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Electronic nose based tea quality standardization

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Abstract

In this paper we have used a metal oxide sensor (MOS) based electronic nose (EN) to analyze five tea samples with different qualities, namely, drier month, drier month again over-fired, well fermented normal fired in oven, well fermented overfired in oven, and under fermented normal fired in oven. The flavour of tea is determined mainly by its taste and smell, which is generated by hundreds of Volatile Organic Compounds (VOCs) and Non-Volatile Organic Compounds present in tea. These VOCs are present in different ratios and determine the quality of the tea. For example Assamica (Sri Lanka and Assam Tea) and Assamica Sinesis (Darjeeling and Japanese Tea) are two different species of tea giving different flavour notes. Tea flavour is traditionally measured through the use of a combination of conventional analytical instrumentation and human or ganoleptic profiling panels. These methods are expensive in terms of time and labour and also inaccurate because of a lack of either sensitivity or quantitative information. In this paper an investigation has been made to determine the flavours of different tea samples using an EN and to explore the possibility of replacing existing analytical and profiling panel methods. The technique uses an array of 4 MOSs, each of, which has an electrical resistance that has partial sensitivity to the headspace of tea. The signals from the sensor array are then conditioned by suitable interface circuitry. The data were processed using Principal Components Analysis (PCA), Fuzzy C Means algorithm (FCM). We also explored the use of a Self-Organizing Map (SOM) method along with a Radial Basis Function network (RBF) and a Probabilistic Neural Network classifier. Using FCM and SOM feature extraction techniques along with RBF neural network we achieved 100% correct classification for the five different tea samples with different qualities. These results prove that our EN is capable of discriminating between the flavours of teas manufactured under different processing conditions, viz. over-fermented, over-fired, under fermented, etc.

Keywords: Electronic nose; Tea flavour; Odours

1. Introduction

1.1. Olfactory system and chemical senses

To humans, the sensation of flavour is due to three main chemoreceptor systems. These are gustation (sense of taste by tongue), olfaction (sense of smell by nose) and trigeminal (sense of irritation). Taste is used to detect non-volatile chemicals, which enter the mouth while the sense of smell is used to detect volatile compounds. Receptors for the trigeminal sense are located in mucous membranes and in the skin, they also respond to many volatile chemicals and it is thought to be especially important in the detection of irritants and chemically reactive species. In the perception of flavour all three chemoreceptor systems are involved but olfaction plays by far the greatest role with other two senses contributing much less to the overall perception (Dutta, Hines, Gardner, Udrea, & Boilot, 2003).

The smell sensation is a chemical and neural process wherein odorant molecules stimulate the olfactory receptor cells that are located high up in the nose in the olfactory epithelium. Odours are of two types, simple and complex. Nature of stimulus and not the quality of sensation distinguish these. A simple odour is one which consists of only one type of odorant molecule whereas a complex odour is a mixture of many, different types of odorant molecules. All naturally occurring odours are complex mixtures. Odorants are typically small hydrophobic, organic molecules containing one or two functional groups. The size, shape and polar properties of the molecules determine its odour properties.

Broad patterns of response are shown by the mammalian olfactory system consisting of a large number of non-specific receptors—with about 300 different olfactory binding proteins having been identified—in a total of about 50 million. These cells send their signals to secondary nodes and then cells located in the olfactory bulb. There is a marked convergence at this stage with between 1000 and

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20,000 primary receptor cells connecting to each secondary cell followed by limited divergence (Pearce, Gardner, Friel, Bartlett, & Blair, 1993). This suggests that the secondary cells be involved in the integration of information, i.e. impulses simultaneously from many input cells. The nature of the primary cells is non-specific in their responses whereas the secondary cells respond to distinct categories of odours. Secondary cells interact with each other and with higher cells as well. Thus the system is a complex non-linear one with both and excitation and local inhibition helping to produce a high degree of sensitivity (ppb or better) and specificity.

The sensors employed here in the electronic nose (EN) are metal oxide films coated across the gap between two thin gold electrodes to form a chemoresistor. Conducting metal chemoresistors respond to a variety of Volatile Organic Compounds present in tea (Dutta et al., 2003; Pearce et al., 1993).

1.2. Tea processing and its flavour

Due to large number of organic compounds present in tea, it is difficult to process tea to an absolute standard. The volatile compounds present in tea determine its quality. Table 1 (Bhuyan & Borah, 2001) describes different volatile compounds present in black tea. In conventional tasting, it is very difficult to keep a consistency in the standard of tea quality from batch to batch during a production process. The quality is ensured by a human taste panel, which may vary due to different factors.

