AROMA DETECTION AND CONTROL IN PASSIVE AND DYNAMIC FOOD SYSTEMS FOR SUPERIOR PRODUCT

By

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ABSTRACT

Zhenfeng Li Ph.D (Bioresource Engineering)

AROMA DETECTION AND CONTROL IN PASSIVE AND DYNAMIC FOOD SYSTEMS FOR SUPERIOR PRODUCT

Passive (static) and dynamic studies have shown aroma to be an important aspect of food quality, which can be used to differentiate, classify, and grade foodstuffs, and in some cases it can be used to predict other quality characteristics. Monitoring and control of food aroma changes during food processing can significantly improve the quality of the final product in terms of flavour, color, taste, and overall appearance. Hence, it is a prominent and urgent field of study in the post production systems.

Passive aroma detection of unprocessed foods and dynamic aroma detection during food processing was undertaken using a fast GC analyzer – zNose. During the study on the passive aroma detection, the aroma of Chinese spirits (Fenjiu) and mango (*Mangifera indica* L.) fruits, (i.e., liquid and solid states, respectively) was analyzed. In the study of Chinese spirits, aroma profiles of Fenjiu liquor samples of different quality levels were acquired and used for quality classification and prediction. Measurements of dielectric properties of the samples were also conducted to estimate alcohol concentration. In the study of mango fruits, aroma changes of mango samples were monitored during their shelf life and used to evaluate mango quality. Ripening and rots were detected with 80% and 93% accuracy, respectively.

During the study of dynamic aroma detection, a real-time aroma monitoring and control system was developed for use during microwave drying. Aroma signals of a processed food item were detected with zNose and analyzed with a fuzzy logic algorithm to determine the optimal food drying temperature. Phase control was used to adjust the microwave power level to meet temperature

requirements. Carrot (*Daucus carota* L.) and apple (*Malus domestica* Borkh) were selected as representatives of vegetables and fruits. In carrot drying, samples could be dried in a short time at high temperatures but the interior of some sample cubes was burnt. Drying at a lower temperature extended the drying process, but led to a great loss of aroma in the finished product. The best results were obtained at 60°C. Based on these results, a fuzzy logic controller was designed and employed to control the drying process according to carrot aroma changes. To investigate the possibility of aroma improvement without zNose assistance, a linear control method was developed whereby a temperature control profile imitated the fuzzy logic control, but aroma control was not included. With these new control strategies, the carrot color and flavour were significantly improved and less time and power were consumed. Similar results were achieved when apple was microwave-dried. Apple aroma was monitored online during microwave drying processes and controlled with similar fuzzy and linear control strategies. Apple color, aroma, and overall appearance remained intact with the new strategies and less time and power were consumed. In contrast to the carrot drying, a different linear temperature profile was required for apple drying in terms of aroma retention.

RÉSUMÉ

Zhenfeng Li Ph.D (Géne des bioressources)

DETECTION STATIQUE ET DYNAMIQUE D'ARÔMES ALIMENTAIRES

Des études statiques et dynamiques d'arômes alimentaires ont démontré que l'arôme constitue un important aspect de la qualité des aliments, qui peut servir à différencier les aliments et de les classer selon leur niveau de qualité, et, en certains cas, à prévoir certains autres aspects de leur qualité. Une régie des arômes durant la transformation des aliments peut améliorer la qualité du produit final par rapport à sa saveur, sa couleur, son goût et l'ensemble de son apparence. La surveillance et la régie des arômes alimentaires est donc un important et urgent domaine d'étude pour l'industrie de la transformation.

La détection statique d'arômes d'aliments non-transformés et la détection dynamique d'arômes durant les étapes de transformation fut entreprise grâce à un analyseur CPG rapide — zNose. Lors du volet de détection statique d'arômes, l'arôme de spiritueux Chinois (Fenjiu) et celui de fruits de mangue (*Mangifera indica* L.) (matière liquide et solide, respectivement) furent analysées. Durant l'étude des spiritueux chinois, les composants de l'arôme provenant d'échantillons de Fenjiu de différentes qualités furent détectés et servirent à leur classification et à des fins prédictives. Le contenu en alcool fut mesuré grâce aux propriétés diélectriques des échantillons. Durant l'étude sur les fruits de mangues, l'arôme des échantillons de mangue fut suivi durant leur durée de conservation à l'étalage, et servirent à évaluer leur qualité. Leur mûrissement et pourriture furent détectés avec une précision de 80% et 93%, respectivement, en utilisant des signaux d'arômes spécifiques.

Pour le volet de détection dynamique de l'étude, un suivi des composantes aromatiques et un système en temps réel de régie de la transformation à base de signaux CPG d'arômes furent développés pour un système de séchage par micro-

ondes. Les signaux des composantes des arômes furent détectés par zNose et analysés avec un algorithme de logique floue afin de déterminer la température de séchage idéale. Un réglage de phase servit à ajuster le niveau de puissance des micro-ondes de façon à répondre aux demandes de température. Les carottes (*Daucus carota* L.) et pommes (*Malus domestica* Borkh) furent choisis commes exemples représentatifs de légumes et de fruits. Lors du séchage de carottes, les échantillons pouvaient être séchés rapidement à une température élevée, mais l'intérieur de certains cubes-échantillons brula. Un séchage à une puissance moins élevée, allongea la période de séchage, mais eut pour résultat une importante perte d'arôme du produit final. Les meilleurs résultats furent obtenus à 60°C. Fondé sur ces résultats, un contrôleur à logique floue fut conçu et employé pour contrôler le processus de séchage entier selon le niveau de composantes de l'arôme de carotte. Afin d'étudier la possibilité d'amélioration l'arôme sans l'assistance du système zNose, une méthode de régie linéaire fut développée de par laquelle un profil linéaire de température imitant le système de contrôle floue opéra, sans qu'une détection des arômes soit nécessaire. Avec ces nouvelles stratégies de régie, la couleur et la saveur des carottes furent clairement améliorées, et la transformation nécessita moins de temps et consomma moins d'énergie. Des résultats semblables furent obtenus lorsque des pommes servirent comme matériel expérimental. L'arôme de pomme fut suivi en direct lors du séchage par micro-ondes, qui fut régi par des stratégies de contrôle floue ou linéaire semblables à celles pour les carottes. La couleur, l'arôme et l'apparence générale des pommes sont demeurées intactes avec les nouvelles stratégies de régie, et moins de temps et d'énergie furent dépensées. Un profil de températures linéaire différent de celui des carottes fut nécessaire pour retenir l'arôme des pommes durant le séchage.

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NOMENCLATURE

ε'	Dielectric constant
ε''	Dielectric loss factor
τ_I	Integral time constant
τ_D	Differential time constant
AC	Alternative Current
ANN	Artificial Neural Network
ART	Adaptive Resonance Theory
AT	Actual Temperature
CA	Cluster Analysis
CDA	Canonical Discriminant Analysis
CP	Conductive Polymer
Cts	Counts
DAQ	Data Acquisition
DC	Direct Current
DFA	Discriminant Factor Analysis
F	Control voltage corresponding to final drying power
FAO	Food and Agriculture Organization of the United Nations
FIS	Fuzzy Inference System
FLC	Fuzzy Logic Control
GA	Genetic Algorithms
GC	Gas Chromatography
GC-FTIR	Gas Chromatography-Fourier Transform Infrared spectroscopy
GC-MS	Gas Chromatography-Mass Spectrometry
$g(s)$	Transfer function
$G(s)$	Feed forward transfer function
HPLC	High Pressure Liquid Chromatography
$H(s)$	Feedback transition function
I_m	Maturity index
I/O	Input/Output

K	Proportional gain of the PID controller
MC	Moisture Content
MLR	Multiple Linear Regression
MOS	Metal Oxide Semiconductor
MV	Minimum control voltage corresponding to maximum power
NFS	Neuron-Fuzzy Systems
NIRS	Near Infrared Spectroscopy
NMR	Nuclear Magnetic Resonance
PCA	Principle Component Analysis
PCR	Principal Component Regression
PID	Proportional Integral Differentiate
PLS	Partial Least Square
PT	Preset Temperature
PWM	Pulse Width Modulation
RBF	Radial Basis Function
RR	Respiration Rate
RT	Room Temperature
RT	Retention Time
SAW	Surface Acoustic Wave
SCR	Silicon Controlled Rectifier
SH-SAW	Shear Horizontal-Surface Acoustic Wave sensor
SOM	Self-Organizing Map
TRIAC	Triode for alternative current
TSS	Total Soluble Solid
$\bar{u}$	A vector of inputs
V	Control voltage
$\dot{\bar{x}}$	A vector representing the "change in state"
$\bar{x}$	A vector representing the system "state"
$\bar{y}$	A vector of outputs

CHAPTER I

GENERAL INTRODUCTION

Food aroma measurement and control, microwave drying, and control strategies are integrated in this study. The background information is provided in this chapter.

1.1 Food flavour and food flavour evaluation

Food flavour (odour and taste) is one of the most important intrinsic attributes of food quality. This attribute not only determines a consumer's acceptability and preference, but also reflects the food quality itself. A "pleasurable" flavour may emanate from a good quality food; a "stinky" flavour may diffuse from a rotten one. Food flavour and food quality are inseparably linked.

The human nose and tongue are commonly used as "instruments" to evaluate the quality of food products. In the food industry, panels of trained inspectors are employed for food quality monitoring, evaluation, and control. However, training a panellist is a very time-consuming and expensive process, and such a panel can normally work for a very short period of time every day. Moreover, the response of the human nose and tongue are subjective, scarcely repeatable, vary between individuals, and affected by physical and mental conditions.

Gas chromatography-mass spectrometry (GC-MS), gas chromatography Fourier transform infrared spectroscopy (GC-FTIR), high pressure liquid chromatography (HPLC), near infrared spectroscopy (NIRS) and nuclear magnetic resonance (NMR), etc., are analytical and quantitative techniques which have been used for flavour analysis. Although they are capable of giving precise and reliable results, the use of such instruments is usually tedious, time-consuming, and expensive, and more importantly, their results correlate poorly with human perception. Therefore, new techniques allowing faster objective, on-line flavour characterization without the need for complex equipment or skillful operators are an urgent demand of the food industry. This need promoted the emergence of the electronic nose and tongue.

The first attempt for an instrumental nose started in 1961 when a mechanical nose was developed by Moncrieff. Three years after, an electrical nose was reported by Wilkens and Hatman in 1964 (Gardner and Barlett, 1994). At this early stage, the main effort was focused on the development of selective sensors sensitive to specific chemical components. But this attempt was soon abandoned when, in the 1990s, more than 6000 compounds were found to contribute to food flavours, instead of the mere 500 first believed to be involved (Acree and Teranishi, 1993). It is obviously impossible to have a sufficiently vast array of sensors to detect each single chemical compound, when the flavour of one food may include hundreds, if not thousands of flavour-related compounds.

In the early 1980s, a new concept in sensor applications emerged, which employed an array of non-selective sensors, enhanced through the use of special mathematical data processing (artificial neural networks, principal components analysis, fuzzy logic, etc.) for pattern recognition. In 1982, Persaud and Dodd first built a formalized prototype of an electronic nose system, which Gardner and Bartlett (1994) later defined as:

> ***"An electronic nose is an instrument, which is comprised of an array of electronic chemical sensors with partial specificity and an appropriate pattern-recognition system, capable of recognizing simple or complex odours."***

This definition restricts the electronic nose to an array of sensors which are specifically used to sense odorant molecules in an analogue to the human nose. A considerable number of sensors based on this principle have been developed over the past two decades. However, new technologies which emerged thereafter, using new flavour sensing systems based on different operating principles had to choose different names, such as artificial sensor array, artificial nose, gas sensor array, etc. Given their small size, low cost, high sensitivity and fast response for chemical sensing applications, surface acoustic wave (SAW) sensors have received a great deal of attention in recent years. There are basically three applications of SAW sensors: a SAW sensor array coated with different polymers which have partial selectivity to various gaseous components; a shear horizontal surface acoustic wave sensor (SH-SAW) which is designed for liquid taste analysis; and an uncoated piezoelectric quartz crystal sensor with only one detector, termed as zNose™ (7100 Fast GC Analyzer, Electronic Sensor Technology, New Bury Park, CA, USA), which is commercially available since 2003. The zNose incorporates a fast chromatography column and a SAW crystal sensor to separate and identify chemical odour components, respectively. This device is capable of detecting chemicals at picogram sensitivity within one minute.

Research into an electronic nose was immediately followed by intensive study of electronic tongue, which mimic human's tongue to taste sourness, saltiness, bitterness, sweetness, and umani in aqueous surroundings. The term "electronic tongue" is believed to have first been introduced at the 10th European Conference on Solid State Transducers (Leuven, Belgium, Sep. 8-11, 1996) in 1996, whereas the first concept of taste sensors was published in 1990 (Sehra *et al.*, 2004). Two commonly used electronic tongue designs are potentiometric and voltammetric, with the former already having been made commercially available (Toko, 1998). Besides these two techniques, SH-SAW sensors, mentioned above, have also shown some promise in the development of the electronic tongue.

Over the last twenty years, numerous applications of the electronic nose and tongue to food flavour detection and food quality evaluation have been reported. These novel techniques have the advantage of being rapid and non-destructive, and their results correlate directly with the way humans perceive food products. Amongst a number of applications, the zNose instrument has already been used to detect off-flavours in wine (Staples, 2000), evaluate the aroma of black tea (Staples, 2002) and classify honey (Lammertyn *et al.*, 2004).

Applications of the electronic tongue can be found in beverage analysis (Lvova *et al.*, 2002), wine classification (Riul *et al.*, 2004), vodka quality assessment (Legin *et al.*, 2005), and the qualitative analysis of natural water (Martinez-Manez *et al.*, 2005).

Just as the combined function of the human nose and tongue are inseparable, the electronic nose and tongue have been combined as fusion sensors by many researchers, and this fusion sensor technique has already seen some applications in food flavour detection. Li *et al.* (2000) classified wines with a combination of taste and gas sensor arrays, with their outputs subjected to fuzzy logic data processing. Bleibauma *et al.* (2002) used a similar combined technique to evaluate the quality of apple juice, while Winquist *et al.* (1999) tested several other juices with a combination of a gas sensor array and voltammetric electronic tongue.

Despite these encouraging findings, the electronic nose and tongue are still mostly limited to the laboratory scale and are seldom found in practical industry applications. This is in part attributable to sensor technology not being fully developed and in part to the fact that insufficient research has been applied to various food products' aroma and flavour components. In order to optimize quality evaluation methods and to enhance the marketability of food products with respect to their flavour features, there is a considerable need to apply these new instruments to different food products and find their possible industrial realization. Each different foodstuff may need special attention devoted to its particular aspects, and correlations need to be built between flavour and quality. Further, different food varieties need to be checked with different instruments to find which are best applicable to a specific situation. With stricter regulatory limits on food flavours in some countries, these needs are becoming more pressing both for manufactures and consumers.

In contrast to visual and auditory sensations, which can be recorded, transmitted and retrieved by machine, we still cannot adequately express, define, or explain our smell and taste sensations beyond the range of our nose and tongue.

With the help of these technologies, new advancements may be achieved in the near future.

It is believed that the 21st century can be the flavour age. The culture of flavour developed with human civilization and the good flavour of foodstuffs will give great pleasure to the senses of human beings. In this process, the new developments of the electronic nose and tongue will undoubtedly play a leading role.

1.2 Microwave drying

FAO reported that 2.0 billion tons of cereals were produced in 2006 (FAO, 2006) to support 6.6 billion people, and this production is predicted to reach 2.4 billion tons in 2015 and 2.8 billion tons in 2030 (FAO, *World agriculture: towards 2050,* 2002). Although a large quantity of food is available in one place or another on earth, a good portion of the food produced never benefits mankind and many people still remain hungry. While estimated 3-11% of the world's annual food production deteriorates due to improper storage, drying, or other preservation issues, the mean (2000-2006) annual world food shortage (supply minus demand) only represents 2% of world food production (FAO, 2000-2006). Consequently, the need for new food preservation methods and the improvement of existing methods is urgent.

Compared to canning, freezing, and aseptic processing, drying, which has been practiced since time immemorial, is an efficient, cost-effective way of food preservation. Considering energy use efficiency, environmental concerns, and increasing demands to feed a growing population, along with the goals of enhancing product quality and reducing spoilage, much research has been carried out to develop new techniques for food drying. Agricultural, industrial, and domestic applications of drying technologies over the preceding century have expanded our understanding and led to the development of many new drying techniques. Four main types of drying — hot air convective drying, vacuum drying, freeze drying, and microwave drying — have demonstrated their respective superiorities in regard to specific products.

Over the past two decades, there has been an increasing interest in microwave drying, which can overcome certain limitations of conventional thermal food treatment methods. It has advantages in various aspects of performance, such as higher drying rates, shorter drying time, lower energy consumption, and better quality of the dried products (Mullin, 1995; Orsat *et al.*, 2006; Raghavan *et al.,* 2005a; Sanga *et al.,* 2000,). For ideal drying conditions to prevail, microwave energy applied to the product must be controlled at suitable levels. In most modern microwave ovens, power is controlled in an intermittent manner. To achieve the best drying quality of products, a number of studies have been conducted to investigate the best ON: OFF cycle ratio within a predefined time interval. Changrue *et al.* (2004) recommended that, to achieve an efficient drying process, carrot cubes (1000 mm^3) should be dried under a fixed power density of 1 W/g with 70°C hot air. The power to the magnetron was manually controlled with an initial pattern of "55 sec ON: 5 sec OFF" followed by a pattern of "30 sec ON: 30 sec OFF" when the moisture content of the carrot cubes dropped below 30% (wet basis). Sunjka *et al.* (2004) tested different pulse modes and power levels and combined microwave drying with mechanical and chemical pre-treatment to improve the cranberry drying process. It was determined that high-quality, dried cranberries could be obtained under a microwave power level of 1.25 W/g with a pulse pattern of "30 sec ON: 45 sec OFF". Liang *et al.* (2003) concluded a "10 sec ON: 45 sec OFF" cycle was suitable for rose flower drying.

Due to their large variations of inherent properties, bio-products may react differently under microwave treatment. Each bio-product may need a specific intermittent power scheme. Furthermore, the best power control scheme also depends on the size, quantity, and moisture content of the material. Any changes in these factors would affect the efficiency and results of the drying process. More stable and convenient power control methods are needed to optimize the microwave drying process.

