7.47	7.49	8.72	14.09	12.52	9.65	11.25	11.88
15	15.21	16.81	10.74	13.44	14.05	17.39	15.58	13.9	16.44	15.83
20	21.47	19.73	17.26	19.39	19.46	20.94	21.7	20.98	20.32	20.98
25	24.35	25.57	25.61	25.34	25.22	23.98	24.76	24.52	24.21	24.37
30	29.65	28.5	33.96	31.29	30.85	28.04	27.82	30.47	29.38	28.93

5. Results and Discussion

The results of the developed system are observed at four stages. The gas sensor array connected to the Arduino system records the sensor response. This response is verified for each sensor using the Keithly 2110 multimeter. Also, the configuration of timers for real time is verified in the first stage. In the second stage, the sensor responses for varying ammonia concentrations are stored as excel sheets. Based on these responses, a parametric regression model is developed. This model is validated by estimating ammonia concentrations for samples with unknown values. The validation results show a maximum absolute variation of approximately 10%. Using this model, the signature prints of the sensor array in terms of ammonia concentration are acquired for varying concentration of nitrobenzene and nitrotoluene. This model is validated for concentrations of NB and NT which are not used for model development. This constitutes the third stage of observation of results. In the fourth stage, the complete system is run as a whole. The sensor array when subjected to normal air indicates absence of explosive, while the mixture of air and aroma of NB or NT indicates presence of explosive. The results of system are illustrated in Table 4. The percentage error in concentration estimation decreases with increase in concentration. As the main objective of this system is to detect the presence of explosive, the system performs successfully in 92% cases. If the mean concentration is a non-zero, positive value, a message in the GUI is displayed saying “Explosive is present”.

The developed approach is a novel method by which presence of an explosive can be detected. As each explosive or material used for developing explosives does not have readily available dedicated sensors for its detection, its timely detection is not possible. This approach enables nitroaromatic explosive detection in terms of signature print of a nitroaromatic compound, typically ammonia, nitric oxide or nitrogen dioxide. Once the

parametric regression model and multi-variate regression model are developed, the timely detection will be possible using the system. Even though the development of parametric linear regression model and multivariate regression model is time consuming due to sample preparation requirement of ammonia, NB and NT, the explosive detection algorithm executes in approximately 8 seconds after the signature print of sensors is acquired.

6. Conclusion

A modeling approach for detection of Nitrobenzene and Nitrotoluene as potential nitroaromatic explosives is developed in this paper. The system utilizes a sensor array for developing the system model in two stages, viz. Parametric Regression Model and Multivariate regression model. Once the data driven model is developed upon rigorous experimentation, the system is tested for presence of explosive. The system detects the presence correctly in 92% test cases.

Acknowledgement

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Authors

Dipali Ramdasi, is a post graduate from University of Pune and pursuing research at the Department of Instrumentation and Control, Government College of Engineering, Pune, India. She is a faculty member at the Maharshi Karve Stree Shikshan Samstha's, Cummins College of Engineering for Women, Pune, India. Her areas of interest are Gas sensors, Sensing systems, Embedded systems.

Rohini Mudhalwadkar, is a Doctorate from University of Pune and a faculty at Department of Instrumentation and Control, Government College of Engineering, Pune, INDIA. Her areas of interest are Sensor design, Analytical Instrumentation and Biomedical Instrumentation.