The aroma and flavour are two quality factors of tea, which depend upon the number of volatile compounds present and their ratios. Human panel tasting is inaccurate, laborious and time consuming due to adaptation, fatigue, infection and state of mind. An EN can be a better alternative to conventional methods for tea tasting and quality monitoring during production process. An EN is an increasingly fast, reliable and robust technology. Tea industries all over the world presently use certain standard terminology of tea flavour, however, there is no mention about a quantitative description or score on these flavour terms. The Tocklai Tea Research Association, Assam, (India) has adopted standard terminology but some of them overlap. Twenty-five non-overlapping flavour terms have been identified (Bhuyan & Borah, 2001) (Fig. 1) out of about 40 generally used flavour notes. An EN may provide amore objective platform to augment the conventional methods for tea tasting and quality monitoring during production process.

1.3. Purpose of electronic nose

As stated above, an attractive and alternative strategy for monitoring the quality of tea samples manufactured under different processing conditions potentially can be achieved by sensing the organic aromatic volatiles emitted by tea samples, using EN systems (Benady, Simon, & Miles, 1995; Simon, Hetzroni, Bordelon, Miles, & Charles, 1996). EN systems appear to be very promising for a number of reasons. The main ones are that EN systems are based on inexpensive, non-specific solid-state sensors, which are sensitive to the gases that are emitted by tea samples. Furthermore, once an EN has been 'trained', it does not require a skilled operator and can potentially obtain the results in the order of few tens of seconds. In the EN system, a pattern recognition engine enables the system to perform

Table 1
Ratios of main volatile compounds to total volatile compound in black tea

Compound	Sri Lanka		India					Japan
	Rt[b]	Uva	Var. Assamica	Hybrid of Assamica* Sinesis				Beniho mare
			Assam Dimbula	Darjeeling				
				(1)	(2)	(1)	(2)	
t-2-Hexenal	0.40	3.10	2.60	4.90	3.10	0.70	0.30	1.50
cis-3-Hexenal	0.53	2.80	4.30	0.20	3.80	1.40	0.10	6.10
t-2-Hexenyl formate	0.65	9.50	11.80	11.9	5.00	5.70	3.10	5.20
Linalool oxide (furanoid-*cis*)	0.77	3.40	3.20	3.50	3.60	8.20	4.70	3.80
Linalool oxide (furanoid-*trans*)	0.83	10.30	8.80	8.00	12.0	16.7	12.0	12.0
Linalool	1.00	24.00	15.50	18.3	32.8	15.6	13.7	9.30
Phenylacetaldehyde	1.20	0.20	0.50	4.00	5.00	1.10	1.80	1.00
Linalool oxide	1.40	0.30	0.40	0.40	trace	trace	1.00	6.00
Pyranoid-*cis*	1.50	18.60	18.80	9.00	13.2	9.80	5.30	4.90
Methysalcylate	1.67	1.3	2.20	3.30	1.60	7.30	15.9	21.7
Geraniol	1.71	1.00	1.90	4.30	1.00	1.70	2.00	2.60
Benzylalcohol 2-phenylethanol	1.78	0.20	0.90	4.30	1.00	2.00	6.70	7.50
cis-Jasmone + β-ionone	1.83	0.20	0.10	7.4	1.50	0.50	4.40	0.30

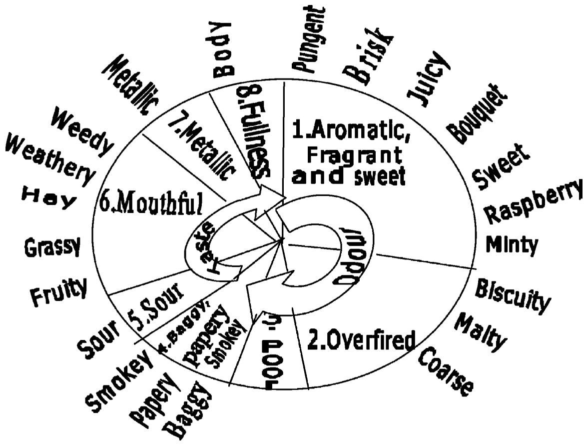

Fig. 1. Flavour wheel used to illustrate the international flavour terminology for tea. There are about 40 flavour terms out of which only 24 non-overlapping terms have been used.