Among various modern power control methods, phase control is widely used in both industrial and domestic applications for its simplicity and high efficiency. Cheng *et al.* (2006a; 2006b) developed a resistive-capacitor based phase control circuit using a Triac for the power control of a microwave oven power and obtained different power levels by changing the value of the resistor. However, it was found to be difficult to manually obtain suitable resistive values to match different power level requirements for different objects, and more experiments are needed to find the optimal resistor values for different types of materials to optimize drying process. Moreover, the most important factor for the drying processes — the temperature of the food — was still left uncontrolled and could only be estimated by experimentation. For accurate and automatic adjustment of the power level and control of the temperature during the drying process, more intelligent control strategies are needed.

1.3 Aroma studies in food drying process

The main objective of any industrial food process consists in satisfying the consumer demand in terms of quality with minimum cost. Among many quality aspects, food aroma is a key quality which determines consumers' preference and acceptance. However, due to the sensitivity of volatile aroma compounds to either physical or chemical treatments, preservation of preferred aroma and the avoidance of unacceptable aroma is a crucial task for food engineers. This situation becomes much more serious in the process of food drying where high temperatures and moisture removal activity often lead to significant volatile compound loss.

A great number of studies have been conducted concerning various foods' changes in aroma under different drying treatments, but most were restricted to a aroma comparison of the initial and final products, whereas only a few reported aroma analysis throughout the food drying process. Kompany and Rene (1993) studied the retention of five major flavour components of cultivated mushrooms and investigated the effect of processing conditions on the kinetics of loss of these components. Boudhrioua *et al.* (2003) measured the aromatic changes of banana during convective air-drying at different temperatures (40°C, 60°C, and 80°C). Krokida and Philippopoulos (2006) analysed the volatility of some representative aroma compounds during freeze- and convective-drying of apples. These pioneering works all produced important information and gave positive incentives to the food industry.

However, all these aroma studies were dependent on offline aroma measurement and the duration of measurements were usually very long (hours). Therefore, much detailed information within such intervals, which might be very important for some food products, was lost. Moreover, offline measurement can never support the real time use of aroma parameters to facilitate online control of the drying process. Hence, online aroma detection has long been recommended (Kompany and Rene, 1995), yet none has been reported up to present. The reason is the lack of a rapid and reproducible method for aroma detection.

With the advent of zNose, a rapid detection and analysis of food aroma profiles first became possible. This new instrument has the capability of detecting food aroma as fast as in seconds and the chemical components can be immediately identified. However, the application of the instrument is still restricted to offline detection (Lammertyn *et al.*, 2004; Gan *et al.*, 2005) and no online use has yet been reported.

Despite considerable work in attempting to control microwave power and temperature during microwave drying, no aroma evaluation was hitherto reported for this process. Consequently, how product quality, in terms of aroma, varies according to the controls imposed on microwave drying is still unknown. Moreover, the problem of non-uniformity of microwave heating which may result in partial interior burning of the product is still a critical challenge for microwave researchers.

CHAPTER II

GENERAL HYPOTHESIS, OBJECTIVES AND SCOPE

2.1 Hypothesis

Under static conditions or when undergoing processing, agricultural products naturally release volatile organic compounds. These volatiles can be detected by human sensory organs or through a range of instrumented tools. The volatile components that are sensed may be related to the commodity's quality characteristics. In this study, the following hypotheses will be tested through the use of electronic nose technology for both aroma detection and in the control of a microwave drying system:

1) In passive (static) status, the aroma which emanates from an agricultural products can be detected and used for the purposes of product identification and quality evaluation;
2) During the microwave drying process, the released volatiles from agricultural products can be measured. Such aroma signals, if measured rapidly, can be used to obtain the highest possible quality of the final products, through aroma-based control of the drying process.

2.2 Objectives

The research includes both passive (static) and dynamic studies of food aroma. The main objectives of passive aroma study were to evaluate food characteristics by detecting their aroma signals. The main objectives of dynamic aroma study were to maintain food quality according to aroma retention requirements during microwave drying. Specific objectives were:

1) The application of electronic nose technology in identification of Chinese spirits in different grades;

2) The evaluation of ripeness and rot development of mango fruits during their shelf life;
3) The design of a system with a capacity for food aroma monitoring and control during microwave drying;
4) To test the designed control system with carrot samples;
5) To test the designed control system with apple samples.

2.3 Scope of study

Numerous agricultural products' volatiles can be monitored. However, in passive (static) aroma detection portion of this study, the scope was limited to Chinese spirits as a representative liquid product, and mango fruits as a representative solid product. The dynamic aroma study was limited to aroma monitoring and control in microwave drying, which is an efficient and fast drying method. This method challenges the designed system's capacity to avoid the burning of products, a frequent problem during microwave drying. Carrot and apple were used as representatives of vegetables and fruits, respectively.

CHAPTER III

LITERATURE REVIEW

To better understand each part of the integrated study, a thorough literature review is provided in this chapter. Flavour detection by human being and machine, different drying technologies, various control strategies are introduced in detail.

3.1 Food flavour

3.1.1 Human olfaction and gustation

Smell and taste are two of the five senses human beings use for perception of the environment. The study of olfaction and gustation can be dated back to the fourth century B.C. when the Greek scholar Aristotle classified odours as "sweet, pungent, harsh, sour, succulent, fetid" and listed the basic tastes as "sweet, bitter, sour, salty, astringent, pungent, harsh" (Cain, 1978). By today's standards, a meaningful study of olfaction and gustation was begun by German psycho-physicians during the scientific revolution of the nineteenth century and was replaced by electro-physiologists after the Second World War when recordings from single muscle fibres were possible (Bartoshuk, 1978). The understanding of olfaction and gustation has rapidly increased during the last 30 years, yet this knowledge is incomplete and imprecise.

The human olfactory system consists of the olfactory epithelium, olfactory bulb, and limbic system in the brain (Appendix A). Odour sensations are induced by the interaction of odorants with specialized receptor cells in the olfactory epithelium by means of G-protein binding. There are more than 100 million receptor cells (50 million per nostril) (Gardner and Barlett, 1994), but only about 100 different types of G-proteins, i.e., 100 different kinds of chemosensory receptors. As humans are capable of discriminating more than 10,000 different odours, there must be a partial overlap among these chemosensory receptors. The information generated from receptor cells is sent to the olfactory bulb where about 5,000 glomeruli nodes are followed by 100,000 mitral cells. Signals are amplified by three orders of magnitude in the olfactory bulb, drifts are removed, and 10,000 different odours are discriminated. But the detailed mechanisms operating in the olfactory bulb are still unknown (Gardner and Barlett, 1994).

The basic units to sense taste are taste buds, which are situated in special papillae or other regions of the oral cavity. Each taste bud is composed of 70 to 150 specialized epithelial cells called taste receptor cells. Taste receptor cells are connected to sensory neurons through synapses and transfer information to the brain via cranial nerves (Appendix B). These neurons are found to have broad, overlapping response patterns. An individual neuron is non-specific but the pattern of activity across multiple neurons is unique for a given taste stimulus (Schiffman and Pearce, 2004).

The sense of taste is basically divided into five discrete modalities: salty, sour, sweet, bitter, and umami. All other tastes are combination of these five basic tastes. Recent research has found that: (i) NaCl and LiCl are two typical stimuli of saltiness; (ii) the presence of the hydrogen ion, acetic acid, or citric acid is the cause of sourness; (iii) sweetness is due to sucrose and glucose, etc.; (iv) taste of bitterness comes from quinine, caffeine, and $MgCl_2$; and (v) the monosodium salt of L-glutamic acid (monosodium glutamate; MSG) contained in seaweed, disodium inosinate (IMP) in meat and fish and disodium guanylate (GMP) in mushrooms are typical stimuli for the taste of umami (Bartoshuk, 1978). These five tastes all have their own specific transduction mechanisms in the taste buds.

3.1.2 Electronic noses

The physical structure of human nose, tongue, and their interactions with the brain are base of the development of machine analogues. Similar to the human olfactory system, the core of an electronic nose system is a sensor array that detects signals which are not specific to a particular chemical constituent which is being measured, but a pattern of signals which can be related to certain features or qualities of the food sample. A trained computer programme can recognize the class of response patterns related to a particular sample or environment under study.

A typical electronic nose includes an odour handling and delivery system, a sensor array, a signal processing unit, and an odour profiling presentation unit.

3.1.2.1 Odour handling and delivery

Like the nasal cavity of the human olfactory system, the first part of an electronic nose is an odour handling and delivery system, which captures the vapours and improves the capacity and reliability of the odour sensing system by means of increasing odour density or stabilizing its flow rate. Three types of systems (sample flow, sample static, and preconcentrator) are used for different purposes in various electronic noses.

In a sample flow system, sensors are placed in the vapour flow path, which allows the rapid exchange of vapours. This system may have different approaches such as headspace sampling, diffusion, permeation, bubbler, and sampling bag. All have their own *pros* and *cons*.

In a static system, sensors are exposed to vapour at a constant concentration and measurements are made on the steady-state responses of the sensor to the odours when vapour equilibrium is reached.

The technique of preconcentration originates from gas chromatography, where a preconcentrator tube is used to enhance the sensitivity of the sensor, and an absorbent, such as Tenax TA, Tenax GR, Carbopack B, Carbotrap, Carboxen 569, Carbosieve SIII, etc. (Groves *et al.*, 1998), is used to separate the vapours according to their different desorbing capability. After the vapours accumulated in the tube have been absorbed on the absorbent, various heat pulses are applied to the tube to selectively desorb and separate the vapours.

3.1.2.2 Sensors

At the beginning, people thought an ideal gas sensor should respond to only one specific odour. A number of such specific sensors had been developed in 1970's and 1980's (Gardner and Bartlett, 1994), but a problem occurred when a complex odour, composed of several hundred chemical components, was to be measured. It is not only unnecessary, but also impossible to develop a large enough number of sensors to detect every constituent in a single odour. Today's new electronic noses use partially specific, overlapping sensors, i.e., they can broadly respond to a range of gases. Nine different general types of sensors (Table 3.1) (Gardner and Bartlett, 1994), among which the three most common are metal oxide semiconductor (MOS), organic conductive polymer (CP), and surface acoustic wave (SAW) detector, have been used in recent studies.

Table 3. 1 Main types of gas sensor arrays used in electronic noses (Gardner and Bartlett, 1994)

Array type	Sensing channels	Country of origin
Sintered metal-oxide chemo-resistors	6-12	Japan, UK
Lipid layers (on piezoelectric crystals or SAW)	6-8	Japan, USA
Phthalocyanine chemo-resistors	5	UK
Organic polymers on chemo-resistors	12-20	UK, USA
Electrochemical	2-18	USA
Pd-gate MOSFET	10	Sweden
Optical FET camera	324 pixels	Sweden

3.1.2.3 Interfacing circuit, signal sampling and digitizing

The sensor responses in a sensor array are time-dependent variables describing the aroma. This signal includes both transient responses and stable responses. Some electronic nose systems have been designed based on transient responses including rising and decaying signals, but most of them have been based on stable signals.

As the original responses are typically not electrical signals, they are usually transduced to electrical signals to facilitate signal processing by means of interfacing circuits. The interfacing circuit may vary depending upon the measurement methods of the gas sensors.

The electrical signals generated by the interfacing circuits are often low in amplitude and may contain unwanted noise. They must be processed by buffering, amplifying, filtering, and other functional circuits (e.g., linearization, integration, differentiation, logarithmic and antilogarithmic conversion, peak-to-peak and phase detection, temperature compensation, etc.) before they reach a computer. A conditioning circuit is used to amplify the signals to a required level, e.g. 0-5V, remove most noise, and accomplish some basic mathematical operations.

Following the signal conditioning system, usually there exists a data acquisition system, which may be composed of sample/hold, anti-aliasing, and analog-to-digital conversion. These tasks are usually accomplished by a microprocessor. According to the Nyquist sampling theorem (Appendix C), the sampling rate must be at least twice that of the signal frequency, otherwise signal aliasing may happen.

After digitization, the signal is preprocessed through several subsequent steps including: baseline manipulation (to offset the influence of the background flavour signal), compression (to reduce the amount of data), and normalization (to facilitate data processing), and finally the digital data is processed by a computer through multivariate pattern analysis, such as principle component analysis or artificial neural networks.

3.1.2.4 Pattern recognition

The response vectors generated by the sensor array are usually analyzed by pattern recognition method. By this method, a known odour is first measured and the result is used to train the output of the sensor array based on a set of descriptors. Then the response of an unknown odour is tested against the knowledge base and the predicted class membership is given.

Commonly used statistical methods for pattern recognition include principal components analysis (PCA), partial least squares (PLS), multiple linear regression (MLR), principal component regression (PCR), discriminant factor analysis (DFA), and cluster analysis (CA). Commonly used non-parametric methodologies are artificial neural networks (ANNs), fuzzy inference systems (FIS), self-organizing maps (SOM), radial basis functions (RBF), genetic algorithms (GAS), neuron-fuzzy systems (NFS), and adaptive resonance theory (ART).

As a linear classification method, PCA has been widely used in various studies to display the response of an electronic nose to a simple or complex aroma. Capone *et al.* (2001) used a thin film semiconductor (SnO_2) sensor array coupled with dynamic PCA to recognize the rancidity of two different kinds of milk. Lammertyn *et al.* (2004) tested a zNose unit's results with PCA to classify honey based on rapid aroma profiling. Lozano *et al.* (2005) recognized 29 typical aromas in white wine with a SnO_2 multi-sensor array electronic nose combined with a PCA technique. Gomez *et al.* (2006) monitored mandarin maturity with a specific electronic nose device (PEN2) coupled with PCA.

ANNs have frequently been used in odour recognition to obtain better results. In a typical ANN system for an electronic nose, the number of input nodes usually corresponds to the number of sensors in the array, the number of hidden neurons is determined experimentally, and the number of output units is often equal to the number of odours analyzed (Hines *et al.*, 2004). ANNs have been combined with various chemo-sensors in many food flavour analysis applications, including microbial quality classification of grains (Jonsson *et al.*, 1997), classification of aromatic species (cinnamon, red pepper, thyme, pepper, and nutmeg; Brezmes *et al.*, 1997), identification of odours of liquors (beer, spirit, samshu, wine; Yang *et al.*, 2000), recognition of methanol VOCs (Penza and Gassano, 2003), and detection of beef spoilage (Panigrahi *et al.*, 2006). The shortcoming of ANNs is their low speed and long time in searching for the optimal architecture and parameters, as there are many algorithms and experiments are required to choose the optimal one.

3.1.3 Electronic tongues

As the wet counterpart of the electronic nose, an electronic tongue functions according to similar principles as the electronic nose, combining signals from non-specific and overlapping sensors with pattern recognition methods. For an electronic tongue, the sample handling and delivery system is a trivial issue, and interfacing and conditioning circuits are similar to those of electronic noses.

3.1.3.1 Sensors

Among the sensors commonly used for electronic tongues (Table 3.2), potentiometric and voltammetric devices were the first developed and the most studied, while surface acoustic wave (SAW) and optical chemical sensors have been developed more recently. The potentiometric sensors are sensitive and selective, but they are also sensitive to electronic noise, putting high demands on the electronics and measurement set-up. The voltammetry technique has advantages of high sensitivity, versatility, and robustness, but it suffers from low selectivity and difficult data interpretation, which lead to development of special mathematical models to compress the complex data they produce (Holmin *et al.*, 2001; Artursson *et al.*, 2002).

Table 3. 2 Main types of sensors used in electronic tongues (Holmin *et al.*, 2001)

Sensor type	Sensing channels	Country of development
Potentiometric	5 - 45	Russian, Italy, Japan, Spain, Brazil
Voltammetric	3 - 6	Sweden
SAW	1	UK
Optical	100 pixels	USA

3.1.3.2 Pattern recognition

Similar pattern recognition techniques as those employed by electronic noses are also found in electronic tongue applications, amongst which PCA, PLS, and ANNs are the most commonly used.

An interesting improvement in this field is the combination of FIS with ANNs. As all the five basic taste modalities (saltiness, sourness, sweetness, bitterness, umami) are usually described by humans as a matter of degree, it is rational to adopt fuzzy inference in taste perception with electronic tongues. If FIS is further combined with ANNs in different stages such as input processing, weight updating, and output defuzzification, a powerful and fast technique can be achieved. This technique may possibly support a promising connection between human perception and machine measurements. Li *et al.* (2000) tried this approach and realized an accurate, convenient, and efficient method for wine analysis using a sensor fusing technique.

3.1.4 Impedance analysis of food products

The electronic nose is mainly used for volatile organic compounds and the electronic tongue is limited to liquid food samples. Following these developments, it is not strange if people ask a further question like: "is there any technique for non-volatile compound evaluation, especially for those with little smell but strong taste?"

From the 1960s onward, studies have been conducted to analyze the dielectric properties of food products in the radio frequency and microwave ranges, with the main goal of searching for their optimal heating and cooking profiles. A few were extended to moisture content determination, pest control, maturity assessment, and quality measurements (Nelson, 2004).

Although these studies have targeted microwave heating and cooking, some attractive side-effects can be found from the point of view of the flavour researcher: their dielectric properties may be associated with their taste. Sakai *et al.* (2005) developed a food model system in 2005 to study microwave heating, and their results showed that the dielectric properties of the model could be adjusted by adding sucrose and sodium chloride, and a procedure for determining their concentrations was proposed. Tanaka *et al.* (2005) studying soy sauce found sodium chloride influenced its dielectric constant ε' and loss factor ε''. In an experiment conducted by Nunes *et al.* (2006), milk spoilage was found by measuring its dielectric properties. From these experiments, the possible relationships of dielectric properties with their individual taste factors can be hypothesized, because sucrose is the main cause of sweetness, sodium chloride is the stimulus of saltiness, and spoilage comes with development of organic acids which lead to sourness. Thus, by measuring the dielectric properties of food products, it should be possible to determine their taste profiles.

New techniques further support this hypothesis: instruments that can measure total impedance (resistance, capacitance, and inductance) from which capacitance is determined by measuring the dielectric properties have recently become available. With more parameters, the characteristics of food products with regard to their taste can be achieved in a novel way. More importantly, this technique can be adapted both to liquid and solid state food samples, which is surprising news for flavour researchers.

3.2 Drying technologies

By far most foods consumed by mankind are of biological origin, derived either from plant or animal materials. While such foods nourish human beings, they can also serve as suitable substrates for a number of micro-organisms involved in the deterioration of foods. The war between human beings and micro-organisms is always ongoing. Long before knowing of the existence of micro-organisms, people employed methods such as salting, smoking, heating, freezing, or canning, to prevent or inhibit spoilage. Among these methods, an effective and broadly applied method is to reduce the food's water content through drying or dehydration, thus limiting the activity of micro-organisms.

