Calibration and Characterization of MQ Sensors for E-Nose Application

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***Abstract*— The Earth's atmosphere comprises of various mixture of gases which includes Nitrogen (78%), Oxygen (21%) and trace gases (1%). Trace gas is a gas that makes up an extremely small portion of a mixture of gases and these trace gases include carbon monoxide, methane, carbon dioxide, hydrogen, argon, neon, etc. The concentration of these gases if increased slightly will cause a harmful effect on human health. These gases are emitted by different sources. So, there is a need for detection and concentration estimation of these gases. The concentration of trace gases present in the atmosphere has recently increased which will have an adverse effect on human health. Determining the concentration of these gases has become more crucial. Most of the gas sensors that are used to determine the concentration are not efficiently calibrated. So, a simple and effective procedure has been identified to calibrate these gas sensors and the same is applied for an E-nose application where concentration of gases emitted by the samples are determined and its characteristics are studied. In this work, an E-nose system has been developed by calibrating the array of gas sensors. The semiconductor gas sensors used are MQ3, MQ4, MQ7, MQ8. These sensors are used to sense the gases produced by the food samples and the signals from the MQ sensors are carried to the Raspberry Pi where the concentration of the sensed gases is determined in PPM using the calibration equation. These PPM values are displayed on the monitor through which the sensor characteristics can be studied using the graphs.**

Keywords — Calibration, Raspberry PI, Gas Sensor

I. INTRODUCTION

There are various atmospheric gases which make up air. The main gases are nitrogen and oxygen, which make up 78% and 21% of the volume of air respectively. Oxygen is utilised primarily by animals, including humans, but also to a small degree by plants, in the process of respiration (the metabolism of food products to generate energy). The remaining 1% of the atmospheric gases is made up of trace gases. These include the noble gases, very inert or unreactive gases, of which the most abundant is argon. Other noble gases include neon, helium, krypton and xenon. Hydrogen is also present in trace quantities in the atmosphere, but because it is so light, over time much of it has escaped Earth's gravitational pull to space. The remaining trace gases include the greenhouse gases, carbon dioxide, methane, nitrous oxide, water vapour and ozone, so-called because they are involved in the Earth natural greenhouse effect which keeps the planet warmer than it would be without an atmosphere. The abundance of a trace gas can range from a few parts per trillion (ppt) by volume to several hundred parts per million by volume (ppmv).[1] When a trace gas is added into the atmosphere, that process is called a source. There are two possible types of sources - natural or anthropogenic. Natural sources are caused by processes that occur in nature. In contrast, anthropogenic sources are caused by human activity. Some of the sources of a trace gas are biogenic, solid Earth (outgassing), the ocean, industrial activities, or in situ formation.[1] A few examples of biogenic sources include photosynthesis, animal excrements, termites, rice paddies, and wetlands. Volcanoes are the main source for trace gases from solid earth. The global ocean is also a source of several trace gases, in particular sulphur-containing gases. In situ trace gas formation occurs through chemical reactions in the gas-phase.[1]

A. GAS SENSORS

Recently, gas sensing, as a typical application in intelligent systems, is receiving increasing attention in both industry and academia. Gas sensing technology has become more significant because of its widespread and common applications in the following areas: (1) industrial production (e.g., methane detection in mines) (2) automotive industry (e.g., detection of polluting gases from vehicles) (3) medical applications (e.g., electronic noses simulating the human olfactory system) (4) indoor air quality supervision (e.g., detection of carbon monoxide) (5) environmental studies (e.g., greenhouse gas monitoring) [2]. There are different types of Gas sensors - Electrochemical Sensors, Catalytic Bead (Pellistor) Sensors, Infrared Sensors, PID Sensors, Metal Oxide Sensors /Semiconductor Gas Sensors (MOS), and Thermal Conductivity Sensors.