complex aroma analysis of the sensor signals. Artificial neural networks (ANNs) have been extensively used to perform this pattern recognition, and good results have been reported previously in the classification of foodstuffs, such as eggs (Dutta et al., 2003), beverages (Gardner, Hines, & Tang, 1992), coffees (Gardner, Shurmer, & Tan, 1992), fish and meat (Schweizer-Berberich, Vaihinger, & Göpel, 1994; Winquist, Hornsten, Sundgren, & Lündstrom, 1993). The back propagation trained Multilayer Perceptron (MLP) paradigm is the most popular pattern recognition method in aroma analysis today. Other promising techniques include Learning Vector Quantization (LVQ), Probabilistic Neural Network (PNN) and Radial Basis Function (RBF). In this paper, we report on the use of an EN, employing an array of four tin oxide sensors, in combination with different pattern recognition engines (MLP, LVQ, PNN and RBF) to predict the quality of tea. This paper includes sections concerned with experimental procedure, data analysis, comparative evaluation of results and conclusions.

2. Experimental description

2.1. Materials

We had received five tea samples manufactured under different processing conditions from Assam, India. These five different tea samples with different qualities are as follows, (1) Drier month tea sample; (2) Drier month again over-fired tea sample; (3) Well fermented normal fired in oven tea sample; (4) Well fermented over-fired in oven tea sample; (5) Under fermented normal fired in oven tea sample. Each sample, without any additional manipulation, was placed over the period of the experiments. Monitoring is the important issue here because we wished to keep the experiment as simple as possible. We recorded temperature and humidity so that we could attempt to 'correct' for their effects if necessary.

2.2. Test procedure and data acquisition

The sensor system comprises four tin oxide odour sensors from the same manufacturer (Table 2) housed in a sensor chamber (Dutta et al., 2003). The sensors were chosen on the basis of sensitivity of the sensors to different gases; the selected sensors are designed to respond to gases such as the cooking vapours, ammonia, hydrogen sulphide, alcohol, toluene, xylene, etc. as specified by the manufacturer. The electrical conductance of the sensors varies in the presence of reducing/oxidizing gases. A thin plastic tube was connected from the input to the sensor chamber to one of the two holes in the cover of both plastic vessels (tea vessel and reference vessel). A diaphragm pump (Vacuum Pump Manufacturing Co. Ltd, UK) was used to facilitate sampling of the headspace of the vessels (Fig. 2) (Dutta et al., 2003). The headspace of the vessel containing the tea samples (5 tea samples with 5 different qualities were used for experiment) and the reference vessel were sampled in sequence as follows:

- *Tea vessel*. A sample measurement typically took 5 min to complete. The flow rate was 2 l/min. The air removed from the vessel by the pump was replaced by air from the room (Fig. 2).
- *Reference vessel*. Here, the tube from the input to the sensor chamber was connected to a plastic vessel which was full of pure water. Air from the room was pumped into the sensor chamber through this vessel. In this way the sensors were allowed to return to their baseline level over a period of some 20 min after sampling the headspace of the tea vessel. This was to make sure that the EN system was responding to the tea aromas rather than to any residual smell of the plastic vessel or only to the different environment in the reference plastic vessels (5 l). Fig. 2 shows the experimental set-up. The vessels had two small holes in their covers, to allow the headspace to be analyzed with the EN equipment. The ambient conditions (temperature and humidity) of the room in which the tea samples were kept were

Table 2
Summary details of commercially available metal oxide sensors used in our EN system

Sensor	Manufacturer	Sensitivity to
TGS 880	Figaro Engineering Inc.	Cooking vapours
TGS 826	Figaro Engineering Inc.	Toxic gases (ammonia (NH_3))
TGS 825	Figaro Engineering Inc.	Toxic gases (hydrogen sulphide (H_2S))
TGS 822	Figaro Engineering Inc.	Organic solvents (alcohol, toluene, xylene, etc.)

