

Abstract

There has been increasing amount of focus on research devoted to meliorate various sensor based measurement systems for developing quick, accurate, reliable, simple and cost effective systems. In particular, innovation in the field of sensor instrumentation and measurement is experiencing fast-growing demand, which is the key to create intelligent sensing devices. Notably, new technical interventions in this direction have profoundly advanced sensor based research, which facilitate to accurately interpret the physical phenomenon.

One of the most widely investigated sensors is metal oxide semiconductor (MOS) gas sensor or chemical sensor due to excellent reliability traits ingrained in them [10]. MOS gas sensors are chemoresistive sensors that were originally used to detect dangerous gases in mines and industries where conventional human testing is hazardous. In the last two decades, these sensors are increasingly used for detection of various volatile organic compounds (VOCs) and aroma assessment applications [11]. Typically an array of gas sensors using different material or different doping concentration is used to develop an electronic nose (E-Nose) system which mimics the human olfaction system [1]. Through the E-Nose, unique signatures from a particular gas are collected. The response patterns from the sensor array are then processed in pattern recognition software to identify the target gas.

MOS gas sensors have shown significant promise in tea aroma assessment [3-9, 18], by allowing the researchers to draw correlation between system's predictions with tea taster's evaluation. These reliable associations have been demonstrated to be useful for quick and accurate tea aroma evaluation, mostly using a computer assisted technology (CAT) based system. However, the CAT-based systems are mostly laboratory type, costly and power consuming, so are ineffective to be used as a field type testing gadget. Thus these systems can be considered as off line type, so the situation demands for a hand-held portable embedded E-Nose system for tea aroma prediction.

It is also reported by many authors that a simple RC circuit can be interfaced to a microcontroller (μC) as a direct interface (DI) to acquire the response pattern from sensor's yielding analog voltage as output (e.g. MOS gas sensor circuit) without intervening an analog to digital converter [2, 13, 16, 17, 19]. Although DI technique is adopted by various researchers to interface sensor analog output to the μC , it is mainly

concentrated on a single sensor, while sensor array interface by DI approach remains relatively unexplored. Therefore, another goal of this research is to develop an accurate and reliable technique for measurement of sensor array responses using direct interfacing circuit (DIC) and implement it in an E-Nose set up.

Objectives

The objective of this research work is threefold:

1. Design and implementation of a μ C based novel field-type, cost effective and low power consuming hand-held tea aroma assessment system using MOS gas sensors.
2. Design and implementation of a DIC based protocol for E-Nose as a modest method of gas classification.
3. An optimized error compensation technique to address the nonlinearity in DIC for analog sensors and estimation of uncertainty in DIC-based E-Nose.

With the aforementioned objectives, the research work was carried out, which is concisely discussed below.

Review of Literature

Pioneering work on CAT-based tea aroma classification in different possessing stages has been successfully established and also the correlation of the sensors responses with tea taster's score (grade) and analytical instrument was determined [3-5, 8, 9, 18]. The main disadvantage of these designs is the requirement of a heating source to generate the VOCs from the tea samples [3, 4]. Although, the most convenient way of heating the samples is by using illumination through a halogen lamp, however it requires a large power source, which affects the portability issue [3, 4].

Therefore, researchers are motivated to develop embedded system based E-Nose for tea quality prediction [6, 7]. However, requirement of various devices in odour delivery and refreshing of the sensor chamber makes them power consuming, bulky and costly. Even though the potential of MOS gas sensors for tea aroma prediction has been identified, these systems are inappropriate to be used as a field type instrument due to their large size which in turn has impeded their widespread acceptance. Thus the advance of μ C based E-Nose system enabling simple, cost effective, light weight and low power consumption is one of the aims of this research.

In the past, researchers have proposed cost effective methods of measuring analog voltage without the use of an ADC and directly interfacing the analog voltage to the μC , using a direct interfacing circuit. Such analog voltage measurement circuits, first demonstrated by Peter et al. [13] in a PIC 16CXX μC , are simple and remarkable in terms of performance. Over the decades, a number of such circuit designs were proposed for measurement of analog voltage [2, 13, 16, 17, 19]. Among them the most simple is demonstrated in [2], which shows that by using only two I/O pins of the μC an RC circuit based DI circuit can effectively measure analog voltage. The DI circuit requires only two resistances, a capacitor and the μC digital I/O pins to have tri-state capability.