Thermal drying, a very common and diversified process, converts solid, semi-solid, or liquid foodstuffs into a solid product through the heat-driven process of evaporating the liquid into a vapour phase. Mujumdar and Suvachittanont (2000) stated that over 500 types of dryers had been reported in the literature, and that over 100 distinct types were commonly available. Currently, the most popular drying methods are solar drying, hot air convective drying, freeze-drying, vacuum drying, and microwave drying. Both energy and mass transfers take place during the drying process. The former include conduction, convection, and radiation, while the latter represent the moisture removal. Besides its application in the preservation of foodstuffs, drying is also necessary in producing easy-to-handle, free-flowing solids, reducing transportation costs, and achieving desired product quality.

3.2.1 Solar drying

Solar drying is one of the oldest drying methods and has long been used to dry fish, meat, and grains. This method has the proven capacity to generate foodstuffs of high quality with low spoilage. With the worldwide tightening of energy policies in recent decades, people have realised the importance of solar energy and developed new scientifically-proven techniques to use it more efficiently. Some new solar drying systems like sun-hoods (Andrassy, 1978) and multi-rack dryers (Mathur et al., 1989) were developed for the drying of cash crops, such as chillies, grapes, saffron, and fruits (Mathur *et al.*, 1989; Raghavan *et al.,* 2005b).

While solar drying is a cheap, easy, and popular method, its application is restricted by the long drying time and the need for favourable weather. For example, Tulasidas (1994) showed that 6-9 weeks were required to dry grapes to a water content of 25-35%, and further steps were still required to dry them completely.

3.2.2 Hot air convective drying

The principle of hot air convective drying is based on conventional heat transfer from heated air to the materials being dried. Hot air is forced through the materials and drives the moisture diffusion process that results in drying. This method has been widely used in industry. Different types of dryers (tunnel, belt-through, and pneumatic conveyor) have been developed and employed in commercial production (Jayaraman and Gupta, 1995). In hot air drying, the inlet air temperature, air velocity, physical properties of the foodstuff, and design characteristics of the drying equipment can influence the drying rate and the final outcome.

Compared to solar drying, hot air convective drying can greatly shorten the drying time from several weeks to several days. However, some studies have reported that the taste, colour, and overall quality of dried food products could be improved by using alternative methods, such as microwave drying (Tulasidas, 1994).

3.2.3 Freeze-drying

Not all products can be exposed to high temperature during the drying processes. For example, some pharmaceuticals are heat-sensitive. Similarly, some fruits and vegetables lose their aroma and flavour if they remain at high temperatures for a significant period of time. For such cases, freeze-drying is an alternative.

Defined as a drying process in which the solvent and the medium of suspension are crystallized at low temperature and thereafter sublimated from the solid state directly into the vapour phase (Oetjen, 1999), freeze-drying was introduced on a large scale in World War II, when it was used in the production of dried plasma and blood products (Barbosa-Canovas and Vega-Mercado, 1996). The primary object of freeze-drying is to preserve biological materials without injuring them by freezing the water they contain and then removing the ice by sublimation. Freeze-drying requires several successive steps, including pre-freezing, primary drying, secondary drying conditioning, and rehydration. While freeze-drying is critical for blood plasma and certain foodstuffs because it stops the growth of micro-organisms, inhibits deleterious chemical reactions, and maintains a product's integrity along with an excellent rehydration capacity, its greater expense and technical sophistication render it difficult to apply to all commercial drying needs.

3.2.4 Vacuum drying

Unlike freeze-drying where water sublimates from the frozen state, under vacuum drying, water evaporates from its liquid state and the material is subjected to a low-pressure environment, such that the boiling point of water inside the material is reduced. During such a drying process, the main heat transfer modes are conduction and/or radiation. Improved product quality is associated with low temperatures and reduced oxidation.

For reasons similar to freeze-drying, vacuum drying is also an expensive drying method. It is only used for costly products like citrus juices, apple flakes, and heat-sensitive products.

3.2.5 Microwave drying

The potential of microwave energy for thermal processing of agricultural commodities was recognised in the 1950's. Due to economic and technical barriers, only in recent decades the low cost, mass-produced domestic and industrial microwave equipment have found applications in the drying of food and biological commodities.

Compared to traditional drying methods, microwave drying is a new and distinctly different drying method. Convection drying depends on the relatively slow processes of convective heat transfer from the medium to the surface, followed by conductive transfer to the interior of the materials. By contrast, heating with microwave energy is a volumetric process in which the electro-magnetic field interacts with the material and causes instantaneous heating of foodstuffs.

Microwave heating and drying present the following advantages over conventional thermal heating/drying methods (Mullin, 1995; Orsat *et al.*, 2006; Raghavan *et al.*, 2005a; Sanga *et al.*, 2000).

1) Heating is instantaneous due to radiative energy transfer, hence the surface-to-centre conduction stage is largely eliminated. Moreover, rapid, efficient and accurate control of heating rates can be achieved by controlling the output power of the generator.
2) During conventional drying, moisture is initially evaporated from the surface while the internal water diffuses to the surface slowly. Under microwave drying, internal heat generation leads to an increase in internal temperature and vapour pressure, both of which promote liquid flow towards the surface, thus increasing the drying rate.
3) More of the applied energy is converted to heat within the target material, because transfer of energy to the air, oven walls, conveyor, and other parts is minimal given their low dielectric constants. This can result in significant energy savings.

4) Drying time can be shortened by 50% or more, depending on the products and the drying conditions.
5) Microwave drying equipment occupies less space and reduces handling time.
6) Microwave drying improves product quality and, in some cases, eliminates case hardening, internal stresses, and other problems of quality such as cracking. The exposure to high temperatures is shorter, resulting in less degradation of heat-sensitive components such as vitamins and proteins.
7) Microwaves can be conveniently combined with other methods of drying, such as hot air drying, freeze-drying, and the application of a vacuum.

Microwave drying of grapes is not only faster but also requires less energy consumption than conventional drying (Tulasidas, 1994). In the drying of osmotically pre-treated strawberries or blueberries, Venkatachalapathy (1998) showed that microwave drying required shorter drying time than freeze drying, while maintaining the same final product quality. Sanga *et al.* (2000) also reported that the use of microwaves in freeze-drying could substantially increase drying rate and, consequently, decrease drying time.

Beaudry *et al.* (2001) compared hot air drying, freeze-drying, vacuum drying and a combination of hot air and microwave drying of cranberries. She concluded that microwave-assisted hot air drying resulted in the shortest drying time and acceptable colour, taste and texture. Sunjka *et al.* (2003) compared microwave-assisted vacuum drying to microwave-assisted hot air drying and concluded that the microwave-assisted vacuum drying offered a slight advantage in product quality and process efficiency. Liang *et al.* (2003) dried flowers with microwaves in conjunction with a colour-protecting treatment, which offered a number of advantages over conventional methods, including faster heating, more uniform drying, and little variation in colour values.

However, microwave-drying systems are not without disadvantages (Sanga *et al.*, 2000). These disadvantages can be summarized as follows:

1) High initial cost for purchase and installation;

2) Possible aroma loss and colour change due to charring or scorching;
3) Possible physical damages caused by localized areas of continuously rising temperatures;
4) Specific sample sizes and shapes of products are usually required because of microwave's limits of penetration.

3.3 Microwave and power control technologies

Given the low-cost, well-established and stable technology of commercial microwave ovens, most microwave drying experiments have used such units as they are, in modified forms, or in user-constructed units (Beaudry *et al.*, 2001; Liang, *et al.*, 2003). The power of the microwave can be varied by controlling the high voltage transformer with different techniques.

3.3.1 Microwave technology

Microwave technology was developed during World War II, when vacuum tubes termed magnetrons were invented and perfected. These magnetrons were capable of generating many kilowatts of electromagnetic power at previously unattainable frequencies (Buffler, 1993). By the middle of the twentieth century, microwave ovens had been made available for commercial and consumer use. Sales rose from 100,000-125,000 units per annum in 1971 (Zante, 1973) to over one million units per annum in 1975 (Buffler, 1993). By 1985, over 50% of U.S. households owned microwave ovens, and food companies had begun to develop microwaveable products (Buffler, 1993). Today, the microwave oven is need daily in almost every household.

Fig. 3.1 (Buffler, 1993) shows the block diagram of a microwave oven. The line power (110 V in Canada) is converted to 4 kV by a high voltage transformer and supplied to a magnetron, which generates the microwave. The microwave is guided to an applicator through a waveguide for heating or drying.

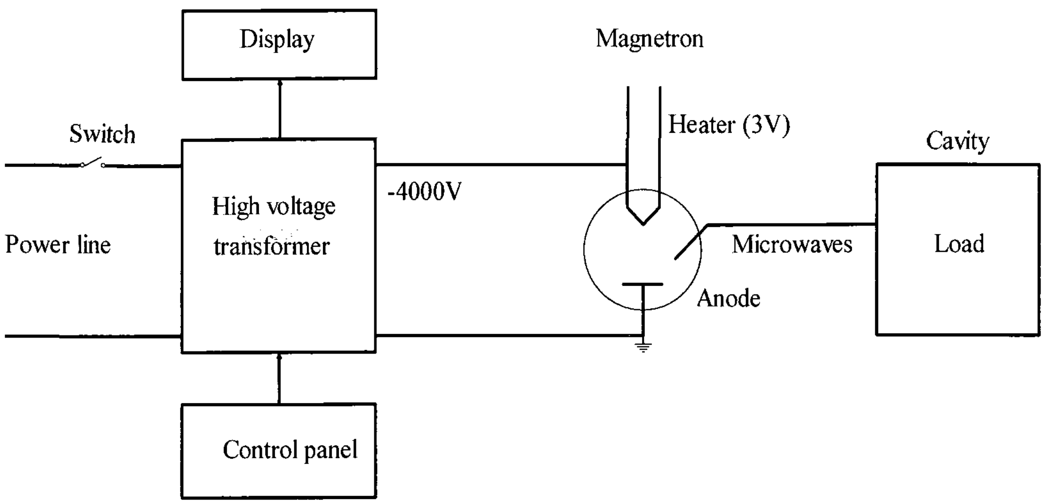

Fig. 3. 1 Block diagram of microwave oven
(Buffler, 1993)

Conventional modes of foods heating include conduction, convection, and radiation. Microwaves are not heating in themselves. Microwave-generated electric fields are a form of energy, converted to heat through their interaction with charged particles and polar molecules (Buffler, 1993).

Microwave ovens heat by radiation, using a monochromatic form of electromagnetic waves. The waves are of a single wavelength, longer than those used in conventional radiation, but shorter than those of common radio waves (Zante, 1973). The present range of frequencies defined as microwaves is from 0.3-300 GHz, with corresponding wavelengths of 1.0 m to 1.0 mm. Under microwave heating, heat is transferred to the food by ionic and dipolar interactions.

3.3.1.1 The Magnetron

As the heart of the microwave oven, the magnetron is a device that efficiently produces continuous electromagnetic waves that serve as the source of energy within the microwave cavity. The magnetron is a specialized vacuum tube surrounded by a support frame and cooling fins. An antenna, generally mounted atop the tube, radiates the microwaves and transfers them to a cavity or applicator. Within the magnetron there exists a cylindrical copper tube, capped at both ends with copper plates to maintain a vacuum. The tube is equipped internally with 12 coppers plates or vanes, which do not extend completely to the center, but leave an empty cavity where a spiral wire filament is located (Buffler, 1993).

When the filament is heated, it emits electrons, forming an electron cloud in the centre of the vacuum tube. When 4 kV are applied between the vanes and the filament, an electric field is produced that rapidly accelerates the electrons away from their cloud toward the vanes. The magnetic field generated around the vanes causes the electrons to move in a curved path rather than a straight line as they jump from the cloud to the vanes. If the strength of the applied magnetic field is adjusted properly, the electrons will just skim by the tip of the vanes without striking them.

The twelve vanes form two electric circuits, with adjacent vanes being in different circuits, and every other vane being connected by electrical connections termed strapping. As an electron approaches a vane, it induces an equal but positive charge in this vane group, while the other vane group develops an equal and opposite (negative) charge. Consequently, adjacent vanes have alternating positive and negative charges. Because unlike charges attract and like charges repel, electrons adjacent to positively charged vanes would be accelerated, while those adjacent to negative vanes would be retarded. The retarded electrons fall behind and are met by the accelerated group forming clusters of electrons. As these electron clusters remain within the circular enclosure of the vanes, the positively charged vane group would begin to alternate between positive and negative charges, while the initially negatively charged vane group will alternate in a matching but oppositely charging sequence. If the velocity of these electrons is adjusted according to the space between vanes, it is possible to generate a vane group charge that alternates 2.45×10^9 times per second. Using the antenna, this alternating charge produces a radiating 2.45 GHz microwave signal (Buffler, 1993). These waves are transmitted through a wave-guide to a feed box from where they are distributed into the oven cavity.

3.3.1.2 Power supply of the microwave oven

As a magnetron of a commercial microwave oven requires 4 kV for its operation, a high voltage supply is required. For most household microwave ovens, this voltage is provided by a half-wave doubler's power supply circuit. In this type of circuit, half of the voltage, 2kV, is supplied by the transformer, and then is doubled to 4kV by a capacitor-diode combination. A transformer within the power supply portion of the system raises the voltage from the power line to that required by the supply circuit. The microwave output power from a magnetron can be varied by a number of techniques:

1) Variable voltage supply. If the line voltage supplied to the transformer can be varied, so can the magnetron output power. Variable transformers and electronic circuits are available for this purpose, but are seldom used because of their high cost. The sensitivity of output power to line voltage is also a concern to microwaveable-food developers, as well as the consumer.
2) Resistive control. A resistance, serially connected to the diode in the capacitor-diode combination (the capacitor-diode combination used to raise the 2kV to 4kV) can limit the charge, hence reducing the voltage to below 4 kV. Power can then be controlled by switching between resistors. This technique is inefficient because the resistor must dissipate unused power, thus wasting heat and energy. Furthermore, the heated resister must be cooled by some means. As a result, this concept has been used sparingly.
3) Capacitive control. By switching capacitors, the voltage supplied to the magnetron can also be changed. This technique is difficult to implement because the switching must take place at high voltages. It has thus seen only limited applications.
4) Duty cycle control. Almost all microwave ovens use duty cycle control to vary oven power. The power is simply turned on and off periodically. The

ratio between the time the oven is turned on and the time it is turned off determines the mean power delivered to the load.

Most commercial microwave ovens apply "on-off" power control of the voltage supply (110V) to change the power output of the magnetron in an "on-off" mode (Duty cycle control). Different power levels can be achieved by different intervals of the "on" and "off" time. This method has also been extended to research experiments on the drying of bio-products. Many researchers are seeking for the best "on-off" intervals to achieve the best drying result for their specific products (Changrue *et al.*, 2004; Liang *et al.*, 2003). Even some specially designed feedback systems also use the "on-off" control of the power supplied to the magnetron to achieve temperature control in the drying process (Ramaswamy *et al.,* 1991; 1998). Other power control methods have seldom been used until now.

3.3.2 Power control strategies

While it is difficult to vary the output of the 4 kV power supply to the magnetron in a microwave oven, it is possible to control the power supplied to the primary coil of the main transformer. Four main kinds of power control methods are available: resistive power control, variable transformer, phase control, and pulse width modulation (PWM).

3.3.2.1 Resistive power control

Prior to the advent of electronic switches and PWM technologies, adjustable resistors had been widely used for controlling the magnitude of load voltages. Even today, resistive control remains in use in relay-based starters for electric motors and near-obsolete, adjustable-speed drive systems, and some are employed in the power control of microwave ovens (Beaudry *et al.*, 2001).

In some low-power electrical and electronic circuits, in which the issue of power efficiency is not of major importance, the use of small rheostats and potentiometers is still possible. However, in high-power circuits, one must consider the power losses associated with the resistors, which are unacceptable in many practical power control systems. Apart from economic considerations, large power losses in the resistor would require an extensive cooling system, because most of the lost electric energy would be converted to heat.

3.3.2.2 Variable transformer

Most current power controls of experimental microwave equipments have employed this technology, because powerstats are efficient and commercially available. However, the adjustment of the power output is usually manual and cannot be adopted in any automatic control system.

3.3.2.3 Phase control

As a widespread device for power conversion and power control, the phase-control rectifier has been used for several decades. Before new techniques such as PWM were developed, phase control had been the dominant mode of power control.

Phase-controlled rectifiers are applied in a number of different ways: half-wave resistive, full-wave resistive, half-wave inductive, and full-wave inductive. Under these applications, the AC voltage is rectified, and the beginning of conduction is delayed in each half-cycle to achieve a variable output voltage. Fisher (1991) stated that, given their ability to control large currents with relatively small pulsed-gate currents, SCR and Triac were uniquely adapted for power control. Fig. 3.2 illustrates a Triac-controlled, bi-directional power control circuit with a resistive load.

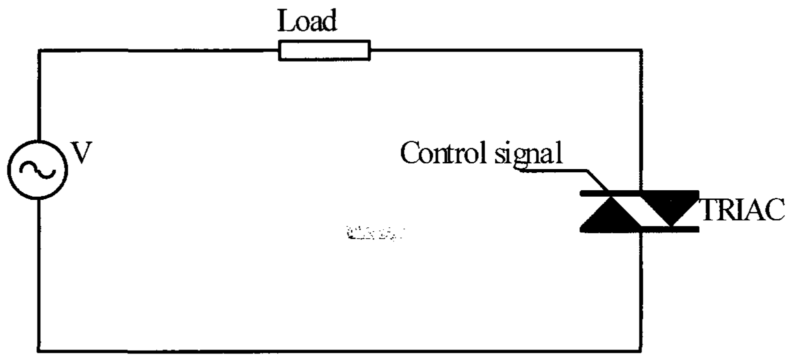

Fig. 3. 2 Principle of TRIAC control

During the first positive half-cycle of the sinusoidal supply, the anode is positive with respect to the cathode. Until the gate is triggered by a proper positive signal from the trigger terminal, the Triac blocks the flow of the load current in the forward direction. At some arbitrary delay angle, a positive trigger signal is applied between the gate and the cathode that initiates Triac conduction. Immediately, the full supply voltage, minus an approximately 1.5 V drops across the Triac, and is applied to the load (Fig. 3.3). For an inductive load, the current would continue but in the reverse direction for a finite time after the supply voltage reverses for another half-cycle. The Triac continues to conduct while the stored inductive load energy is fed back to the supply (Datta, 1985).

By controlling the delay angle with respect to the supply voltage, the phase relationship between the initiations of current flow to the supply voltage may be varied and the load current can be controlled from its maximum value down to zero.