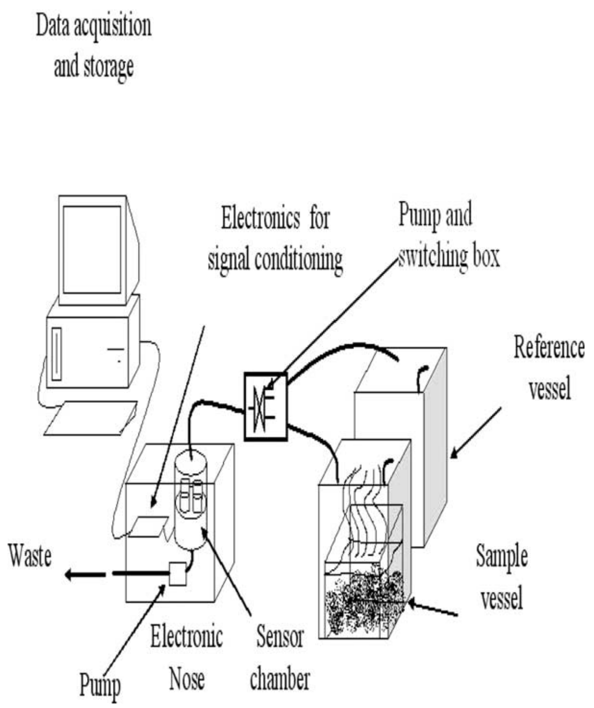

Fig. 2. Set-up for the Warwick MOS based electronic nose system for tea experiment.

monitored for the duration of the experiments. The temperature and humidity variations in the laboratory were typically 25 ± 1 °C and 30 ± 1%, over the period of the experiments.

One measurement comprises taking, alternatively, a headspace sample from the tea vessel followed by the reference vessel. During the process of the measurements, a sample of each sensor's resistance was taken every 5 s and stored in a data file for subsequent processing (Fig. 2). This process was repeated for each of the 5 tea samples in turn.

2.3. Interface electronics

The data acquisition and storage system was controlled usingLabVIEW© software (National Instruments Inc.) (National Instruments Corporation, 1998). A PC-LPM-16 PnP Data acquisition card was used for online data gathering (National Instruments Corporation, 1998).

2.4. Experimental data

In summary five different data-sets were gathered chronologically as follows:

- Data-set 1 (for 'under fermented normal fired in oven' sample)
- Data-set 2 (for 'well fermented over-fired in oven' sample)
- Data-set 3 (for 'well fermented normal fired in oven' sample)
- Data-set 4 (for 'drier month again over-fired' sample)
- Data-set 5 (for 'drier month' sample)

For each class of tea, 150 data vectors were gathered over a period of 10 consecutive days from 150 data gathering cycles. The number of days and samples were limited by practical circumstances though data collection continued 24 h per day, seven days per week, using our online EN data logger system.

3. Data processing

3.1. Signal pre-processing

The choice of the data pre-processing algorithm has been shown elsewhere to affect the performance of the pattern recognition stage (Dutta et al., 2003). In this case a difference model (i.e. static change in sensor resistance) was used: $dR = R_{air} - R_{odour}$. The complete tea data-set was then normalized, by dividing each dR by the maximum value, to set their range to [0, 1].

3.2. Data clustering

The use of Principal Components Analysis (PCA), Self-Organizing Map (SOM) and Fuzzy C Means (FCM) cluster analysis (Dutta et al., 2003; www.mathwork.com) to explore clustering within the data-sets is now discussed. Different 'cluster classification' methods were applied to verify that the categories established by each method were not arbitrary.

3.3. PCA analysis

PCA is a linear method that has been shown to be effective for discriminating the response of an EN to simple and complex odours (Dutta et al., 2003; Gardner, 1991; www.mathwork.com). The results of PCA, using the normalized data as described in the previous sub-section, are shown in Fig. 3. Three principal components were kept, which accounted for 99.8556% of the variance in the data-set (PC #1, PC #2 and PC#3 accounted for 97.9693, 1.0708 and 0.8155% of the variance, respectively, where PC #4 accounted for 0.14444%). Five qualities appear to be evident. It is also clearly evident that the sensors are linearly correlated. Since a reasonable correlation exists between categories, it can be assumed that the categories established by PCA are consistent with the five different qualities of the five tea samples (Fig. 3). The multivariate data analysis suggests there is considerable spread in the data. This spread may be due to a drifting in the sensors' responses.

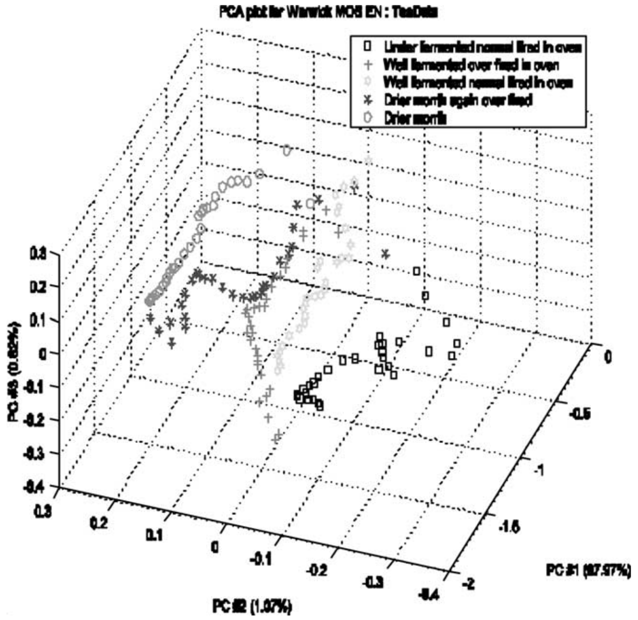

Fig. 3. PCA plot for the tea sample data.