B. GAS SENSORS CALIBRATION PROCESS CONSIDERATIONS

Monitoring ambient conditions is a fundamental feature providing valuable data for both research and production applications. Any basic or advanced experimentation, any standard or crucial step in a production process demands a fine control over working conditions, thus implying the need of a proper monitoring apparatus. Along with temperature and pressure, the ability to check the atmosphere composition becomes relevant since the presence, absence or simply the amount modification of the gas species can strongly affect the final result. The calibration of the majority of commercial gas sensors can be executed by the product supplier or by the user. A calibration performed by the supplier requires a standard fee, to which the shipping cost of the sensor must be added. If the sensor cannot be shipped, or requires a specific calibration in situ, an operator performing the calibration is required and the related cost must be taken into account. The calibrated sensors are then used in the system to sense the gases produced by the food samples which is the E-nose Application through which the characteristics of the sensors are studied. Before trying to

calibrate the gases sensors, the next points must be taken in account which are stated below:

- Due to the low accuracy and repeatability of the gas sensor, each sensor must be calibrated, and it will have its own calibration parameters.
- The calibration of the gases sensors improves the behavior of the sensors, but it does not ensure high-precision response.
- The life expectancy of the gases sensors is about some month. After this period the gases sensors must be replaced and calibrated.

Paper [3] presents a method of calibration of gas sensors using LabView. Calibration is the process of obtaining the most accurate sensor data. The MQ gases sensors are produced to measure different types of gas at the same time but the datasheet formula for calibration is poor. To solve this, one thing to be known is to identify the measured substance and its formula. Here, the acquisition system consists in a sensors network, which transmit data to an acquisition board and to the computer. The identification of the calibration equation is used to calibrate gas sensors. The methods used to identify the calibration are linear regression and samples compare. The reason to study the calibration gas sensors is the error occurred after the data acquisition. Usually, the calibration formula is implemented on the acquisition board. In this project, calibration of gas sensor is implemented using LabView interface. The gases values data are collected with Arduino Mega 2560 board. To receive the data on the computer using LabView 2013, we tested two methods of data acquisition. One of them is using LIFA Base soft for Arduino and Arduino Toolkit Box.

In paper [4], a new temperature-independent set-up for gas sensors calibration making use of permeation tubes is proposed. Calibration is an important step in the evaluation and validation of the performance of gas sensors. Different methods and techniques can be used for producing calibrated gas concentrations. Among the others, permeation tubes show potential advantages for the wide range of compounds for which the method can be used. They work best for liquids with low boiling points and for gases that are liquified at low pressures and room temperature. Permeation tubes offer high accuracy for a range of concentrations from more than 5000 ppm to less than 50 ppb and a wide range of applications, such as in air monitoring, process control and quality, etc. The study and results related to generation of calibrated gas-phase ethanol concentrations (from 1 to 500 ppm) and the calibration of a MOS (Metal Oxide Semiconductor) gas sensor by this apparatus have been reported.

In paper [5], a sensor array for analyzing hydrogen and ammonia gas mixtures in humid air has been developed, built into a rugged system, and calibrated for laboratory testing. The sensor array is comprised of four chemically sensitive Field-effect transistors (CHEMFETs). Chemically sensitive layers for the sensors were developed and tested using a Kelvin probe. A combination of catalytic and noncatalytic thin layers (palladium and polyaniline) was selected for the four-sensor array. The work function responses of the CHEMFET sensor array to mixtures of hydrogen, ammonia, and humid air were measured. Chemometric multivariate methods, linear and nonlinear partial least squares, were used for the calibration of the sensor array using gas mixtures in the concentration range from 0 to 10,000 ppm hydrogen and ammonia in humid air. The sensor array for ammonia showed good sensitivity, selectivity, response time, and stability and is recommended for field deployment. In contrast, the sensor array for hydrogen, though highly sensitive to hydrogen, demonstrated inadequate stability, requiring further development before deployment is recommended.

In paper [6], a cascade of Artificial Neural Network (ANN) committees for the quantification of mixtures. In this structure the committees first analyze the gases that have a better regression and then pass the predicted concentration to the other committees, thus improving the information available for the most difficult gases without increasing the complexity of the ANNs. To test the structure, it was experimented with three different gases: CO, NO2 and NH3. The gas flows were controlled by an automated system that also controlled the environmental conditions and mixed the gases delivering them onto the measurement cell where three small commercial sensors were placed. The sensor data were later analyzed and different calibration methods, such as Partial Least Square regression, committee of Artificial Neural Networks and the cascade of committees of ANNs were evaluated with their measurement uncertainty and compared among them.