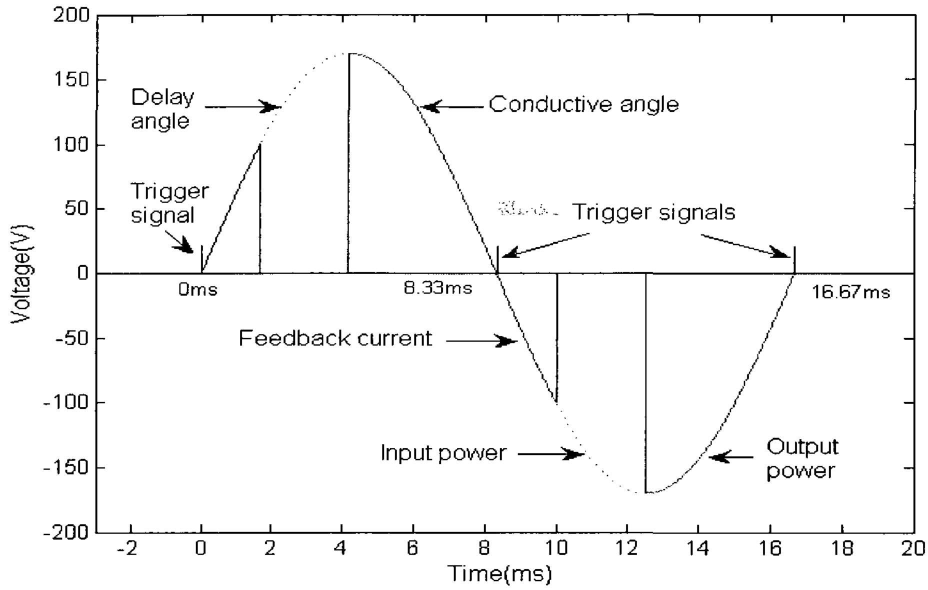

Fig. 3. 3 Resistive load in phase control

3.3.2.4 Pulse width modulation

An alternate method of power control is pulse width modulation (PWM). It can offer better output power spectral characteristics than phase control and is increasingly adopted in modern electronic power converters (Trzynadlowski, 1998).

The principle of PWM is to control the source voltage by using converter switches in such a manner that the output voltage consists of a train of pulses interspersed with notches. It can be used both as a constant voltage source or an alternating voltage source, and can even convert a DC source to a variable AC source by adjusting the ratio of the pulses. By increasing the frequency of the control pulses, current changes between the corresponding "jumps" of the output voltage can be largely prevented and high quality output can be obtained. However, the allowable switching frequency in practical electronic power converters is limited by two factors: (i) transition time of the semi-conductor from the on state to the off state and (ii) speed of the control system, or the so-called switching losses in a practical switch. Moreover, because of the limitation of electrical components, only low power conversion can be achieved with PWM techniques.

3.4 Control theory

Control theory is a multi-disciplinary science that deals with the behaviour of dynamic systems. Its applications can be found in a wide range of systems such as biological, economic, political as well as engineering. From the point of view of engineering, control theory has a history of more than 2000 years and is conventionally divided into four main periods (Bennett, 1996):

1) Early control: to 1900
2) The pre-classical period: 1900-1940
3) The classical period: 1935-1950
4) Modern control: post-1955

The concepts and techniques developed in the first three periods are usually called conventional control and that in the last period is called modern control. Conventional control relies upon developing a mathematical model of the control system. The mathematical models built are often converted to Laplace transforms to facilitate expressing the system equations in the frequency domain and manipulating them algebraically. Fig. 3.4 shows a typical feedback control loop. The input to the system is a reference signal, which represents the desired control value and sometimes called set-point. This reference is fed through a transfer function G(s) to determine the output Y. The output Y is fed back through a feedback transfer function H(s). The difference of the reference signal and the feedback signal is called error or tracking error and used to determine further control. The system serves to bring the output as close as possible to the desired reference input. Due to the complexity of the mathematics, conventional control methods are used mostly for Single-Input-Single-Output (SISO) systems.

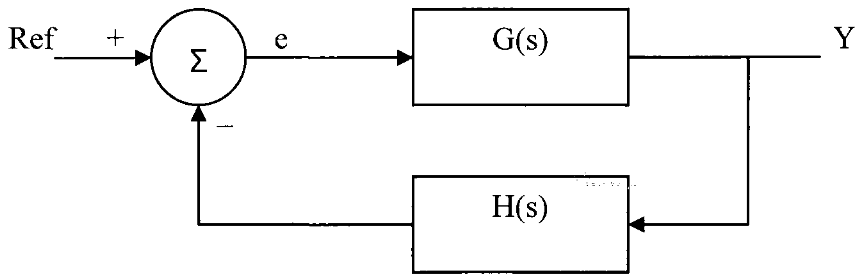

Fig. 3. 4 A feedback control loop

With the advent of computer technologies, modern control methods were developed with a realization that control system equations could be structured in such a way that computers could efficiently solve them. State space equations are employed to describe the control systems. The canonical form of state space equations is given as:

$$\dot{\vec{x}} = A\vec{x} + B\vec{u} \qquad (3.1)$$

$$\vec{y} = C\vec{x} + D\vec{u} \qquad (3.2)$$

where,

$\dot{\vec{x}}$	is a vector representing the "change in state",
$\vec{x}$	is a vector representing the system "state",
$\vec{u}$	is a vector of inputs,
$\vec{y}$	is a vector of outputs, and
A, B, C, D	are constant matrices defined by the particular control system.

Modern control methods can handle complex, nonlinear, and Multiple-Input-Multiple-Output (MIMO) systems. Optimal control, nonlinear control, adaptive control, fuzzy control, etc., have already been successively applied in many fields. Their good performance and cost efficiency have made modern control systems highly desirable.

3.4.1 PID control

In a linear system with continuous variables, a commonly used strategy for process control is the PID control (proportional, integral, derivative). The desired signal is usually subtracted from the feedback signal and a tracking error is obtained. The output of the PID controller is thus a manipulated function of the tracking error. Just as in many other conventional control strategies, the time domain function of the controller is transformed to a frequency domain function by a Laplace transform and a transfer function is obtained to facilitate system analysis.

In proportional mode, the output signal of the controller is proportional to the tracking error. A typical transfer function of a "P" controller is expressed in equation 3.3, where k represents the proportional gain of the controller.

$$g(s) = k \qquad (3.3)$$

In integral mode, the controller responds effectively to tracking errors that build up over time. This is a very important feature because even if the error is small, as long as it persists, a large control signal may be calculated, thus helping to eliminate the error quickly (equation 3.4).

$$g(s) = k\left(\frac{1}{\tau_I s}\right) \qquad (3.4)$$

In derivative mode, the control signal responds to the rate of change of the error signal. The role of this mode is to extrapolate the change in the error and provide an anticipatory control action. Its transfer function is in equation 3.5.

$$g(s) = k(\tau_D s) \qquad (3.5)$$

In practice, the integral and derivative modes are seldom used alone and they are frequently combined with "P" mode to form a PI or PID controller. The performance of a PID controller can be judged by its response speed, stability, and the level of overshooting. To achieve better performance, PID parameters in some systems are adjusted in the control process to meet the design requirements, thus resulting in an intelligent PID controller.

The restrictions of a PID controller include: 1) the system needs to be linear; 2) a reasonably accurate model needs to be available; 3) if the differential mode is included in a controller, a small amount of noise can cause a large amount of output change, i.e., it is noise vulnerable. If a mathematical model of a system is unknown or nonlinear, or if the noise of the system can not be effectively removed before the signal reaches the PID controller, more advance control strategies must be applied.

3.4.2 Fuzzy logic control (FLC)

Since the introduction of fuzzy sets by Zadeh (1965), fuzzy logic control has been developed (Mamdani, 1974) and applied to a number of processes ranging from ship autopilots, subway cars, helicopters, and pilot-scale steam engines to cement kilns, robotic manipulators, fish processing machines, and household appliances (de Silva, 1995). A common feature of these applications is that modeling and control of the systems rely on intuition, sound engineering judgment, past experience, and heuristic procedures. Fuzzy logic control makes it possible to mathematically describe and handle vague, imprecise and complex variables and converts a linguistic control strategy based on an operator's knowledge into an automatic control strategy.

Fuzzy logic controllers can be classified into seven types (Wang and Tyan, 1994):

1) FLC for input selection: The purpose is to select the right input from a set of input functions or a specific value out of a finite choice of parameters.

2) FLC for feedback error/output control: To generate an input control based upon the error/output information resulting from feedback.
3) FLC for controlling parameters of dynamic system.
4) FLC for choosing the best compensator: It is the generalization of Type 3 where the decision is applied to the compensator or subsystem.
5) FLC derived from multiple performance indices.
6) Fuzzy linguistic rules as mathematical model for unknown or complex system dynamics. This type of FLC deals with complex dynamic systems which are difficult to model mathematically.
7) Higher level fuzzy linguistic controller. This is designed to control not one, but many conventional control systems.

The fuzzy controller design procedure consists of the following steps (Zimmermann, 1991):

1) Selection of input/output variables;
2) Definition of rules/fuzzy subsets, such as triangle, rectangle, or singleton;
3) Development of the inference mechanism;
4) Selection of defuzzification strategy, such as centroid, mean of maxima, threshold method, etc.

Just as any other control strategy, fuzzy logic control has its *pros* and *cons*. The advantages of FLC include: 1) increased robustness; 2) a few rules encompass great complexity; 3) FLC is rule-oriented, the order of rules is arbitrary; 4) FLC is tolerant to redundant or contradictory rules; 5) the method can be easily extended to multi-input-multi-output systems (Lee, 1990). The limitations or drawbacks of FLC are that: 1) the response of FLC is difficult to predict with analytical tools and simulation is often the only course of evaluation; 2) it is difficult to program in machine languages.

CONNECTING STATEMENT 1

Background information on food flavour, food drying, and aroma research related to food drying processes was provided in Chapter I. A comprehensive literature review of flavour research, microwave drying, power control methods, and process control strategies was presented in Chapter III. In the current chapter, a description is given about the aroma detection ability of zNose and it is tested for Chinese spirits, which served as representative liquid foodstuffs. Dielectric properties were also studied to predict alcohol concentration.

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Assessment of Chinese Spirits Using Electronic Sensory Tools

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Contributions made by different authors are as follows:

The first author, the PhD student, did the experimental work and prepared the manuscript; the second and third authors are the supervisors who guided the research work; the fourth author is a senior engineer in the Fen Chiew Company who gave technical support regarding the spirit products.

CHAPTER IV

ASSESSMENT OF CHINESE SPIRITS USING ELECTRONIC SENSORY TOOLS

4.1 Abstract

The flavors of Chinese spirits were evaluated using a fast GC analyzer (zNose^{TM}). The first derivative profile, retention time, and peak area were used to discriminate samples at various grades. Principal component analysis (PCA) and canonical discriminant analysis (CDA) were adopted for data analyses, where 100% classification result was achieved. A network analyzer was also included in this study in order to explore the possibility of estimating the concentration of alcohol using the spirit's dielectric properties. The dielectric constant and loss factor of 0-100% ethanol-water mixtures in 10% interval and 42%, 50%, 53%, 62%, 64% ethanol-water mixtures, as well as spirits samples were measured and compared. Alcohol concentration was shown to be the same order as the dielectric properties.

Keywords: zNose, Chinese spirits, dielectric properties, PCA, CDA

4.2 Introduction

Chinese spirit, or white liquor, is one of the six most popular distilled alcoholic beverages (brandy, gin, rum, vodka, whiskey, and Chinese spirits) in the world. With hundreds of brands on the market, each Chinese spirit has its own distinctive and unique flavour. The Liquor Association of China groups them into five categories: Luozhou, Fenjiu, Maotai, Shanhua, and others (Zhang *et al.*, 2005). Among them, Fenjiu is a famous brand with a typical "Fenjiu" flavour which has a history of over 1500 years. It is widely consumed in the northern region of China and has reached a production of more than 50,000 tons in recent years (Fenjiu Group, 2007).

Flavour is the most important grading criteria for all Chinese spirits; hence it is the same for Fenjiu. Though they only account for 1-2% of the total content by volume, acids, esters, aldehydes and ketones have been found to contribute to a spirit's specific flavour. Different Chinese spirits have different combinations of these compounds, thus creating their own special flavours. A Fenjiu spirit may include 200 chemical compounds and this number is still increasing as new and more advanced instrumental techniques are developed. Currently, for both consumers and producers, the identification and grading of Chinese spirits is mainly dependent on the use of oenophiles. However, oenophiles are frequently influenced by their physical and mental condition and can only work for a short period of time in a working day (Li *et al.*, 2006a). Gas chromatography (GC) has been employed for the same purpose (Wu, 2001; Zhang *et al.*, 2005), but it is complicated and time-consuming. Moreover, even if all the compounds in a specific spirit can be identified by GC, it is still difficult to relate the many peaks with the classification derived by oenophiles.

In recent years, the electronic nose (e-nose) has been applied in food flavour detection. With respect to alcoholic beverages, studies in wine identification and classification have been reported (Li *et al.*, 2006a), but seldom have Chinese spirits undergone such testing. Zhang *et al.* (2004) measured five different types of Chinese spirits with doped nano-ZnO sensors and concluded that it was possible to identify different flavour types, but that classification within a given type was difficult. Also, the response of a metal oxide sensor may drift with the presence of moisture and alcohol, which are rich in a spirit headspace. Hence, there is a need to develop a new method to classify and identify Chinese spirits, especially within the same flavour type, or even in the same brand with different grades.

Since 2002, a new e-nose, called zNose™ (7100 Fast GC Analyzer, Electronic Sensor Technology, New Bury Park, CA, USA), has been introduced and is commercially available (Lammertyn *et al.*, 2004). In principle, the zNose™

is a fast GC with a surface acoustic wave (SAW) sensor. Separating compounds with a GC column and detecting the quantity of a compound by the SAW sensor, the instrument can give a fingerprint of a flavour, as well as detailed information about retention time and peak area within a few minutes. Flavours of honey (Lammertyn *et al.*, 2004), apple (Li *et al.*, 2006b), and vegetable oil (Gan *et al.*, 2005) have been measured by zNose^{TM}. With a specialized GC column (DB-5), zNose^{TM} can collect and detect chemicals which have 5 to 22 carbon atoms (C_5-C_{22}). On one hand, this ability is quite suitable for Chinese spirits because it avoids the collection of water and ethanol, which have less carbon atoms and contribute less to the flavour, but are the mass components in a spirit. This ability also overcomes the difficulty faced by metal oxide sensors in a regular e-nose. On the other hand, zNose^{TM} lacks the ability of detecting water and ethanol which account for >98% of the spirit by volume and act as carriers for all the flavours. To electronically detect the quantity of water and ethanol in Chinese spirits, measurements of their dielectric properties were made in this study.

Dielectric properties, or permittivities, have been historically associated with the design of electrical and electronics equipment in certain frequency ranges. With the advent of commercial microwave ovens, an intensive study of dielectric properties has been conducted on agriculture materials and products in terms of their heating and drying (Nelson, 1999). Applications have also extended to the measurement of moisture content (Nelson, 1977), fruit maturity (Nigmatullin and Nelson, 2006) and in quality evaluation (Kuang and Nelson, 1997). Measurements of dielectric properties have also been conducted for alcohol, methanol (Smith *et al.*, 1998) and ethanol (Liao *et al.*, 2001) where they were used as solvents in food drying. These studies inspired us to take advantage of the potential use of the measurement of dielectric properties to characterise the primary constituents of Chinese spirits.

The objectives of this study included:

1) Grading and classifying Fenjiu spirits based on their aroma profiles;
2) Evaluating alcohol concentration by dielectric spectroscopy;

3) Investigating the possibility of predicting alcohol concentration of spirits by dielectric properties at certain frequencies.

4.3 Materials and Methods

4.3.1 Sample preparation

Six Fenjiu samples, Guocanfenjiu, Laobaifen, Yinianfenjiu, Bofen, Xinghuacun, and Chuanzhenjiu were provided by the Fenjiu Group (Shanxi Xinghua Cun Fen Chiew (Group) Co., Ltd., Xinghua Cun, Fenyang, Shanxi, P.R. China). They were graded by age and flavour quality in the factory. The alcohol concentrations of six Fenjiu samples were measured with an alcohol hydrometer.

Analytically pure anhydrous ethyl alcohol (Commercial Alcohol Inc., Brampton, Ontario, CA) and distilled water were used to make two sets of mixtures: 0%-100% ethanol-water mixtures at 10% interval, a total 11 mixtures, as well as 42%, 42%, 64%, 50%, 62%, and 53% aqueous ethanol mixtures, i.e., the same concentration as six Fenjiu samples.

Seventeen samples from each of the six fenjiu samples, for a total of 102, were evaluated using zNose. In the dielectric properties experiment, the dielectric constant and loss factor of each ethanol-water mixtures and Fenjiu samples were measured with three replicates.

4.3.2 Aroma evaluation

In this study, a zNose was used for the detection of volatile compounds. This is actually a miniature, high-speed gas chromatograph (GC) containing a detector, a short separation column, and support electronics. The detector of the zNose is an uncoated, high quality piezo-electric quartz crystal. The crystal operates by maintaining high frequency acoustic waves on its surface. The measured compound lands and sticks on the detector and changes its frequency.

The frequency change (in "Counts") was measured by a microcontroller and processed by software, allowing the compound to be identified and quantified. Before the volatile compounds arrived at the detector, they were separated by a short column, containing an internal coating of a bonded liquid phase. Volatile compounds to be dissolved in this liquid phase exit the column at different times, and this separation is further enhanced by heating the column in a programmable temperature profile. The time one compound is retained in the column is recorded as its retention time (RT), which is unique to each specific component. The area of each peak is considered as qualitative measurement of the quantity of the volatile. The zNose employs a headspace or bubbler technique as its sampling mode. A side Luer needle was used as a sample odour injection tool, and a spark needle as the bubbler generator. A rotating valve was used to shift the machine from a sampling to injection position, a trap was used as a pre-concentrator to collect and hold volatile samples, and high grade helium was used as the carrier gas.

Each spirit sample (5 mL) was placed in a 40 ml vial (98 mm tall and 28 mm outer diameter) sealed with a screw cap equipped with a septum. After 1 hour at room temperature headspace samples were tested. Sampling occurred through a side luer needle (10 s), followed by separation of different compounds on the column (14 s), data acquisition every 0.02 s for 20 s, and baking the sensor for 30 s. The sensor detection temperature was set to 60°C, column temperature was ramped from 40°C to 180°C at a rate of 10°C/S, the sensor baking temperature was 150°C. The carrier gas flow rate was 3.0 $cm^3 s^{-1}$. Between measurement cycles, at least one air blank was conducted to flush the system until the baseline signal peaks were all under 200 counts.