3.4. Combined SOM, FCM and 3D-Scatter plot analysis: a new approach

An innovative data clustering approach was investigated for these tea data by combining the 3D-Scatter plot, FCM and SOM network (Gardner & Persaud, 2000; Jang, Sun, & Mizutani, 1997; Kohonen, 1989). It is depicted in Fig. 4. In multisensor space, normalized data-sets were represented using 3D-Scatter plots. From the FCM approach, a cluster center is found for each group by minimizing a dissimilarity function (Gardner, 1991; Kohonen, 1989). These cluster centers were plotted in multisensor space. So combining the 3D-Scatters plot and FCM, cluster centers were properly located in multisensor space and also within the data (Dutta et al., 2003). Thereafter a [5x1] SOM network was trained with the whole data-sets. After 500 epochs it was clear that the five nodes were approaching to the five cluster centers (estimated using FCM), which is more clearly evident from Fig. 4. So, using these three data clustering algorithms simultaneously, better 'classification' of data into different clusters was achieved (Fig. 4).

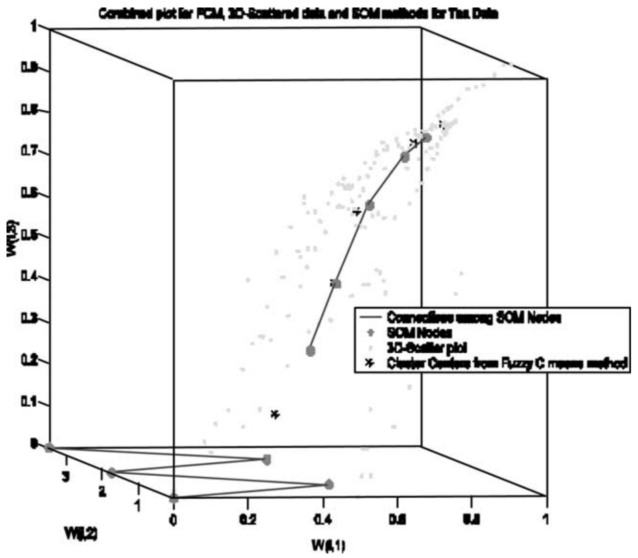

Fig. 4. Combined 3D-Scatter, FCM and SOM plot for tea sample data.

4. Comparative evaluation of neural network classification performance

4.1. Neural networks

The data-sets were analyzed using four supervised ANN classifiers, namely the MLP, LVQ, PNN and RBF paradigms (Dutta et al., 2003; Mao, 2002; www.mathwork.com). Training of the neural networks was performed with 50% of the whole data-set. The remaining 50% of the whole data was used for testing the neural networks. The aim of this comparative study was to identify the most appropriate ANN paradigm, which can be trained with best accuracy, to predict the 'Quality of tea samples'. Table 3, summarizes the architectures of the neural networks, which we used for our experimental training and testing for tea quality determination.

4.2. Dynamic drift correction using additional network training

Drift is a dynamic process, caused by physical changes in the sensors and the chemical background, which gives an unstable signal over the time (Distante, Artursson, Siciliano, Holmberg, & Lundström, 2000). EN data are usually affected by several disturbances in part due to the non-idealities of sensors and in part due to the effects of the environment as far as the composition of the gaseous sample is concerned. In some practical applications, the sample cannot be completely insulated from the surrounding environment and its drift (due to temperature and relative humidity changes) may be so large to completely obscure the intrinsic resolution of the sensor array. The problem could be solved at data analysis level making some assumptions on the nature of the disturbances (Natale, Martinelli, & D'Amico, 2002). Most gas sensors do not give stable responses over a long period of time. So sensor drift is addressed as one of the most serious impairments affecting chemical and biochemical sensors. One possible solution to this problem is to view sensor array as time-varying dynamic systems, whose variation have to be tracked by adaptive estimation algorithm (Holmberg, Davide, Natale, D'Amico, Winquist, & Lundström, 1997).