Paper [7] is presented to monitor the nature of the air. The proposed sensor structure checks the Carbon Monoxide, dust particles and humidity estimations of the encompassing air. It will notify to the people about the danger they will confront. This paper focuses towards helping allergic patients by familiarizing them with the gadget that can secure them from grave circumstances that happen when the air quality reaches beyond sustainable unit. This framework screens air quality and has a microcontroller, interfaced with gas sensors, optical dust particle sensor, humidity and temperature sensor. This arrangement has different units: a detecting unit, processing unit, power unit, and a communication unit. The results are acquired using the Arduino, MQ-7 gas sensor, DHT-22 humidity and temperature sensor, GP2Y1010AU0F optical dust particle sensor and the GSM module. This work will apply the procedures of electrical building with information of ecological designing by utilizing sensor systems to quantify Air Quality Parameters.

Paper [8] reports on energy efficient and low- cost gas sensor nodes for spatially resolved, accurate gas measurements in the event of a disaster. This is achieved by the integration of various low-cost and low-power sensors for gas, temperature and humidity combined with intelligent signal processing and information fusion. The relative position of the sensor nodes are needed to perform a spatially resolved gas measurement. For this, a low-cost localization system for the relative positioning of the sensor nodes is implemented. A map can be created, where

spatially resolved gas concentrations and the gas sources can be displayed.

II. Proposed Methodology

The above figure shows the general block diagram of the implemented system. The MQ Gas sensor is connected to the development board (Raspberry pi)through an ADC(Analog to digital converter) . As the Raspberry Pi cannot interpret the analog data from the senor, this has to be converted to digital form and then fed to the development board. The gas sensor senses the gas and the appropriate analog value is given to the ADC and then converted to the corresponding digital value. Finally, this digital value is passed on to the Raspberry pi and the value is displayed on the monitor.

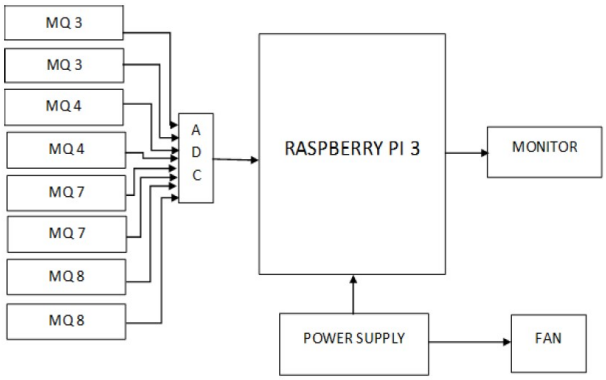


Figure 1: Proposed System

Calibrating an instrument involves comparing the measurements of two instruments; one with a known magnitude or correctness (standard device), against which you measure the unit under test. Calibrations are performed using only a few calibrators to establish the correlation at specific points within the instrument's operating range. While it might be desirable to use a large number of calibrators to establish the calibration relationship, or "curve", the time and labour associated with preparing and testing a large number of calibrators might outweigh the resulting level of performance. From a practical standpoint, a trade-off must be made between the desired level of product performance and the effort associated with accomplishing the calibration.

The instrument will provide the best performance when the intermediate points provided in the manufacturer's performance specifications are used for calibration; the specified process essentially eliminates, or "zeroes out", the inherent instrument error at these points. Figure 2 represents MQ-4 sensor interfacing .The Analog output of the Semiconductor gas sensor is received by ADC. This ADC has a resolution of 10 bits per channel, this ADC communicates with Raspberry Pi in SPI protocol. It has 4 pins MISO,MOSI,CS,SCLK. This works as Master and Slave, here ADC is the slave and it is synchronized to the Raspberry's clock. When Raspberry Pi has to send data, it uses MOSI channel and when it has to receive MISO is used. CS is called channel select which is used to choose a particular channel for input in the ADC.