However, the DIC model poses nonlinearity error and fails to accurately interpret the measured voltage in terms of count value. This affects the accuracy of the measured responses, and can sometime led to trivial interpretation. The minimization of nonlinearity (i.e., errors) in the DIC-based method not only relies on design parameter but also on the calibration techniques [15]. The extent of the nonlinearity indicates the reliability of design model for real-time implementation [14, 15]. Thus an appropriate model is necessary to achieve the lowest possible value of this parameter. Reverter et. al in [15] adopted three such techniques in the context of accuracy and resolution of direct resistive sensor-to-microcontroller interfaces that yielded an error of 0.01%. In the context of achieving suitable measurement setup, various approaches estimated the possible nonlinearity in the interfacing circuits. In [14] the authors estimated the DIC-interfacing error in capacitance measurement. Recently, Kokolanski et. al in [12] proposed a μC -based interface circuit for inductive sensors. The method resulted in nonlinearity up to 0.3% of the full-scale span with 10-bit resolution. A simple direct analog-to-microcontroller interfacing method for measuring analog signal is demonstrated in [2]. The sensor analog voltage is effectively measured using two external resistances and a capacitor. Although it works similar to the measurement obtained using 12-bit ADC, the measured outputs deviate from that of an ideal one which introduces a significant level of nonlinearity. Despite potential benefit due to design simplicity, the research did not explore the use of efficient nonlinearity reduction strategy to enhance the viability of measurement.

Further, DIC implementation for multi-sensor applications with an efficient calibration technique is still in preliminary stage. To further improve the nonlinearity and wider viability of the approach for measurement processes, it is essential to adopt more robust and reliable calibration strategy which motivated this study. Therefore, another

goal of this research is to incorporate an error compensation algorithm in the μC for E-Nose that will instantly reduce the nonlinearity error during measurement.

The efficiency of the E-Nose relies both on the DIC parameters and sensor parameters such as gas concentration, exponent of power law etc and therefore the uncertainties associated with these parameters, their estimations and control are of prime importance to have better output responses. However some external sources such as ageing of sensors, sensor poisoning, and environmental variation, known as sensor drift etc., may influence the outputs which can be compensated by algorithms stated in [20-23].

Motivated by this an attempt is made to estimate the uncertainty in two phases: i) parameters that affect the signal generated by the sensor array on exposure to various gases, and ii) direct-interface hardware parameters. Uncertainty is a non-negative parameter that investigates the individual uncertainty in various parameters which contributes to the final uncertainty of the measurand.

Methodologies/approaches applied

In order to set the objectives a detailed literature survey was undertaken to understand the state-of-the-art techniques in the aforementioned research areas. The literature survey, background information and groundwork about the applied methodologies are detailed in Chapter 1, to establish the research foundation.

Most of the circuits used in the experiments were designed in DipTrace and fabricated in the laboratory itself. The MOS gas sensors used in the E-Nose was procured from Figaro, INC, Japan. Data logging and instrument control was accomplished using LabVIEW using a data acquisition (DAQ) card through a host computer. MATLAB was used for signal processing in the preliminary stages. The sensor data correlation was studied by performing cluster analysis like principal component analysis (PCA), linear discriminant analysis (LDA) and self organized feature map (SOFM) in MATLAB. For classification feed forward back propagation (FFBP) algorithm was coded in MATLAB. The FFBP model created was then implemented in a PIC 18F45K22 μC through which online discrimination is done. In this stage, the instrument control was also monitored by the μC . For coding the μC , MikroC PRO for PIC 6.4.0 compiler was used. The programs were verified, burned and tested in the μC using MikroElektronika EasyPIC v7 development board.

Phase-I: The proposed novel low-cost hand-held tea flavor estimation system.

In chapter 2, sensitivity analysis of 12 MOS gas sensors, to select the suitable sensors using a CAT-based E-Nose system, for tea flavor estimation is reported. The ability of the sensors to classify tea samples using artificial neural network (ANN) is also established. Further to overcome the sample heating problem inherent in CAT-based system a novel sample cum sensor chamber was designed, which was integrated in a μC based hand-held E-Nose system.

Experimental: The experiment was aimed at selection of the appropriate sensors, so, 12 sensors (TGS 813, TGS 821, TGS 822, TGS 825, TGS 826, TGS 832, TGS 2600, TGS 2602, TGS 2610, TGS 2611, TGS 2620 and TGS 2201) were used to examine the sensitivity to tea samples through repeated experimentation. Out of 12, only 4 sensors (TGS 2201, TGS 2602, TGS 2620 and TGS 832) were found suitable for tea aroma assessment. Dry and fresh tea samples from tea gardens were collected to avoid any discrepancy caused by humidity and adulteration affecting the actual sensor response. A total of 150 CTC tea samples of 5 different grades collected from different tea industries are used to perform the first level experiment in a CAT-based offline classifier. After selecting the sensors, the hand-held E-Nose was trained for online testing with 8 different classes of tea samples. The subjective grading and evaluation of the collected samples are performed by three reputed tea tasters on a scale of 0 to 10. The response patterns of 5 different graded tea samples were collected using a CAT-based E-Nose system. Features are extracted from the response patterns of the sensor array by subtracting the baseline voltage level from the peak stable voltage level. PCA is then performed to examine the existence of cluster. A three layer feed FFBP ANN (input layer, hidden layer and output layer) is used to examine the classification accuracy of the system. The best FFBP ANN is determined in MATLAB by varying the number of neurons in the hidden layer. The selected sensors are then integrated in the developed hand held system. Eight different grades of tea samples are used to collect the sensor responses to create the database. Similar to the CAT based system, features are extracted from the responses by subtracting the baseline voltage level from the peak stable level. PCA and SOFM are used to examine the cluster formation. FFBP ANN model is also investigated for the hand-held system in MATLAB to determine the best model. The ANN parameters of the best model are then coded in the μC to perform online testing of the tea samples. Repeatability test of the system is also performed.