In this paper we have considered the two major drift effects from environmental temperature and humidity

Table 3
Architecture of different neural networks and results obtained in terms of ‘percentage correct classification’

Neural networks	Architecture	Classification (%)
Learning Vector Quantization	3 hidden neurons were used for this network. Output class percentage was [0.2 0.2 0.2 0.2 0.2] and learning rate was 0.0125. (www.mathwork.com)	89
Multilayer Perceptron	For this network transfer function was HARDLIM and learning function was LEARNP (www.mathwork.com)	88
Probabilistic Neural Network	It was very similar to RBF network with a competitive output layer and SPREAD constant was set as 1.0 (www.mathwork.com)	94
Radial Basis Function network	Radial basis networks consist of two layers: a hidden radial basis layer and an output linear layer. For this network SPREAD constant was set as 1.0 (www.mathwork.com)	100

variations. Variations of temperature and humidity were monitored and stored throughout the experiment periods. To consider these temperature and humidity drifts effects on the original gathered data sensors’ responses, we added two extra input nodes to our neural network. As these environmental parameters were stored along with the four sensors’ responses it was appropriate to input them together into the neural network. ANNs trained with temperature and humidity were thus able to adjust their weights using information about environmental effects along with sensors’ responses. With these additional network inputs, tea samples were better classified and we achieved up to 100% accuracy (see later sections).

4.3. MLP net

A MLP network (with learning rate equal to 0.3 and a momentum term equal to 0.4) with 6 inputs, 5 hidden and 5 output neurons was able to reach a success rate 88% in classification.

4.4. LVQ net

The networks had 6 input and 5 output neurons and a variable number of nodes in the competitive layer. Two main stages were followed:

- Initially the network was trained with a learning rate equal to 0.01 and the conscience factor was set equal to 1.
- In this next stage, once a ‘relatively good’ solution has been found by modifying the boundaries between zones where misclassifications occur, the solution is further refined. The learning rate was set to 0.0129. The LVQ algorithm was able to correctly classify 89% of the response vectors.

4.5. RBF and PNN

Neurons are added to the network until the sum-squared error falls beneath an error goal (0.000001) or a maximum number (150) of internal neurons was reached. It is important that the spread parameter is large enough so that the radial basis neurons respond to overlapping regions of the input space, but not so large that all the neurons respond in essentially the same manner (Mao, 2002; www.mathwork.com). For both the networks the spread parameter was set to value of 1.0. The PNN was able to classify correctly 94% of the response vectors whereas the RBF network’s level of correct classification was up to 100%.

5. Conclusion

In this paper an effort has been made to discriminate between the flavours of different tea samples using an EN and hence explore the possibility of replacing existing analytical and profiling panel methods. Odour patterns from five different sets of tea samples were gathered with an EN instrument. Five different tea categories were identified with the help of PCA, FCM and SOM of the sensor responses (Dutta et al., 2003; Mao, 2002). From this result it is evident that our metal oxide sensor based Warwick EN was capable of discriminating between the flavours of teas manufactured under different processing conditions, viz. over-fermented, over-fired, under fermented, etc. along with linear data processing techniques like PCA. Then MLP, LVQ, RBF and PNN neural networks were applied to the classification of the state of the tea samples. An accuracy of 100% was reached in the classification using RBF network compared with 94% using a PNN (Gardner & Persaud, 2000; www.mathwork.com). It was found that these performances compared favourably with those achieved with trained MLP (88%) and LVQ (89%). Finally, the training time of RBF and PNN were found to be faster than MLP and LVQ. In this paper we have considered the two major drift effects from environmental temperature and humidity variations. Variations of temperature and humidity were monitored and stored throughout the experiment periods. To consider these temperature and humidity drifts effects on the original gathered data sensors’ responses, we added two extra input nodes to our neural network. As these environmental parameters were stored along with the four sensors' responses it was appropriate to input them together into the neural network. The ANN trained with temperature and humidity was able to adjust its weights and hence compensate for changes in the ambient conditions. For example, tea samples were better classified and achieved up to an accuracy of 100% with a RBF network (Dutta et al., 2003).

In conclusion, we believe that a RBF networked based Warwick Metal Oxide EN provides an attractive means of discriminating among the flavours of teas manufactured under different processing conditions, viz. namely drier month, drier month again over-fired, well fermented normal fired in oven, well fermented over-fired in oven and under fermented normal fired in oven.

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