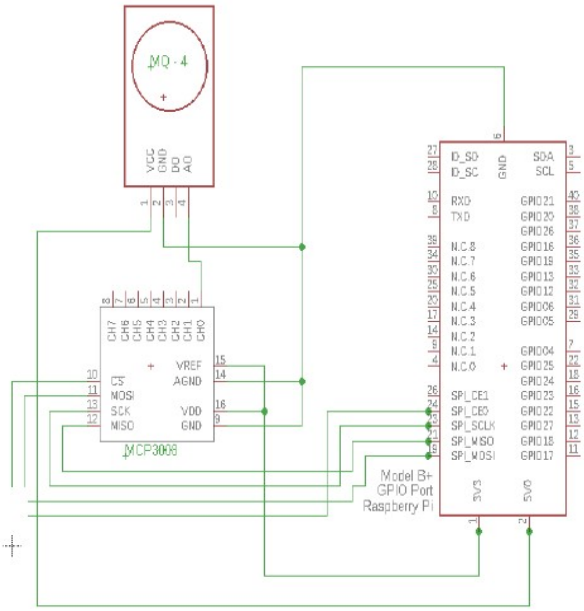


Figure 2: MQ-4 sensor interfacing

III. Flow Diagram of System

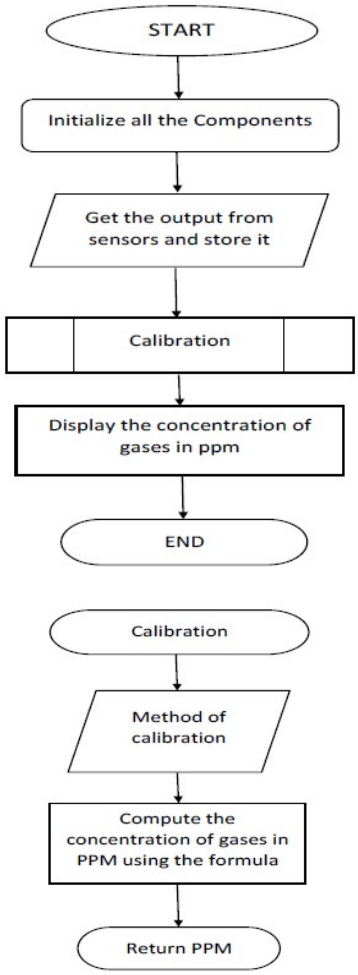


Figure 3: Flow Diagram of proposed system

The flow diagram shown goes by specific intervals.

At the first step of the flow diagram, the setup or the components are initialized. By initializing it means that all the components in the system are being powered up for the beginning of their operation.

• After the initialization of the components, the sensors starts sensing the gas that is present around the environment.

• These sensed values are passed on to the Raspberry Pi through an ADC MCP3008 which converts analog values into digital values and these values are processed and stored in the Raspberry Pi.

• The calibration method is then utilized here , wherein the processor outputs the concentration of the gases in ppm according to the equations used in the Python code. The graphs are then plotted for the ADC and ppm data obtained which specify the concentration of gases detected by the sensors.

• The flow diagram of the system shown in Figure 3. represents the working of the whole system and is capable of detecting and analysing multiple gases unlike the products that are available in the market.

IV. Calibration Procedure and Results

The MQ series gas sensors are resistive chemical sensors. These sensors make use of small resistances and thus gets a higher flow of current, getting a noticeable amount of heat which is used to burn the air to give an analog reading. All the MQ series gas sensors require to be calibrated. These sensors are non-linear and therefore they require to be calibrated accurately. The value of the sensing resistance varies depending on the concentration of the target gas and hence the voltage across it also varies. This varies the output voltage across the load resistor even though the load resistor remains constant.

Without proper calibration, the interpretation of raw analog reading of the sensor is meaningless. The basic measuring circuit for any gas sensor is shown in the circuit. Vc is the circuit voltage which is applied across the sensing resistance (Rs) and the load resistance (RL). The analog values given by the sensor represent the output voltage across the load resistance (RL). The load resistor is in series with the sensing resistor (Rs).