Results and Discussion: PCA of the CAT-based tea classifier forms five distinct clusters to the five different grades of tea samples used. The FFBP ANN programmed in MATLAB to classify tea flavor shows an accuracy of 92 %. Therefore, the selected sensors are capable of successfully classifying the tea samples. The investigation of the responses from the hand held tea flavor estimation system using SOM and PCA shows eight distinct clusters to the eight different grades of tea samples used. The ANN programmed in μC shows an accuracy of 90%. Moreover, the proposed design setup eliminates the need of several components used in traditional E-Nose systems, like-micro pumps, mass flow controller, suction fan, solenoid valves and external heater. As a result, it reduces the power consumption rate. Further it minimizes the size, weight and cost of the system. To test the robustness of the system a repeatability test was conducted by testing samples in random manner over a smaller time period (3 days). The repeatability analysis reveals that the overall repeatability is 80% for a total of 35 inputs. Therefore, the repeatability of the system is found to be marginally excellent.

Phase-II: DIC based E-Nose for gas classification.

Chapter 3 focuses on application of a DIC with μC for measurement of responses from an array of MOS gas sensors and evaluation of its performance.

Experimental: The experimentation was aimed at development of a RC circuit based DIC for measurement of analog output response of a single sensor and then extending it for multisensory framework. The analog output is determined by measuring the charging and discharging cycle of the capacitive network. The delay provided in charging and discharging cycle is chosen in such a way that, the process adopted is able to measure the analog value in terms of count value similar to that of 12-bit ADC. Three MOS gas sensors are chosen based on prior works and their target gases to perform the experiments. The sensor signals are first collected for four different chemicals (acetone, acetic acid, methanol and 2-propanol) by injecting 200 μL of chemical in a sample vial. The sensing and purging time are determined by acquiring the responses using a LabVIEW based DAQ card in a computer. After proper setting the purging and sensing time the peak and the baseline voltages are collected using the DIC by the μC and transferred to a host PC via USB UART link. Features are formed by subtracting the count values for baseline from the peak. For cluster analysis PCA and LDA are used, and for classification task FFBP ANN is used. The best FFBP model is determined in MATLAB and then coded in the μC for online gas discrimination. The scalability analysis is also performed on the system by repeating the experiments by injecting 300

μL of liquid samples to generate new data set. PCA and LDA are also performed to visualize the cluster existence on the new dataset. ANN is then performed in the μC to determine the classification accuracy.

Results and Discussion: It was observed that the DIC characteristic agrees well with that of an ideal 12-bit ADC. However, the system suffers from nonlinearity, due to which a deviation from the ideal value is observed. PCA forms four distinct clusters of the four tested gases. The μC based DIC E-Nose is able to classify the four gases with an accuracy of 98.75%.

Scalability analysis reveals the following: distinct clusters are observed using PCA and LDA in the new data set; the FFBP ANN shows an accuracy of 98.75% to classify gas samples online. Therefore, the proposed methodology sheds new light on DIC method, presenting a promising solution for interfacing a sensor array producing analog voltage.

Phase-III: Nonlinearity compensation of DIC based multisensory measurement

It is observed that the DIC described in previous chapter works similar to the measurement obtained using a 12-bit ADC. However, the characteristics between input analog voltage and count value of DIC possess two distinct slopes depending on the value of input analog voltage and the digital logic high level of the μC . This nonlinearity results in a considerable amount of error in measurement in the DIC approach.

In Chapter 4 a potential solution to compensate nonlinearity in the measured output in an efficient way so as to accurately map the nADC to that of an ideal ADC is proposed. In doing so, several error compensation techniques were investigated to determine the best fit model for real time error compensation using μC . Further, the proposed framework is implemented in a multisensory E-Nose environment to investigate its effect on classification accuracy. The uncertainty caused by the sensor circuit and DIC or nADC (non ADC) parameters in measurement of output responses from a MOS gas sensor array is also estimated. In deriving such a setting, uncertainty principle is utilized to estimate ranges of the uncertainty of each parameter cause a deviation in measurement from the true value. Experimental investigation demonstrates that DIC interfaced MOS gas sensor based E-Nose incorporates various sources of uncertainty.