One of the major problems of the zNose is the drift in retention time, which may be influenced by the pressure of the carrier gas, the system's temperature, and the column status. To minimize the drift, the zNose was preheated for 2 hours to allow stabilization before measurements were made. An n-Alkanes standard solution was used to calibrate the system every hour. n-Alkanes are widely used standards in GC analysis, permitting the conversion of retention time to a Kovats index (an index permitting to identify organic compounds of a mixture, see Appendix C).

4.3.3 Dielectric properties

An Agilent 8722ES S-parameter network analyzer (Agilent Technologies, Santa Clara, CA, USA) equipped with a 2 mm diameter 85070E open-end slim probe was used to measure the dielectric properties of the alcohol samples. The frequency range was set to 100 intervals (101 points) in the range of 200 MHz to 40 GHz. The dielectric constant (ε') and loss factor (ε'') were recorded and analyzed. The probe was cleaned after each measurement.

4.3.4 Data analysis

Nine prominent peaks were chosen from the first derivative plot of each aroma profile. Corresponding peak areas, representing the amount of different volatile compounds in the spirits samples, were calculated using the zNose system's software (MicroSense 4.0) and these values were used in data analysis.

Using the MATLAB program platform (MATLAB 7.0, The Mathworks, Inc., MA, USA), principal component analysis (PCA) was conducted to classify six Fenjiu samples. Error bars of all samples at two typical frequencies (5.37 GHz for ε' and 20.1 GHz for ε'') were plotted.

The PCA technique uses a linear combination of the original variables, which may be dominated by large but not distinctive peak values. To improve the classification model, canonical discriminant analysis (CDA) was attempted, which accentuated the differences among different sample groups. Canonical discriminant analysis (CDA) in SAS/STAT (SAS 9.1, SAS Institute Inc, Cary, NC, USA) was conducted to process the measured data.

4.4 Results and Discussion

The characteristics of the spirit samples tested in this study are summarized in Table 4.1. Samples 1-5 are final products produced by standard fermentation methods and their differences in quality result mainly from different aging treatments. Sample 6 is a raw liquor produced with a special method called "series-fermentation". This method tries to achieve the same effect as aging treatment but in a shorter time.

Table 4. 1 Characteristics of six Fenjiu samples, including relative price, aging and alcohol concentration

Sample No.	Name of Grade	Aging (years)	Order in Price	Alcohol Concentration (v/v)
1	Guocangfenjiu	50	1	42%
2	Laobaifen	10	2	42%
3	Yinianfenjiu	1	3	64%
4	Bofen	0.5	4	50%
5	Xinghuacun	1	5	62%
6	Chuanzhenjiu	0.25	6	53%

4.4.1 Aroma signals

4.4.1.1 First derivative profiles

The first derivative of the frequency signal was collected, transferred to a PC through an RS-232 cable, and stored in a file. The positive values were used to form a profile of the samples, similar to those of a GC chromatograph (Lammertyn *et al.*, 2004). Plots of the positive first derivative of the calibration solution (n-Alkanes) and six Fenjiu samples from the first measurement of each sample show that most Fenjiu samples can be discriminated visually (Figure 4.1).

Their baselines were increased by 100,000 counts in this figure to facilitate visibility.

Samples 1, 2, and 6 had large values in higher retention time peaks, whereas the lower retention time peaks of all six samples were similar. By comparing sample and n-Alkanes profiles, further conclusions can be drawn: samples 1, 2, and 6 were rich in C_{10} compounds, all volatiles in Fenjiu spirits were within the range of C_5 to C_{14}, and the samples had almost no compounds of more than C_{14}.

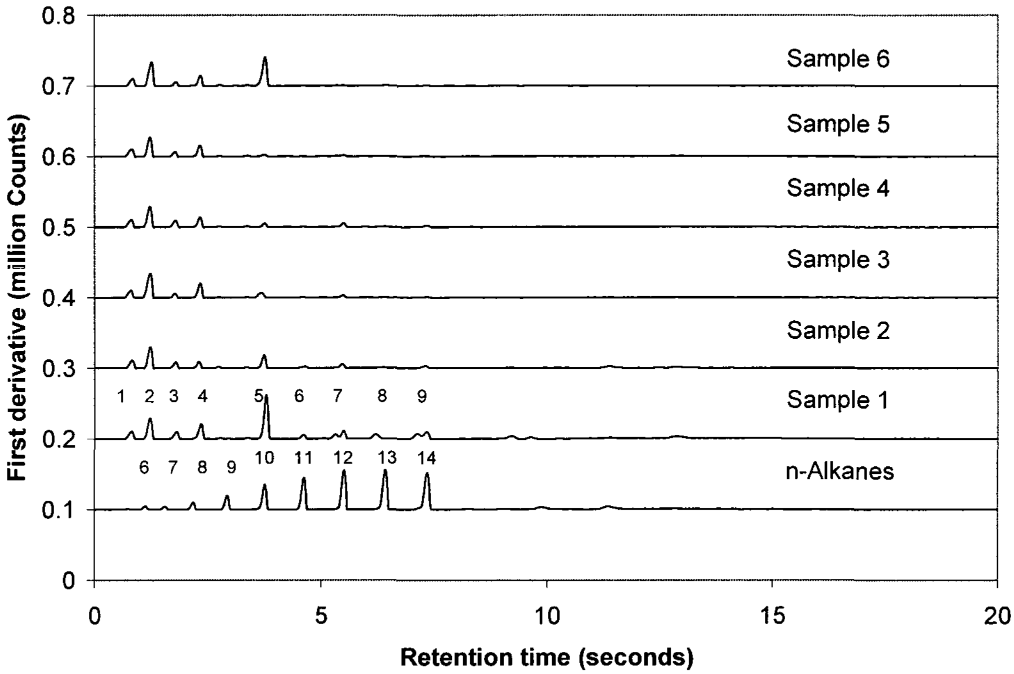

Fig. 4. 1 First derivatives of n-Alkanes (C6-C14) and Fenjiu samples (peak1-9)

The total area under peaks of positive first derivatives of each sample were also calculated (Table 4.2). The order of these values was almost the same as that of the price and aging, except for sample 3. This indicated that expensive, long-aged samples are rich in flavour volatiles, and the "order of sum" method can be used to estimate the flavour quality of Fenjiu spirits in a simple and fast way. This result also shows that the "series-fermentation" technique for sample 6 can improve product quality in a manner equivalent to an aging treatment of 1-10 years.

Table 4. 2 Sum of positive first derivative peak areas of six Fenjiu samples.

Samples	1	2	3	4	5	6
Signal*	1306611	688376	542356	507056	497576	688073
Order	1	2	4	5	6	3

* Sum of positive first derivative

4.4.1.2 PCA analysis

The different peak retention times represented different compounds and the area of each peak represents the amount of that compound. In this study, nine obvious peaks were visually selected and their areas were used for statistical analysis. As a first approach PCA analysis was conducted to classify the six samples, resulting in a 93% explanation of the total variances by PC1 and PC2 (Fig. 4.2). Evaluation of the corresponding loading plots of PC1 and PC2 showed the separation of six samples were mainly explained by the contribution of peaks 2, 4, and 5 (Fig. 4.3). From this analysis, it appeared that the nine peaks were good candidates for the classification of Fenjiu spirits.

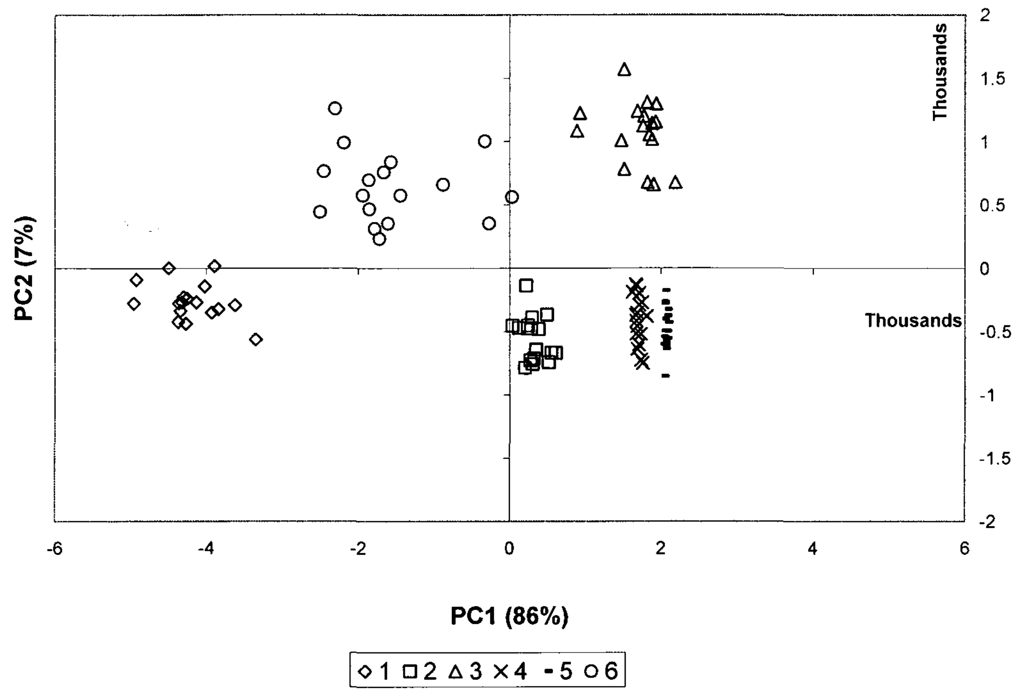

Fig. 4. 2 PCA scores plot of six Fenjiu samples

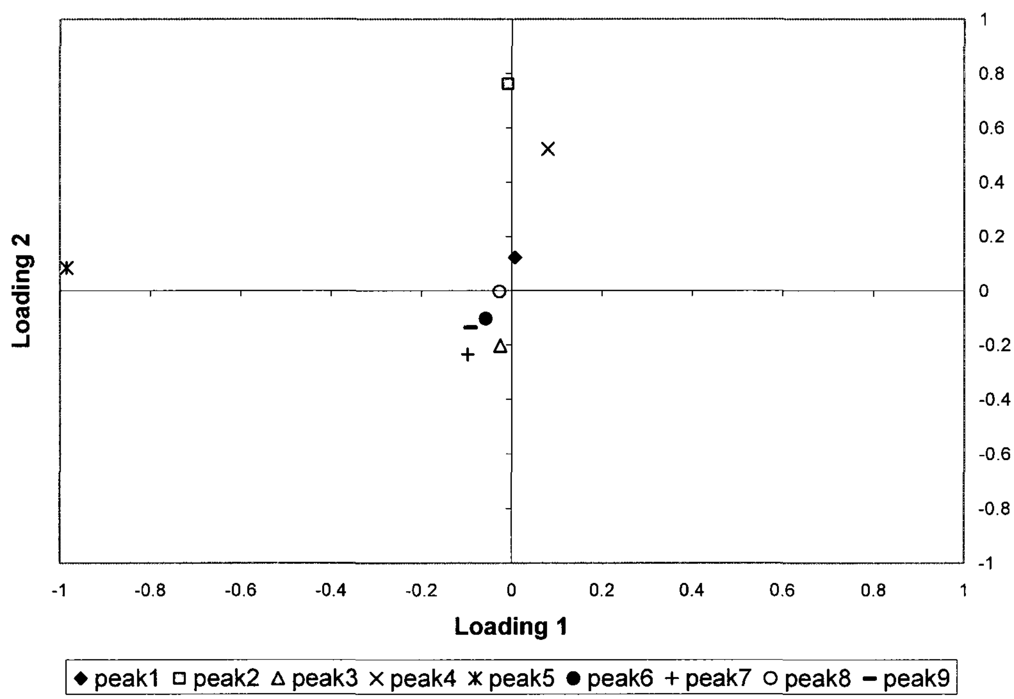

Fig. 4. 3 Loadings plot of the six Fenju samples

4.4.1.3 Discriminant analysis

The PCA analysis indicated that the areas of the nine chosen peaks were potentially valuable in building a classification model. However, to improve the classification model, a canonical discriminant analysis (CDA) was attempted, which accentuated the differences among different sample groups.

In CDA analysis, five canonical variables were calculated and a discriminant function for future classification was built upon these variables. Of 102 (17 for each sample) measurements, 84 (14 for each sample) were used in calibration and 18 (3 for each sample) for validation. This resulted in a 100% accurate classification. Most of the classification can be obtained visually from the first two canonical variates (Fig. 4.4), and any confusion between samples 3 and 6, can be cleared using canonical variates 3 and 5 (not shown).

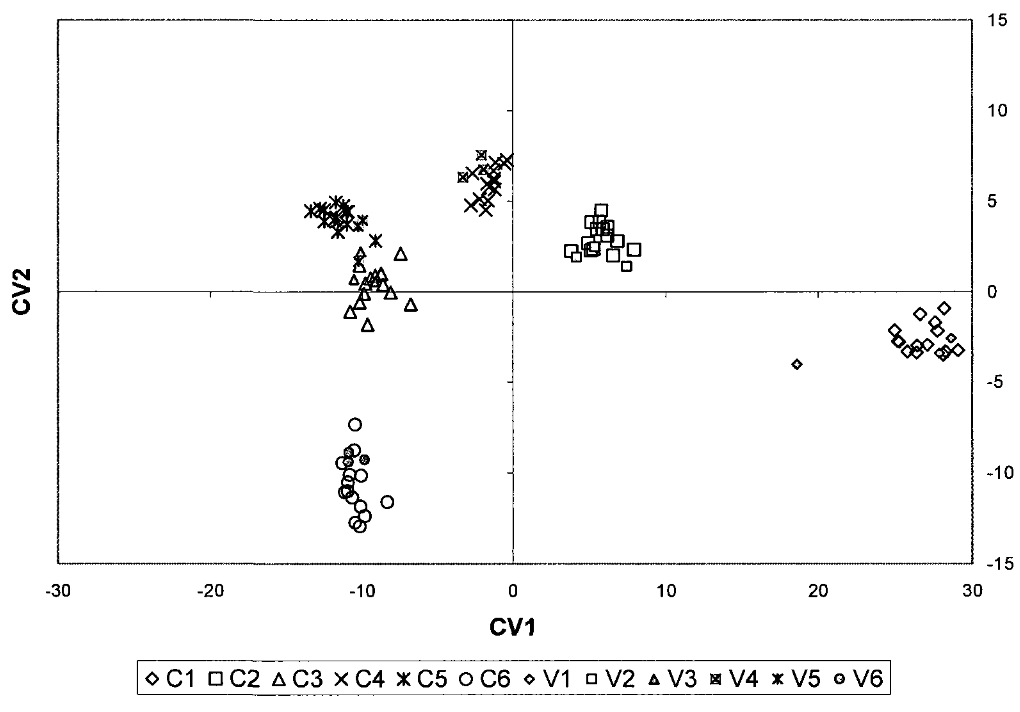

Fig. 4. 4 CDA analysis of six Fenjiu samples showing first two canonical variates: CV1 and CV2
(C1-C6: outline symbols, calibration observations; V1-V6: solid symbols, validation observations)

Zhang *et al.* (2005) used ZnO nanoparticles doped with MnO_2, TiO_2, and Co_2O_3 as electronic nose sensor array to identify five different types of Chinese spirits. Combining principal components analysis and discriminant analysis (PCA-DA), an accuracy of 76.8% was achieved. However, this method was unable to identify different Chinese spirits of the same flavour type, which would be important for liquor manufacturers to evaluate their products on a daily basis. In this study, this problem is successfully solved and the measurement method is simple and fast. Hence, the methodology developed in this study has promising applications in the liquor industry.

4.4.2 Dielectric properties

4.4.2.1 Ethanol-water mixtures (0-100%)

The dielectric constant and loss factor of 0-100% ethanol-water mixtures, in 10% intervals, were measured at room temperature in the frequency range of 200 MHz to 40 GHz (Figs. 4.5, 4.6). Measurements were repeated three times for each mixture and the standard deviation is shown at the fixed frequencies of 5.34 MHz for the dielectric constant and 20.1 GHz for the loss factor. For each mixture, the dielectric constant decreased with increase in frequency, and mixtures with a higher ethanol content had lower dielectric constant values. Given that the error bar at a fixed frequency showed a standard deviation well inferior to the difference between two mixtures differing by 10% in alcohol content; this indicated that it was possible to evaluate the concentration of a mixture in 10% intervals, by its dielectric constant within a specific frequency range, such as 300 MHz to10 GHz.