A. Calibration of MQ-8 Sensor

The Hydrogen concentration is a non-linear function of the normalized ratio, which is defined as Rs/Ro, where Rs is the sensing resistance and R0 is the sensing resistance at 100ppm of Hydrogen. Without considering the influences of temperature and humidity, the sensitivity characteristic curve can be used to obtain the concentration of Hydrogen in ppm. The curve is approximated by a straight line to extract the equation which describes the Hydrogen concentration in PPM. With the consideration of temperature and humidity, the value changes.

The sensitivity characteristic curve which is taken from the MQ-8 datasheet is as shown in the below Figure 4 and it is a plot of Rs/R0 vs. Hydrogen concentration in PPM. The characteristic curve plays a major role in calibrating the sensors. The R0 value for a Hydrogen gas sensor can be obtained by exposing the sensor to 100ppm of the Hydrogen a clean environment and noting the corresponding analog output value. The value of sensing resistance is then calculated using this analog output.

Steps to get the Hydrogen concentration in PPM:

• The sensitivity characteristic curve is considered from the data sheet, using which the values of Rs/Ro for a particular PPM is obtained.

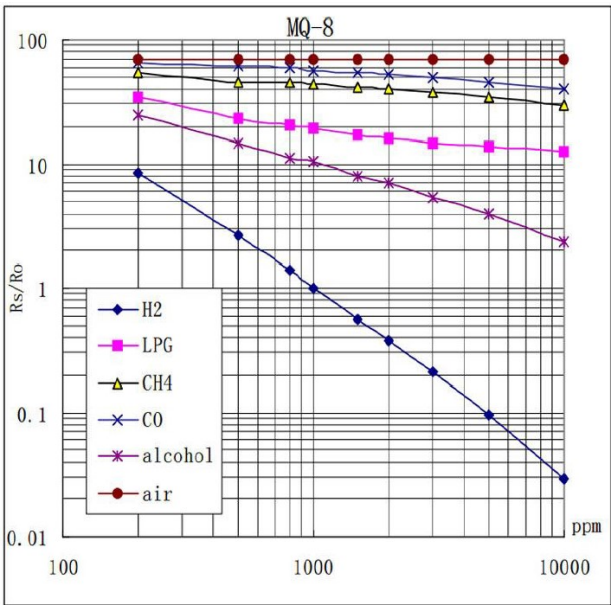


Figure 4: Response curve of MQ-8

B. Case Study

To study the behavior of the prototype a case study is done considering the food samples. Food samples are kept inside the testing environment of fixed volume where the MQ sensors are mounted on the top section of the testing environment. These food samples emit certain gases which are detected by the MQ gas sensors. The concentration of the gases produced by the samples are calculated. For the case study, few food samples were considered whose testing was done for few days to infer the concentration of the gases produced by each sample during different days. Here sample 1 was considered on day 1. The sample used here is Chickpea which was soaked overnight before testing. The concentration of the gas produced by sample 1 was tabulated and graphs were plotted for the same to show the variations in the gases sensed by MQ sensors. Figure 5 shows the prototype with sample 1.

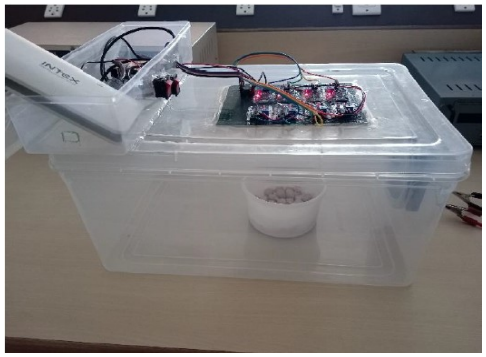
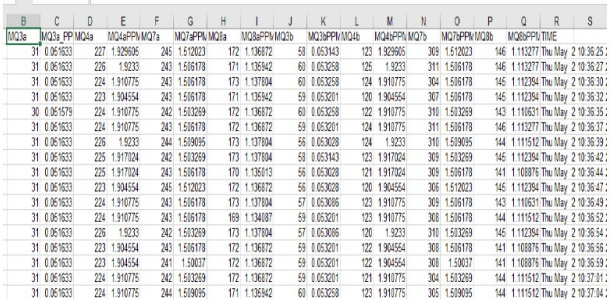


Figure 5: Prototype with sample-1

Initially the prototype was powered ON and the data when no sample was kept was recorded. After a while sample 1 was kept inside the testing environment. The sensors recorded the concentration of the gases produced by the sample 1. Sometime later the sample was taken out of the testing environment and the exhaust fan was switched on to remove the gases from the testing environment. Graphs were plotted for the recorded ADC values and their corresponding PPM values.