Experimental: The major issue in the DIC system is the significant level of nonlinearity due to external DIC components and the counter of the μC . Therefore, a set of error compensating techniques (ECT) were investigated and the best model was implemented

for single sensor measurement which reduced the nonlinearity to 0.02%. Under the improved ECT framework, the algorithm was extended for multi-sensor measurement in an E-Nose setup. A neural network model was integrated with the system which was implemented using a μC for effective discrimination of four different gases. The efficacy of the proposed model of ECT was enlightened by comparing with ADC based approach. The PCA of ADC, error compensated without ADC (C-nADC) and uncompensated without ADC (U-nADC) were compared and the Euclidean distance measures were also performed to determine inter/intra class distance ratio. Further, the effect of compensation on predictive accuracy was determined by comparing the online predictive accuracy of ADC, U-nADC and C-nADC based methods to classify four gas samples. Although the PCA and ANN classification validates the direct-interface E-Nose technology, there is a certain amount of uncertainty in the measurement of sensor response voltage (V_{RL}) due to uncertainty of the sensor circuit parameters and that of the DIC parameters.

Results and Discussion: It was observed from the experimental investigation that the nonlinearity prevalent in U-nADC framework is reduced to a great extent, by the proposed ECT. Therefore, the response characteristics obtained by measuring the analog voltage using the C-nADC framework in the range of $0\text{-}V_{DD}$ V, resembles the ideal 12-bit ADC. Moreover, in the multi-sensor E-Nose setup the sensor array responses measured by the C-nADC and the built-in 10-bit ADC of the μC are within a close approximation. Whereas, the sensor array responses measured by U-ADC significantly deviate from the ADC or C-nADC, which may be presumably due to its deviation from the ideal characteristics. PCA forms distinct clusters for U-nADC and C-nADC, and a partial overlapping is observed in case of ADC measures. It is observed that between n-ADC techniques, C-nADC provides more accurate measurement compared to U-nADC, so the effect of error compensation on the classification accuracy is evaluated. Further, performance of ANN for ADC, U-nADC and C-nADC is compared. In order to classify gas samples FFBP ANN is used. The feature datasets of the three approaches are partitioned as- 60% for training and 40% for testing. The number of hidden neurons (n) for the best classification performance is found to be 4 for implementation in ADC as well as for U-nADC and C-nADC. It is observed analysis that C-nADC results in higher classification accuracy (98.75%) than that of U-nADC (97.91%) and ADC (97.08%) based method in the multisensory E-Nose environment. The weights and biases of the

neural networks with optimal hidden neurons simulated in MATLAB are used to code the ANN algorithm in two separate μC s for online comparison of the proposed method. In one μC both the U-nADC and C-nADC based prediction results are calculated sequentially and displayed in a LCD, and in the other μC ADC based prediction is performed. The nADC and ADC based online gas discrimination of 200 gas samples (50 samples each gas) analyzed accounted for 95%, 96.5% and 98% accuracy for ADC, U-nADC and C-nADC respectively.

The scalability of the proposed approach is also examined in the context of results obtained using the optimized model on new set of data that has not been previously used for this measurement. In order to investigate the systems scalability the system is trained with separate dataset of the gas sensor responses and testing was performed. The experimentation is repeated for three cycles and accordingly performances have been investigated which is found to be satisfactory. The quantitative measure achieved in terms of average accuracy for ADC, U-nADC and C-nADC are found to be 94.66%, 96.833 and 98.16% respectively.

It is observed that the maximum uncertainty of the sensor circuit is contributed by exponent of power law (α), while in case of direct-interface circuit the maximum contributor of uncertainty is from capacitors (C). The extent up to which the individual input parameter will cause variation in the output responses of the sensors under controlled condition was estimated. This gave an insight about the overall deviation that may occur in the responses from which the interval in which the output remains can be interpreted. Further it indicates that the measured sensors responses reliably represent its true value. Since, the uncertainties are very low compared to the output the quality of the developed E-Nose system is accurate and reliable.

Organization of the Thesis

Chapter 1 presents the basic introduction, inclusive literature survey and foundation of the research problem. In *chapter 2*, the circuits, components and methods used to design and develop a hand-held tea quantification E-Nose system is given. *Chapter 3* describes a methodology to directly interface a sensor array to μC . This is exemplified through a MOS gas sensor array integrated in an E-Nose system. In *chapter 4*, the nonlinearities ingrained in the DIC are curtailed using a best fit error compensation technique. Further the uncertainties associated with the μC based DIC E-Nose system are

estimated. *Chapter 5* concludes the thesis with research achievements and future directions.

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