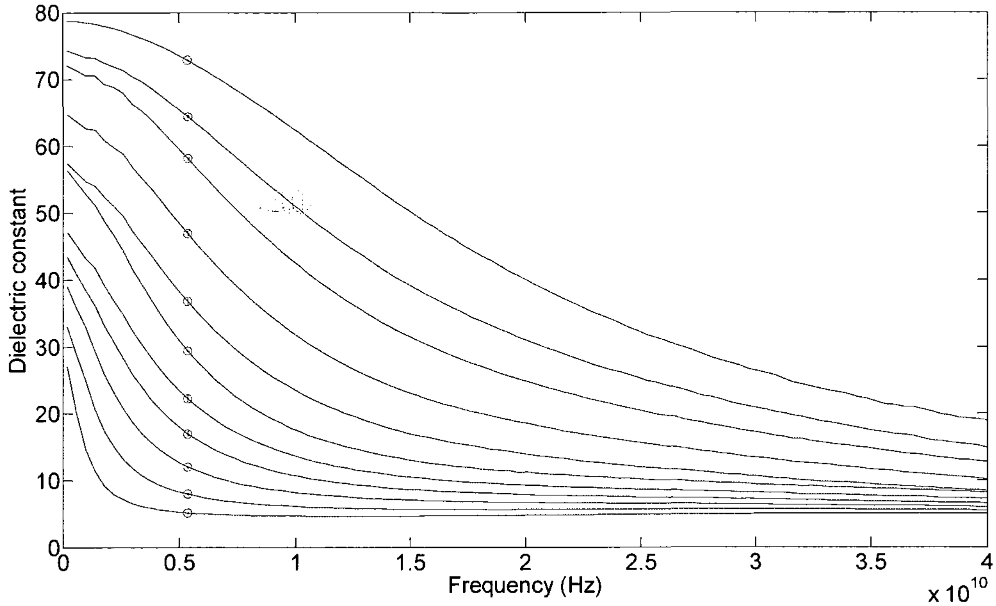

Fig. 4. 5 Dielectric constant of 0-100% ethanol-water mixtures
(from top to bottom: 0-100% in 10% interval)

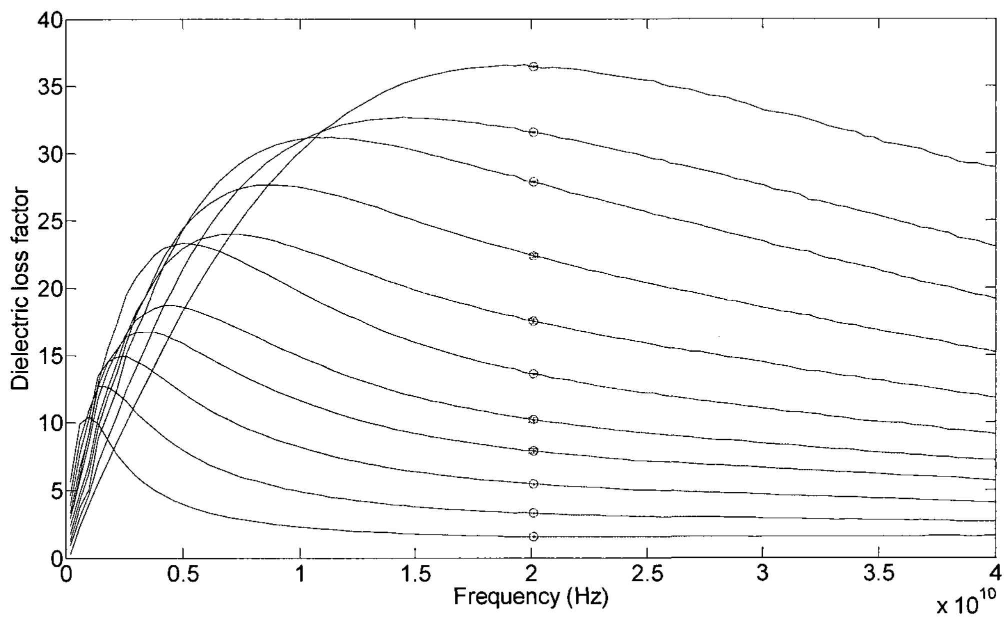

Fig. 4. 6 Dielectric loss factor of 0-100% ethanol-water mixtures
(from top to bottom: 0-100% in 10% interval)

In the case of the dielectric loss factor, for each particular mixture, the value first increased and then decreased above a certain frequency. The values of mixtures with higher ethanol contents were greater at the lower frequencies and smaller at the higher frequencies. The error bar shown at 20.1 GHz indicated that the dielectric loss factor was sufficiently accurate to evaluate the alcohol concentration in 10% intervals without confusion. This result encouraged us to make further estimations of spirits' alcohol concentration using their dielectric properties.

4.4.2.2 Five ethanol-water mixtures

To estimate the concentration of six Fenjiu samples, preliminary measurements of dielectric constant (Fig. 4.7) and loss factor (Fig. 4.8) was conducted with ethanol-water mixtures at the same concentrations as the spirit samples, i.e., 42%, 50%, 53%, 62%, and 64%.

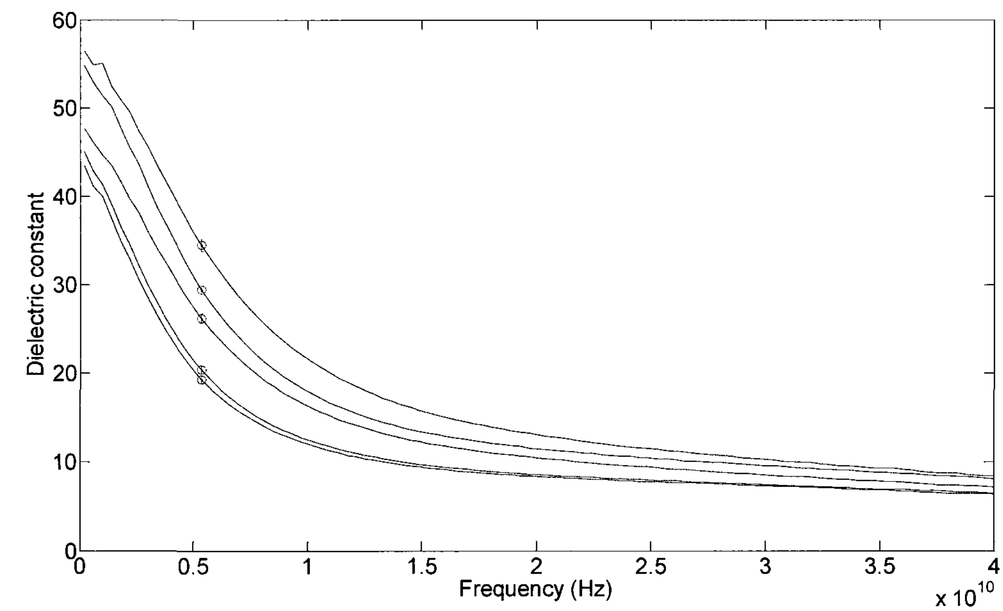

Fig. 4. 7 Dielectric constant of five ethanol-water mixtures (from top to bottom: 42%, 50%, 53%, 62%, 64%)

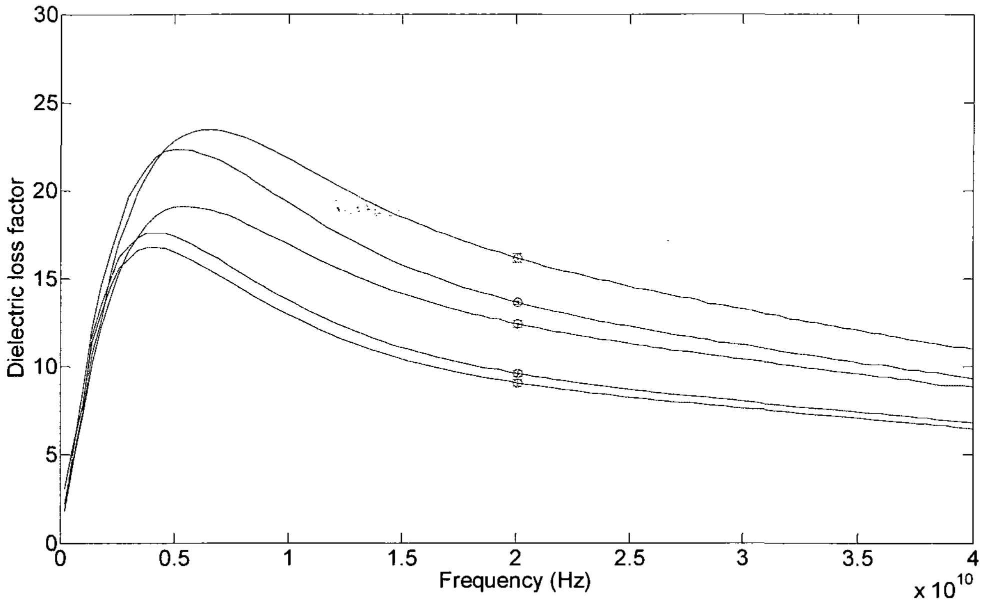

Fig. 4. 8 Dielectric loss factor of five ethanol-water mixtures (from top to bottom: 42%, 50%, 53%, 62%, 64%)

Although the differences were small, the mixtures could still be separated by their dielectric properties, in a similar way to the 0-100% mixtures, but on a smaller scale. Error bars of the standard deviations at two fixed frequencies (5.34 MHz and 20.1 GHz, for dielectric constant and loss factor, respectively) show that it is possible to discriminate sample's alcohol concentrations differing by 2%.

4.4.2.3 Six spirits

The measurements of the dielectric properties of six Fenjiu samples (Fig. 4.9 and Fig. 4.10) showed the alcohol content values to be ordered as expected and similar to those of the five ethanol-water mixtures. Small differences are found between sample 1 and 2, which had the same alcohol concentration but different flavour qualities. However, their error bars do overlap, indicating the unlikelihood of assigning different alcohol concentrations to Fenjiu samples having the same alcohol concentration.

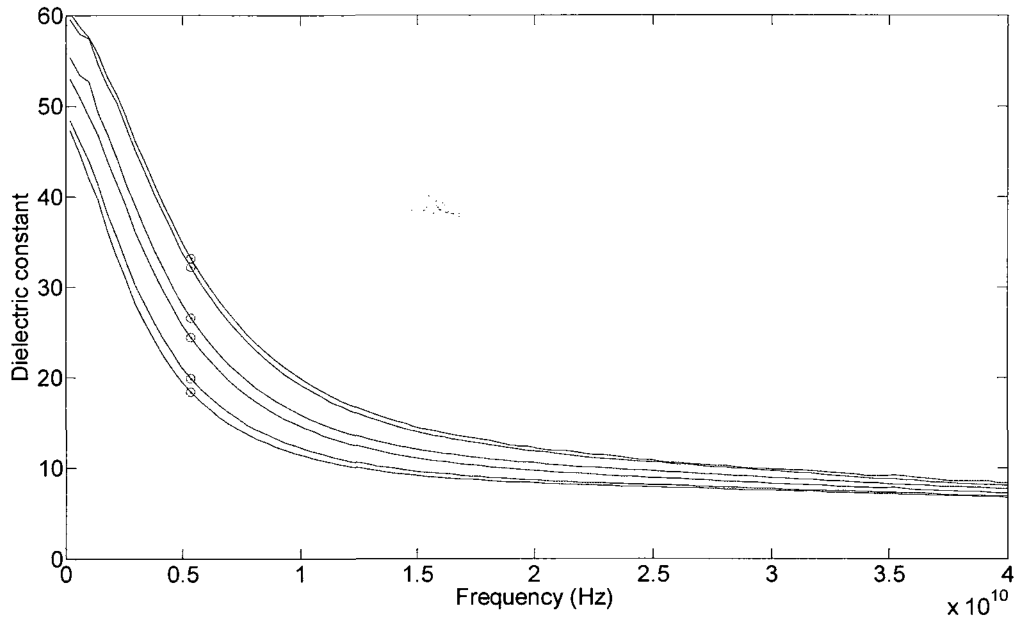

Fig. 4. 9 Dielectric constant of six Fenjiu samples
[from top to bottom: Fenjiu sample
2(42%);1(42%);4(50%);6(53%);5(62%);3(64%)]

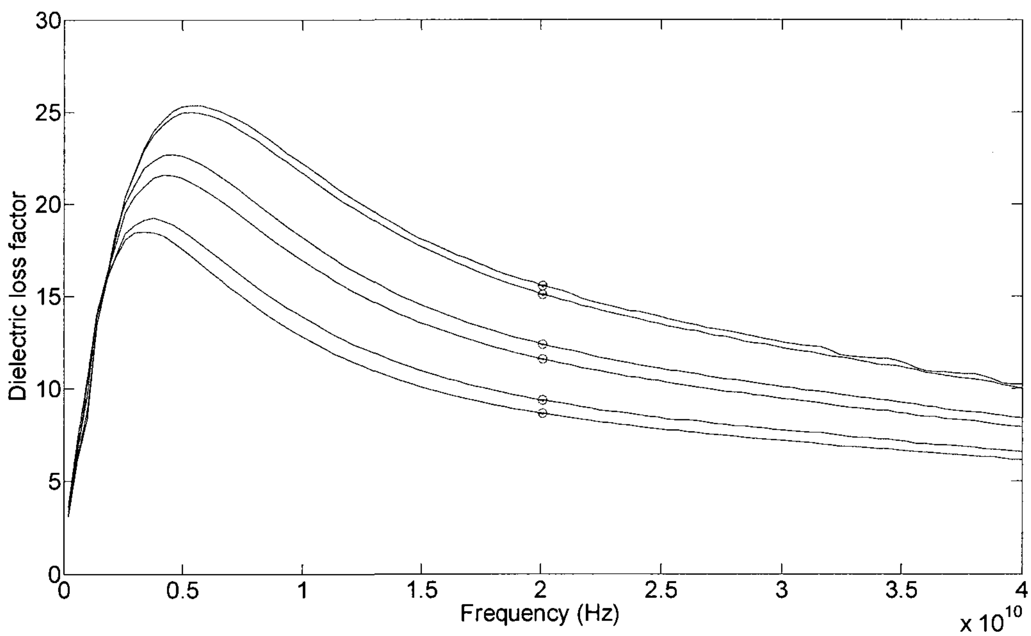

Fig. 4. 10 Dielectric loss factor of six Fenjiu samples
[from top to bottom: Fenjiu sample
2(42%);1(42%);4(50%);6(53%);5(62%);3(64%)]

Comparing these results with those of the five ethanol-water mixtures, it can be concluded that the dielectric properties are mostly determined by their ethanol and water content, which are the major components of the spirits' samples. The 1-2% of other compounds does not have a significant influence with respect to the dielectric properties of the spirits.

Liao *et al.* (2001) measured dielectric properties of alcohols (C_1-C_5) at 2450 MHz and 915 MHz in a temperature range of 25°C to 95°C. They found that at a given temperature the dielectric constant at 2450 MHz was lower than that at 915 MHz. In contrast, the dielectric loss factor at 2450 MHz was higher than that at 915 MHz. The current study confirmed that within the low frequency range (200-5000 MHz) dielectric constants decreased with an increase in frequency, while the contrary was true for the dielectric loss factor. However, in the high frequency range, the trends of dielectric loss factors reverse, where an increase of frequency leads to a decrease in the dielectric loss factors. Therefore, the measurements of dielectric properties in a wide and continuous frequency range are necessary for related applications. The result from this study not only can be used to estimate alcohol concentration of Chinese spirits, but are also useful in evaluation of the dielectric properties of alcohols when they are employed as solvents in microwave (MW) or radio frequency (RF) heating and drying applications.

4.4.2.4 The difference of dielectric properties of five ethanol-water mixtures and six Fenjiu samples

The differences in dielectric constant and loss factor of six Fenjiu samples with respect to their corresponding ethanol-water mixtures were also analyzed. No significant difference was found. This illustrated that the 1-2% compounds in the Fenjiu samples, which were not ethanol or water, were not detectable through the dielectric constant or loss factor measured in the frequency range of (200 MHz to 40 GHz). This may be the result of the equipment's sensitivity, the errors while measuring the concentration of Fenjiu samples by alcohol hydrometer, or errors from the inaccurate measurement of the concentrations of ethanol-water mixtures. These possible errors made the identification of Fenjiu samples strictly by means of dielectric properties analysis very difficult.

4.5 Conclusions

Six Fenjiu samples were well classified by their aroma signals. The retention time and peak area detected with zNose served as useful parameters for classification and prediction of Fenjiu samples. The "order of sum" method, developed in this study, is a simple and fast way to evaluate the products' flavour quality. PCA and CDA were both valuable tools in the spirits' classification and identification.

Measurement of dielectric properties is a potential method to measure alcohol content of the Fenjiu samples, and could serve as a supplement to the aroma measurements in characterizing Chinese spirits.

In this study, all the Fenjiu samples were provided by the factory of the Fenjiu Group (in China). Due to limitations in the volume of alcohol which could be imported, only 200 ml of each sample were brought to the lab and used for measurement. This limitation may account for the 100% accurate classification result by both PCA and CDA. Further experiments, in factory, are recommended in order to compare the results with the assessments of local oenophiles.

4.6 Acknowledgements

The authors acknowledge financial support from Canada Foundation of Innovation (CFI) and Natural Sciences and Engineering Research Council of Canada (NSERC). The Fenjiu Group is also acknowledged for supplying samples.

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CONNECTING STATEMENT 2

As the continuation of the study, the aroma of a solid food sample will be investigated in this chapter. Various quality aspects of mango fruits during their shelf life will be evaluated with the detected aroma signals.

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Ripeness and Rot Evaluation of Mangoes through Aroma Detection

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Contributions made by different authors are as follows:

The first author, the PhD student did the experimental work and prepared the manuscript; the second and third authors are the supervisors who guided the research work.

CHAPTER V

RIPENESS AND ROT EVALUATION OF MANGOES THROUGH AROMA DETECTION

5.1 Abstract

An ultra fast GC (zNose^{TM}), based on an uncoated surface acoustic wave sensor, was employed to detect the aroma of mango fruits. The detected aroma signals were used to identify rot occurrence and evaluate mango ripeness during shelf life. Respiration rate, color, and total soluble solids (TSS) were measured accordingly to indicate mango quality status. Two peaks detected with the zNose predicted rot occurrence with 93 and 88% accuracy, respectively, while another peak was 80% accurate in predicting ripeness with respect to a reference color index. These rot prediction methods could have potential applications in the mango industry for the ultra-fast (less than 1 minute) diagnosis of the occurrence of mango rots.

Keywords: electronic nose, mango, aroma, rot, ripeness

5.2 Introduction

Mango [*Mangifera indica* L.] is one of the most important fruits marketed throughout the world. The total production of mango in the world has steadily increased: from 22 million tons in 1995 to 28 million in 2005, of which roughly 1% were imported to North America (U.S.A. and Canada) (FAOSTAT, 2006), reflecting an increasing demand for the fruits in the world.

Mango fruits are normally harvested at the green mature stage and then transported and stored at low temperatures (10-15°C) to extend their shelf life (Snowdon, 1990). However, severe post-harvest diseases still occur, resulting in rot contributing to substantial post-harvest losses (Beyers *et al.,* 1979; Pantastico *et al.*, 1984; Pesis *et al.*, 2000). Hence, there is a need to monitor the disease severity and rot occurrence during the shelf life of mangoes in order to prevent the spread of diseases. Evaluating maturity and ripeness is another important issue for mango industry. Presently, the pre-harvest maturity of mango fruits is estimated by size, sphericity, firmness, total soluble solids (Jha *et al.*, 2006) and post-harvest ripening is often evaluated by respiration rate and skin color (Lalel *et al.*, 2003).

An attractive flavour is one of the characteristics that make mango a highly prized fruit. The varying flavour at different stages of harvest and after certain storage time is not only distinguishable by human senses, but also reflects physiological and biochemical changes. Some studies of different mango cultivars' volatiles have been undertaken (Macleod and Troconis, 1982; Macleod and Pieris, 1984; Idstein and Schreier, 1985), and flavours at different developmental stages have also been compared (Ibanez *et al.*, 1998; Lalel *et al.*, 2003). Unfortunately, all the methods used in these studies, including GC, GC/MS and SPME, are labour and time intensive, making their application outside the laboratory almost impossible.

In recent years, electronic noses have been employed to evaluate fruits flavours, and a number of applications for different fruits have been reported: apple (Saevels *et al.*, 2004), mandarin (Gomez *et al.*, 2006), orange (Natale *et al.*, 2001), and peach (Natale *et al.*, 2002). In terms of mango, Lebrun *et al.* (2008) recently achieved partial success in using an e-nose FOX 4000 to discriminate mango maturity. One of the restrictions of electronic noses is that they cannot identify the chemical compounds they detect. Also, most of the electronic nose equipments are not portable, which make *in situ* measurements difficult.