Figure 6 shows the ADC and PPM values of the gases produced by the sample 1 under test.



MQ3a	MQ3a_PPM	MQ4a	MQ4aPPM	MQ7a	MQ7aPPM	MQ8a	MQ8aPPM	MQ3b	MQ3bPPM	MQ4b	MQ4bPPM	MQ7b	MQ7bPPM	MQ8b	MQ8bPPM	TIME
31	0.061633	227	1.929606	245	1.512023	172	1.136872	58	0.053143	123	1.929606	309	1.512023	146	1.112277	Thu May 2 10:36:25
31	0.061633	226	1.9233	243	1.516178	171	1.136942	60	0.053258	125	1.9233	311	1.516178	146	1.112277	Thu May 2 10:36:27
31	0.061633	224	1.910775	243	1.516178	173	1.137004	60	0.053258	124	1.910775	304	1.516178	145	1.112394	Thu May 2 10:36:30
31	0.061633	223	1.904554	243	1.516178	171	1.136942	59	0.053201	120	1.904554	307	1.516178	145	1.112394	Thu May 2 10:36:32
30	0.061579	224	1.910775	242	1.503269	172	1.136872	60	0.053258	122	1.910775	310	1.503269	143	1.110631	Thu May 2 10:36:35
31	0.061633	224	1.910775	243	1.516178	172	1.136872	59	0.053201	124	1.910775	311	1.516178	146	1.112277	Thu May 2 10:36:37
31	0.061633	226	1.9233	244	1.519095	173	1.137004	56	0.053028	124	1.9233	310	1.519095	144	1.111512	Thu May 2 10:36:39
31	0.061633	225	1.917024	242	1.503269	173	1.137004	58	0.053143	123	1.917024	309	1.503269	145	1.112394	Thu May 2 10:36:42
31	0.061633	225	1.917024	243	1.516178	170	1.136913	56	0.053028	121	1.917024	309	1.516178	141	1.108876	Thu May 2 10:36:44
31	0.061633	223	1.904554	245	1.512023	172	1.136872	56	0.053028	120	1.904554	306	1.512023	145	1.112394	Thu May 2 10:36:47
31	0.061633	224	1.910775	243	1.516178	173	1.137004	57	0.053086	123	1.910775	309	1.516178	143	1.110631	Thu May 2 10:36:49
31	0.061633	224	1.910775	243	1.516178	169	1.134897	59	0.053201	123	1.910775	308	1.516178	144	1.111512	Thu May 2 10:36:52
31	0.061633	226	1.9233	242	1.503269	173	1.137004	57	0.053086	120	1.9233	310	1.503269	145	1.112394	Thu May 2 10:36:54
31	0.061633	223	1.904554	243	1.516178	172	1.136872	59	0.053201	122	1.904554	308	1.516178	141	1.108876	Thu May 2 10:36:56
31	0.061633	223	1.904554	241	1.50037	172	1.136872	59	0.053201	122	1.904554	308	1.50037	141	1.108876	Thu May 2 10:36:59
31	0.061633	224	1.910775	242	1.503269	172	1.136872	59	0.053201	121	1.910775	304	1.503269	144	1.111512	Thu May 2 10:37:01
31	0.061633	224	1.910775	244	1.519095	171	1.136942	60	0.053258	123	1.910775	305	1.519095	144	1.111512	Thu May 2 10:37:04

Figure 6: ADC and PPM values of sample 1 on day 1

The above tabulated values can be used to plot graphs which show the gases produced by the sample 1. Figure 7 shows the ADC values vs Time graph of the sample 1.

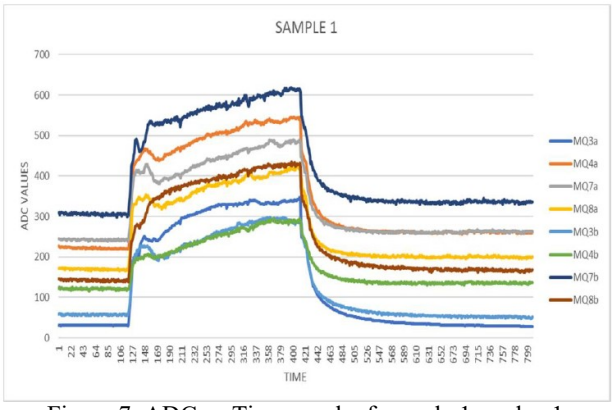


Figure 7: ADC vs Time graph of sample 1 on day 1

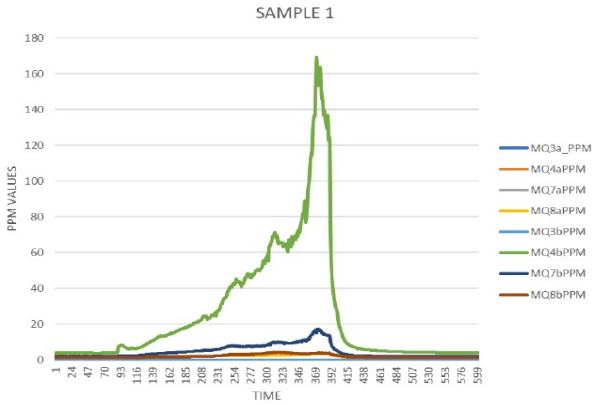


Figure 8: PPM vs Time graph of sample 1 on day 4

Figure 8 shows the PPM vs Time graph of sample 1 taken on day 4. It is observed that the PPM values have increased as compared to that of sample 1 taken on earlier days.

CONCLUSION AND FUTUREWORK

Calibration of gas sensors using calibrating equipment is more expensive. A simple procedure for calibrating gas sensors is discussed in this paper where we have obtained the power equation and through this we have derived the PPM equation which is later used in the code to determine the value of the gas in PPM. The paper restricts to calibration of MQ-3,MQ-4,MQ-7 and MQ-8 sensors which can be later deployed for various applications. Calibration of gas sensors using calibrating equipments is an expensive one as it costs too much. A simple procedure for calibrating gas sensors is discussed in this paper. Calibration procedures which involve temperature and humidity may give little more accurate results but they also increase the complexity. The proposed system for gas concentration detection and monitoring system can be used in number of daily life aspects. In order to get to know the changes of toxic and non-toxic gases in the environment. The values from the system stored in Raspberry Pi can be used in future depending upon the suitable applications . The proposed system can be used by laymen in all conditions and by using array of sensors a faulty sensor can be identified. The importance of gas sensing is set to grow with increasing requirements for safety and environmental protection across many industries. The current range of gas sensing technologies have served us well but the future holds many new possibilities. Power and size reductions and an improvement in ruggedness will allow a new generation of body worn devices. Ways will be developed to improve performance whilst at the same time reduce cost; new sensors will be targeted at enhancing the sensing capability.

The power consumed by pellistor and infrared types of gas sensors has limited their use in portable instrumentation to some extent due to battery capacities. In a very competitive market there is a constant pressure to reduce sensor price and manufacturers will be looking to minimise production costs using lean manufacturing techniques, automation or perhaps by manufacturing in low cost economies as is common for consumer electronics. Sensor designers continue to strive for improved performance in areas such as accuracy, drift, stability, cross sensitivity to other gases and response to environmental parameters such as temperature, pressure and airflow. Finally, the growth of body worn gas sensors will also demand an improvement in toughness for some sensor types. Technologies such as infrared have struggled to dominate the portable market as most IR sensors use a relatively fragile filament-based infrared source that is susceptible to shock such as dropping an instrument. The accuracy of the system can be enhanced by increasing the number of sensors used which can be realized by using multiple ADCs in order to increase the number of channels required.

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