In this study, an ultra fast portable GC (zNose^{TM}), which combines the fast speed of an electronic nose and the chemical identification ability of a GC, was employed.

The objectives were:

1) To predict rot occurrence of mango fruits by detecting aroma signals;
2) To evaluate mango ripeness over the period of their shelf life.

5.3 Materials and Methods

5.3.1 Experimental materials and procedure

One hundred and twenty mango fruits of cv. 'Tommy Atkins' (*Cocanmex S.A. de C.V.*) produced in Mexico were obtained from a local fruit distribution company (Aliments IMEX Foods Inc., Montreal, Quebec) and stored at room temperature (20°C-22°C) without any treatment. Twelve mangoes as green and as hard as possible were selected as control samples and the remaining 108 were used as regular samples. The control samples were measured every day for 31 days until all were rotten. Nine of 108 regular samples were randomly chosen and measured daily; hence, the measurement of regular samples lasted for 12 days.

Airtight glass jars (2.3 litres) were used for both respiration rate (RR) and volatile measurement. An aperture was made in the metal cover, fitted with a rubber septum for gas sampling and sealed with Teflon material. The containers were tested to be free of zNose-detectable aroma volatiles when empty. A single mango was put in a container for 2 hours prior to measurement. This equilibrium time was obtained from preliminary experiments where, in 10 hours, the volatiles signal reached 80% of its highest value within 2 hours (results not shown).

5.3.2 Respiration rate (RR)

Gas samples (5 mL each) were taken from the glass jar and CO_2 was measured with a SRI 8610A Gas Chromatograph (SRI Instruments Inc., Las Vegas, USA). The sensor temperature of the Gas Chromatograph was set to 55°C and the flow pressure was 483 kPa (70 psi). The measured RR was compared with the aroma profiles acquired from the zNose in an attempt to assess ripeness of mango fruits. All measurements were repeated twice and mean values are reported.

5.3.3 Aroma detection system

A zNose (7100 Fast GC Analyzer, Electronic Sensor Technology, New Bury Park, CA, USA) was used for volatile compound detection in this study. It is actually a miniature, high-speed gas chromatograph (GC) containing a detector, a short separation column, and support electronics.

The detector of the zNose is an uncoated, high quality surface acoustic wave (SAW) crystal. The crystal operates by maintaining high frequency acoustic waves on its surface. The targeted compound lands and sticks on the detector and changes its frequency. The frequency change is measured by a microcontroller and processed by software, allowing the compound to be identified and quantified.

Before the volatile compounds reach the detector, they are separated by a short column, which contains an internal coating of a bonded liquid phase. Volatile compounds dissolved in this liquid phase can be made to exit the column at different times when heated to high temperatures. This separation is further enhanced by heating the column in a programmable temperature profile. The time one compound remains in the column is recorded as its retention time (RT), which is supposed to be unique for each specific component, while the area of each peak is considered a quantitative measurement of the quantity of the particular volatile (expressed in Counts).

The zNose employs headspace or bubbler techniques as its sampling modes. A side Luer needle was used as a sample odour injection tool, and a spark needle as the bubbler generator. A rotating valve was used to switch the machine from sampling configuration to an inject configuration. A trap was used as a preconcentrator to collect and hold volatile samples, and high grade helium was used as the sample carrier gas.

All samples in this experiment were tested using the following mode of operation: sampling through side Luer needle for 10 s, separating different compounds in the column for 14 s, acquiring a frequency signal every 0.02 s for 10 s, and baking the sensor for 30 s. The sensor detection temperature was set to

60°C. Column temperature was ramped from 40°C to 180°C at the rate of 10°C/s, while the sensor baking temperature was 150°C. The carrier gas flowed at a rate of 3.0 cm^3 s^{-1}. Between each measurement, at least one air blank was run, such that baseline peaks were all under 200 counts before resuming sample runs. All measurements were repeated twice and mean values are reported.

5.3.4 Subjective assessments of ripeness and rot

Ripeness development was assessed by viewing mango fruit's skin. A color index was recorded according to the following rating scale (Shorter and Joyce, 1998): 1, 100% green; 2, 75% green; 3, 50% green and 50% yellow; 4, 75% yellow; 5, 100% yellow. In this study, the color index was expressed in percentage to facilitate comparison with respiration rate and aroma signals in a numerical format.

Disease severity, the percentage of fruit surface covered by black lesions, was also visually observed and recorded (Kobiler *et al.*, 2001). Fruits were considered rotten when disease severity, usually caused by side and stem end rots, covered more than 1% of their surface area. The degree of rot was continuously monitored until 50%-70% of the surface area was damaged.

5.3.5 Objective color and total soluble solids (TSS) measurements

The skin color was also measured with a tristimulus colorimeter (Chroma Meter CR-300, Minolta Co. Ltd., Japan) in the L*, a*, b* colour space. Four evenly distributed places along the equator were selected and a mean value was used. Three values were obtained: "L" measures lightness and varies from 100 for perfectly reflective white to zero for perfectly absorptive black; "a" measures redness when positive, gray when zero, and greenness when negative; and "b" measures yellowness when positive, gray when zero, and blueness when negative.

Total soluble sugars (TSS) of 108 regular samples were measured with a handheld refractometer (ERMA, Japan) after RR, aroma, and color measurements. Four pieces of flesh were drawn from 10 mm below the locations of colorimeter measurements and juice hand-squeezed from them for TSS measurements. Mean TSS values were used for computation of a maturity index (I_m, %) as follows: $I_m = (TSS/8) \times 100$ (Jha *et al.*, 2007). A correlation analysis of colorimeter readings and I_m was attempted.

5.4 Results and Discussion

5.4.1 Preliminary aroma estimation

A total of ten obvious peaks were detected by zNose from unripe, ripe, and overripe mango samples. The aroma profiles of a representative sample (control sample 8) from 1 to 24 days are depicted in Fig. 5.1. All other samples had similar profiles with quantitative differences. As peaks a-c only appeared when the mango fruits were fully rotten and beyond the scope of this study, only peaks 1-7 will be discussed. By comparing their Kovats indices with the literature (Macleod and Troconis, 1982; Macleod and Pieris, 1984; Macleod and Snyder, 1985), the corresponding chemical compounds were tentatively identified (Table 5.1).

Table 5. 1 Peak 1-7 chemicals of mango fruits

$Peak_1$	**$Peak_2$**	**$Peak_3$**	**$Peak_4$**	**$Peak_5$**	**$Peak_6$**	**$Peak_7$**
Methyl butanoate	α-Pinene	3-Carene	Limonene	Hexyl butanoate	2-furfural	α-Terpinolene

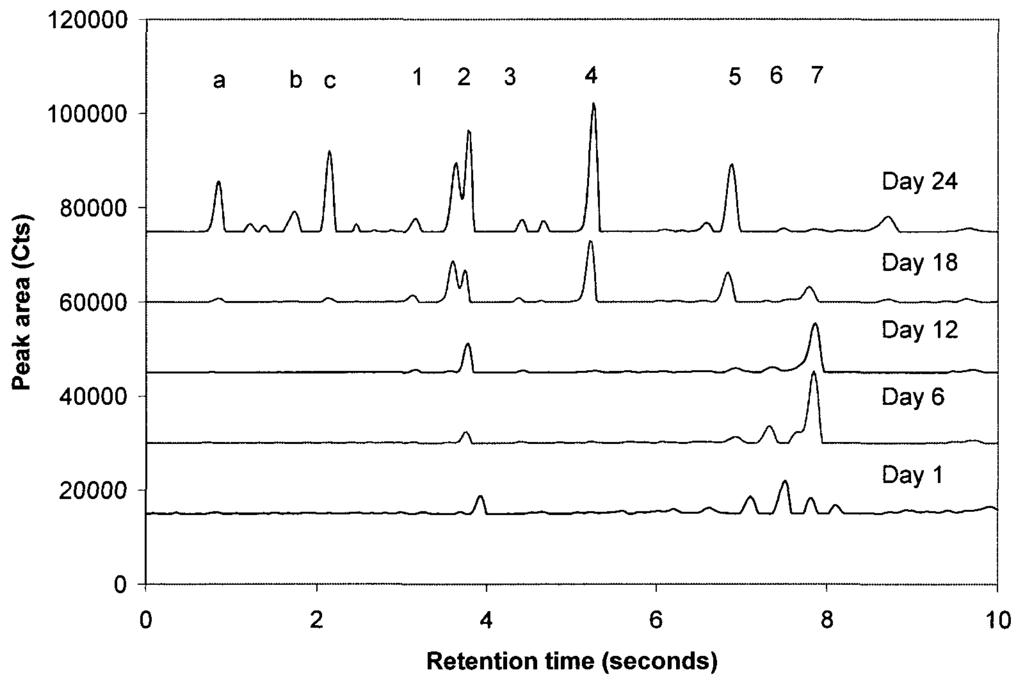


Fig. 5. 1 Aroma profiles of control sample 8

Peaks 1, 3, 4, and 5 increased with storage time, $Peak_2$ first decreased then increased, $Peak_6$ decreased all the way, $Peak_7$ increased first and then decreased. These variations in aroma compounds may relate to the biochemical changes in mango fruits and may represent different stages of mango ripeness and rot development. After comparing with RR and subjective assessments, Peaks 4, 5, and 7 were chosen as key parameters for ripeness evaluation and rot prediction. Fig. 5.2 shows the development of Peaks 4, 5, and 7 in control sample 8 at 24 days. The corresponding RR, observed rots percentage, and subjective color assessments are also shown in Fig. 5.2. To facilitate comparison with RR and observed rots percentage values, original aroma signals, i.e., peak areas were modified as: peak areas × 0.005.

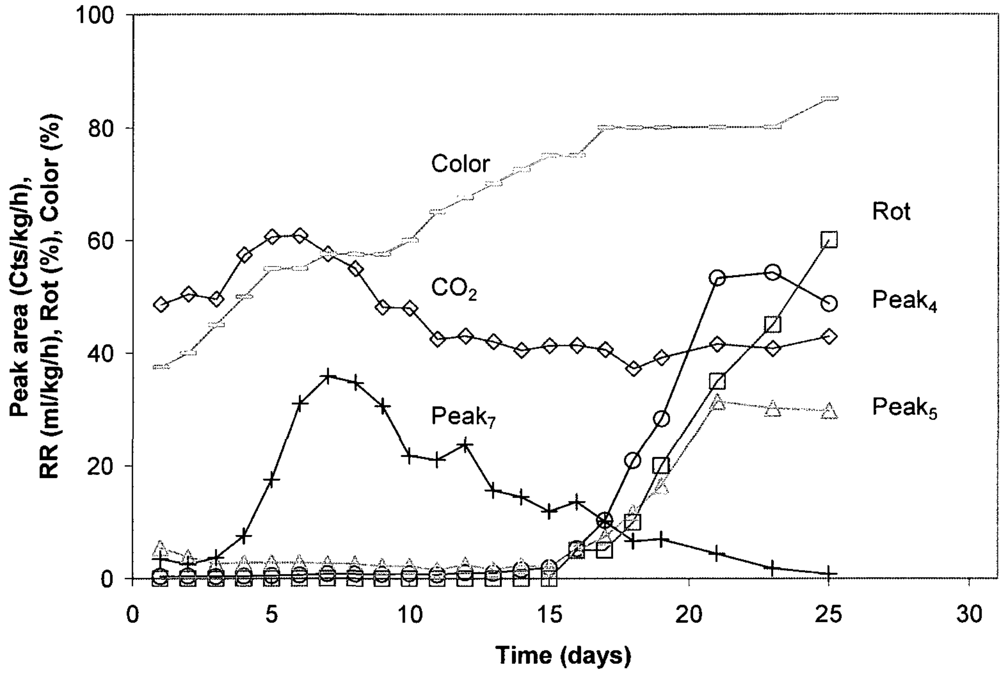
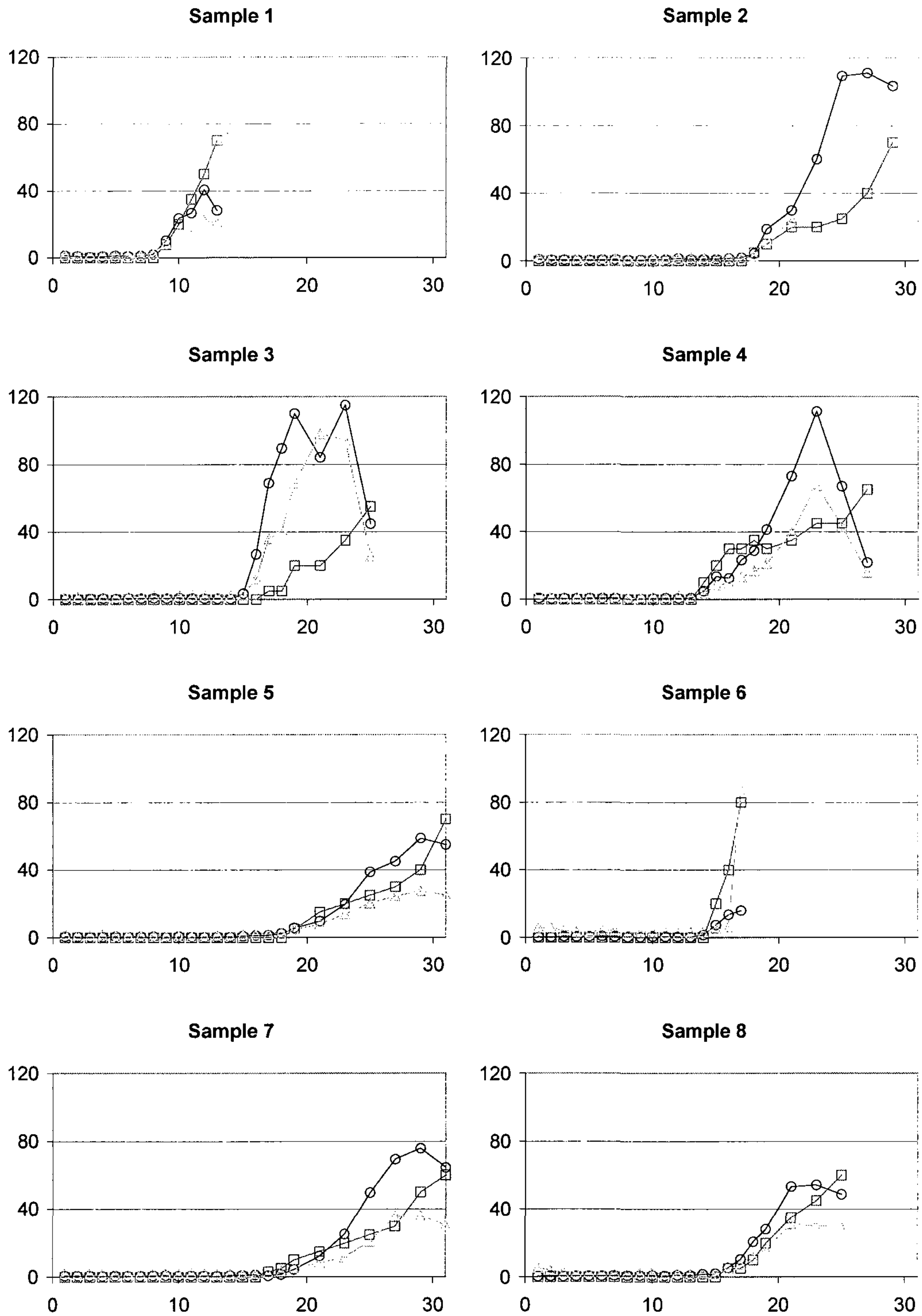


Fig. 5. 2 Peaks 4, 5, 7 and RR, rot, color assessments of control sample 8

By close observation of Fig. 5.2, one sees that Peaks 4 and 5 increased almost at the same time as did the degree of rot, while $Peak_7$ was tightly related to RR and subjective color assessment. These trends imply that $Peak_4$ and $Peak_5$ may be related to mangoes' rotting status, while $Peak_7$ may be related to mangoes' ripeness development. Hence, the prediction of rot and ripeness with aroma signals was attempted.

5.4.2 Rot prediction

The potential of predicting rot occurrence with $Peak_4$ and $Peak_5$ was tried first. The curves of $Peak_4$, $Peak_5$, and observed rotting of the 12 control samples are presented in Fig. 5.3. For samples 1, 5, 6, 7, and 10, $Peak_4$ and $Peak_5$ increased along with the increase in observed rot, while for samples 2, 3, 8, 9, 11, 12, the appearance of $Peak_4$ and $Peak_5$ preceded observed rot. Only the aroma signals of sample 4 appeared 1 day later than observed rots. Hence, the occurrence of rot in mango fruits can possibly be predicted with $Peak_4$ and $Peak_5$.


Sample 1
Sample 2
Sample 3
Sample 4
Sample 5
Sample 6
Sample 7
Sample 8
120
80
40
0
0
10
20
30

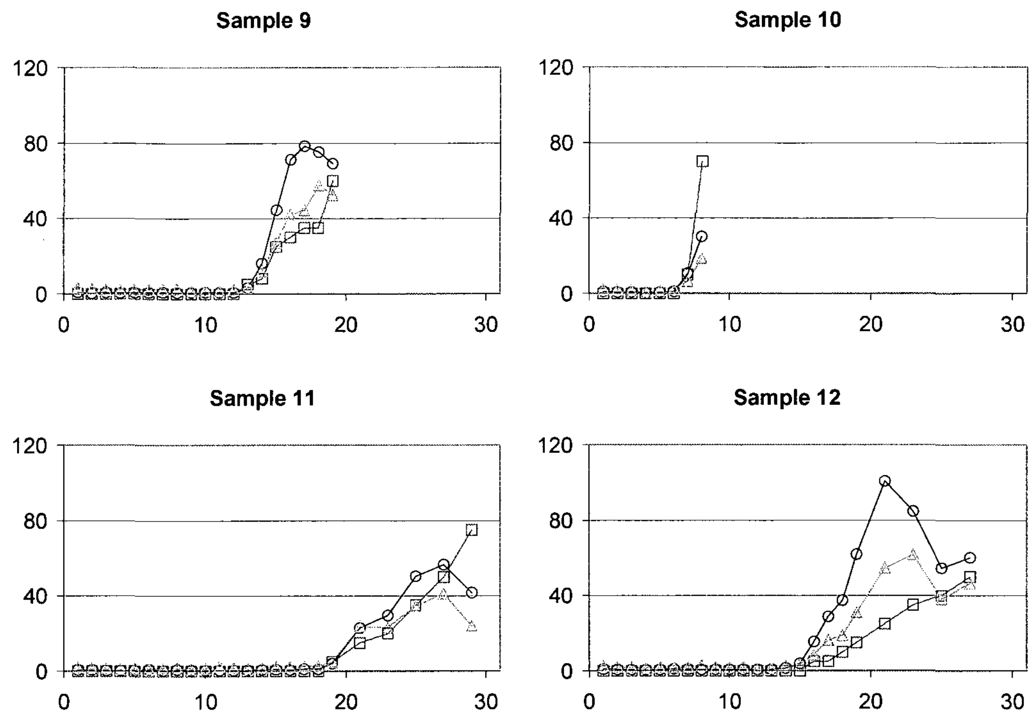


Fig. 5. 3 Rot prediction with $Peak_4$ and $Peak_5$ in 12 control samples
[□, Observed rots (%); ○, $Peak_4$ (Cts/kg/h); Δ, $Peak_5$ (Cts/kg/h)]
[X axis: time (days)]

To analyze rot prediction with $Peak_4$ from a statistical standpoint, two simple thresholds were selected: mango fruits were considered rotten when $\geq 1\%$ of their surface area was covered by lesions (Kobiler *et al.*, 2001); meanwhile, a threshold value of 3 (counts/g) was chosen for $Peak_4$, representing an optimum prediction criterion. If a mango's $Peak_4$ value exceeded 3 and the corresponding rot occupied an area >1%, the sample was considered correctly classified as a rotten mango. On the other hand, if a sample mango's $Peak_4$ value was <3 and the corresponding rot, if any, was <1%, it was correctly classified as non-rotten mango. Otherwise ($Peak_4$ >3, rot <1% or $Peak_4$ < 3, rot >1%) the classification is incorrect.

Rot predictions for 245 volatile measurements on 12 control samples over 31 days using these two threshold criteria are shown in Fig. 5.4. Misclassified non-rotten mangoes accounted for 2 of 167 and misclassified rotten mangoes accounted for 3 of 78 measurements. In total, 240 of 245 measurements were correctly classified equalling a percent correct of 98%.

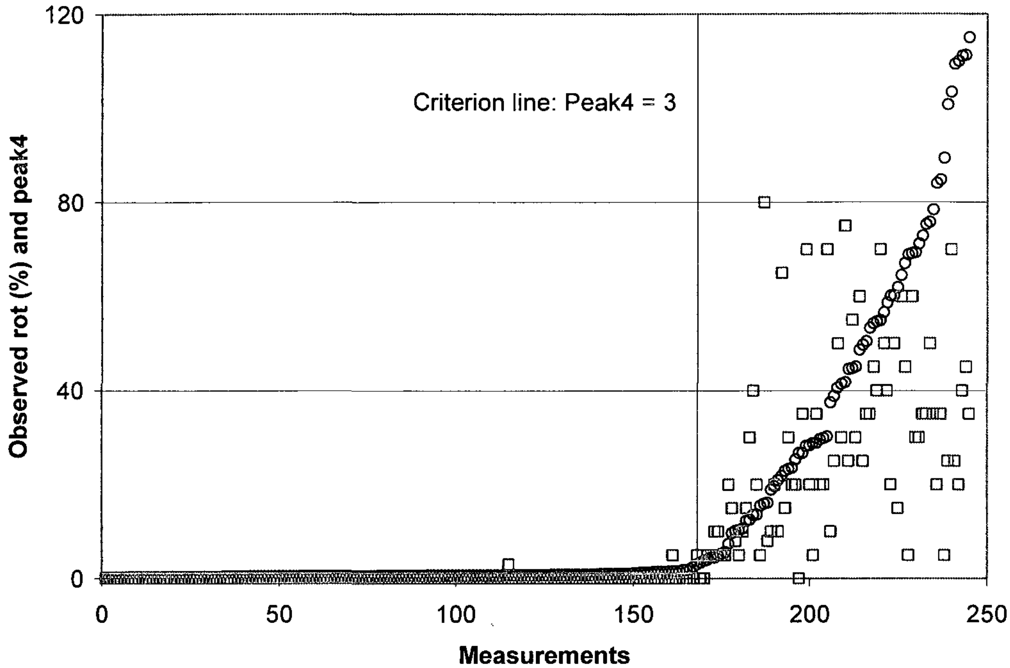


Fig. 5. 4 Rot predictions for 12 control samples using Peak$_4$
(○: Peak$_4$; □: observed rot)

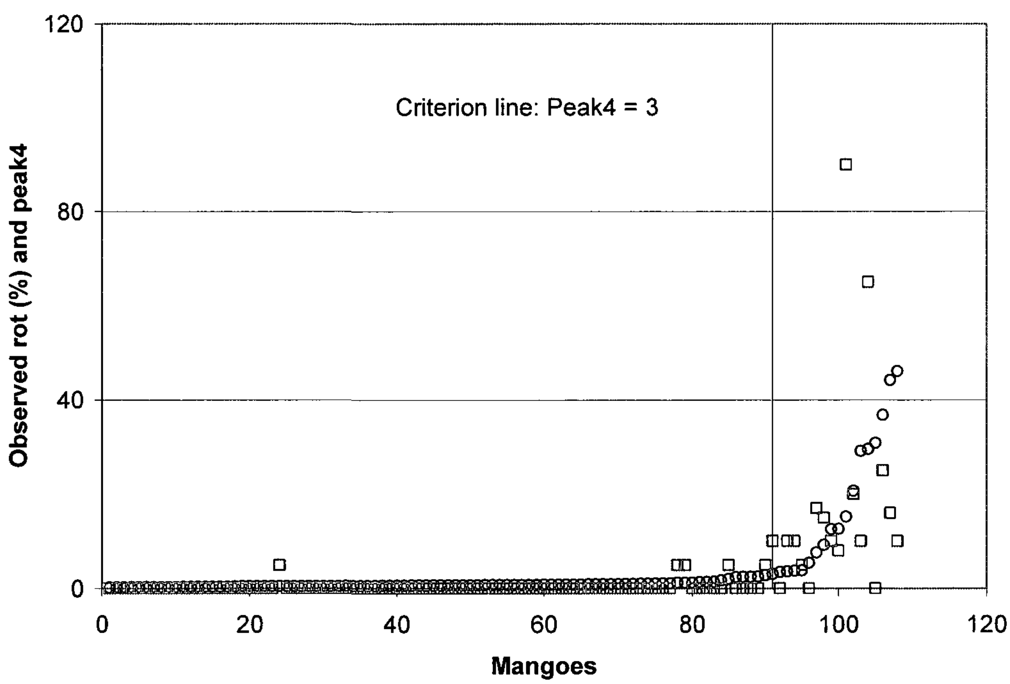


Fig. 5. 5 Rot predictions for 108 regular samples, based on Peak$_4$
(○: Peak$_4$; □: observed rots)

When 108 regular samples were classified as to the presence or absence of a threshold of 1% rot by area, using $Peak_4$ levels, 5 rotten mangoes in 90 samples were misclassified as non-rotten and 3 non-rotten mangoes in 18 samples were misclassified as rotten, for a 93% accuracy (Fig. 5.5).

A similar analysis was conducted for rots predicted with a threshold value of 5 for $Peak_5$. In 245 measurements of the 12 control samples collected over 31 days, non-rotten mangoes were misclassified 8 of 172 times, while rotten mangoes were misclassified 4 of 73 times. Overall, 12 of 245 measurements were misclassified, corresponding to a 95% accuracy (Fig. 5.6).

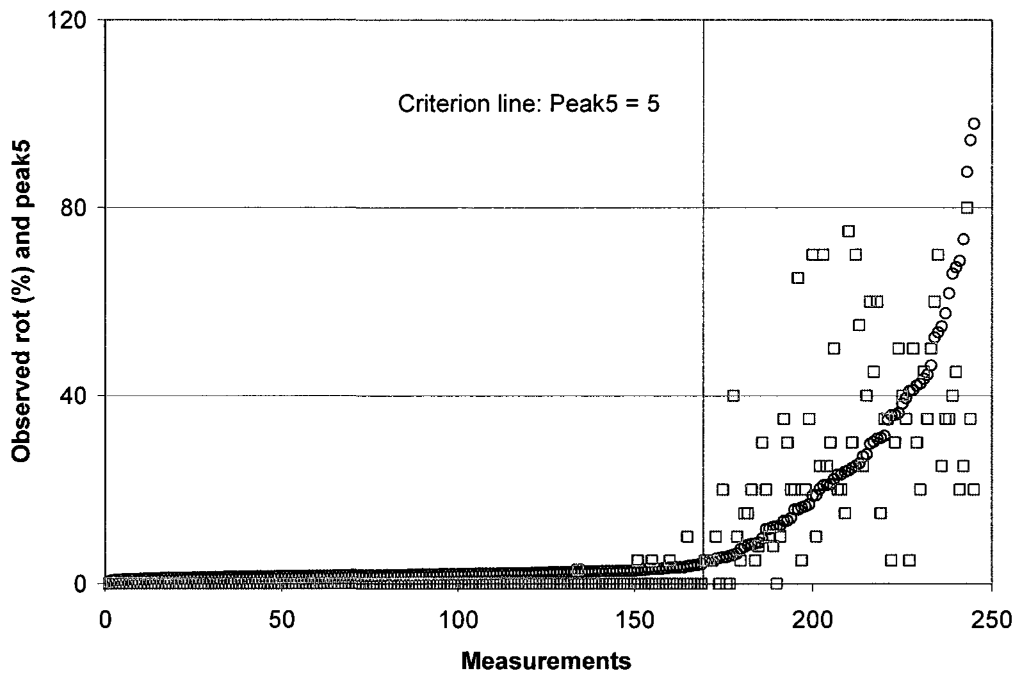


Fig. 5. 6 Rot prediction for 245 measurements of 12 control samples using $Peak_5$ (○: $Peak_5$; □: observed rot)

A similar attempt of rot prediction in 108 regular samples using $Peak_5$ showed a misclassification of non-rotten mangoes in 10 of 96 cases, and the misclassification of rotten mangoes in 3 of 12 samples. In total, 13 in 108 samples were misclassified corresponding to an 88% accuracy (Fig. 5.7).

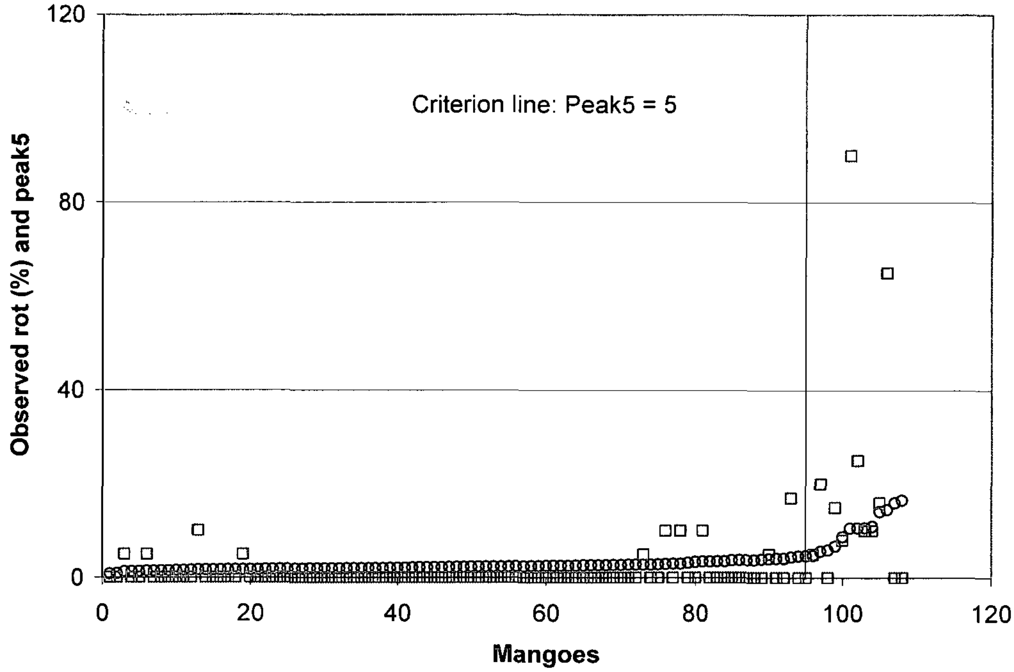


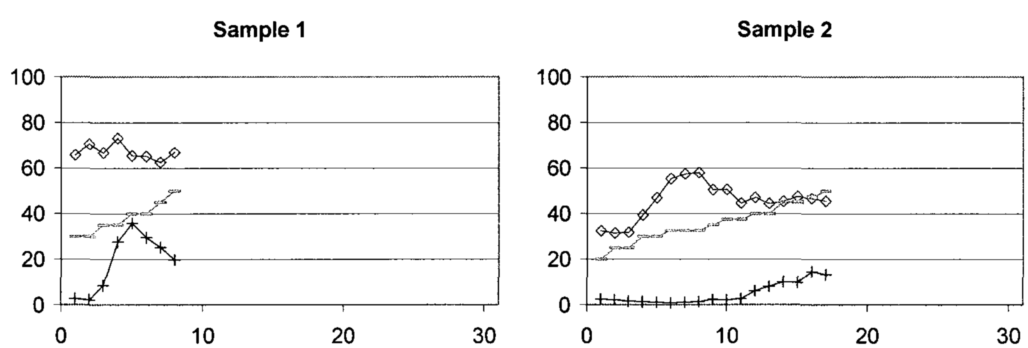
Fig. 5. 7 Rot prediction for 108 regular samples using $Peak_5$
(○: $Peak_5$; □: observed rot)

Similar results were reported by Moalemiyan *et al.* (2007) for mango *cv.* Keitt. Using GC/MS to analyze volatile organic compounds, they found 1-Pantenol to be symptomatic of stem-end rot and thujol of anthracnose infection. Although volatile compounds are cultivar dependent, similar observations in these two studies confirmed that the prediction of mango rot by way of volatile organic compounds is a promising method for mango industry. However, their observations were not consistent for all replicates, whereas in this study, a high accuracy of rot prediction was achieved. Moreover, GC/MS usually requires hours of operation for volatiles measurements, whereas the zNose only requires 1-2 minutes.

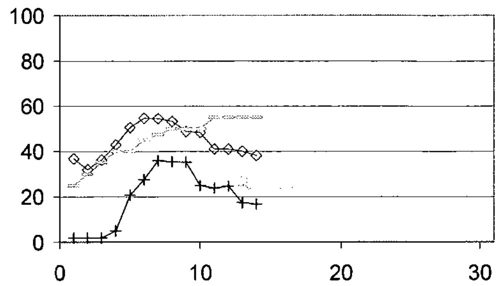
5.4.3 Ripeness evaluation

The possibility of evaluating mango ripeness with $Peak_7$ was also evaluated. To avoid disturbance by rot aroma signals, only measurements from fruit without rot symptoms were used, i.e., any fruit-measurement where observed rot exceeded 1%, or $Peak_4$ exceeded 3, or $Peak_5$ value exceeded 5 was removed, leaving a total of 164 fruit-measurements. The respiration rate, subjective color assessments, and $Peak_7$ values of the 12 control samples' 164 measurements are presented in Fig. 5.8.

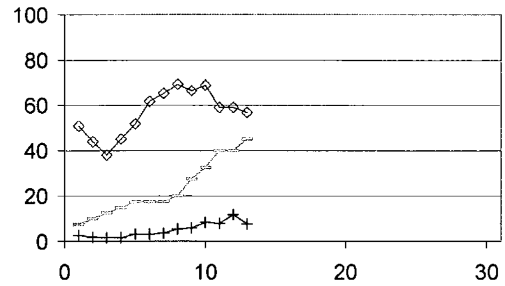
Increases in RR of control samples were all followed by increases in $Peak_7$, sometimes soon, sometimes later. As the climacteric rise is a characteristic phenomenon of the ripening process (Biale *et al.*, 1953), RR and $Peak_7$ might be used as parameters for ripeness evaluation. However, RR values varied widely among different samples, although for a single mango, the RR has a climacteric peak. For example, RR of sample 1 was between 60 and 80 $mL{\cdot}kg^{-1}h^{-1}$, but the RR of sample 11 was between 20 and 40 $mL{\cdot}kg^{-1}h^{-1}$. The lowest value of sample 1 was larger than the highest value of sample 11. Hence, RR can not be used as a general rule to judge mango ripeness. By contrast, $Peak_7$ value increased from near zero to higher values and has the potential to be used for estimation of mango ripeness. To estimate the potential of evaluating mango ripeness with $Peak_7$, the subjective color assessment was employed as the ripeness reference (Biale *et al.*, 1953). A sample was considered ripe when 50% of its skin was yellow. The TSS was not adopted for reasons which will be outlined in 5.4.4.



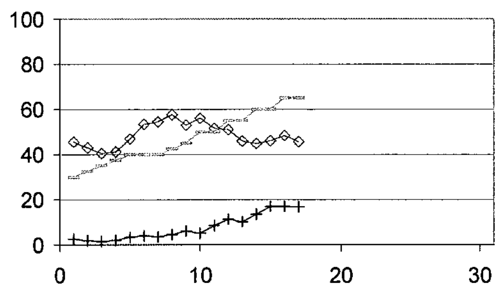
Sample 3



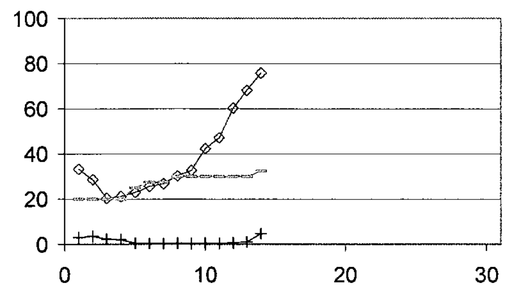
Sample 4



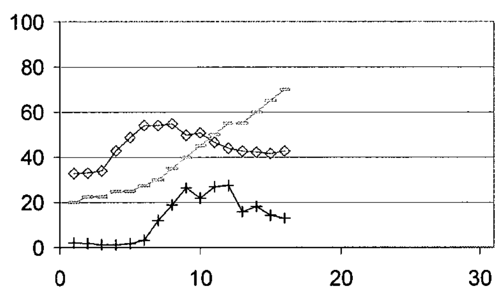
Sample 5



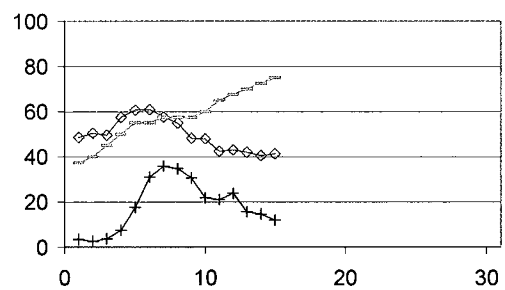
Sample 6



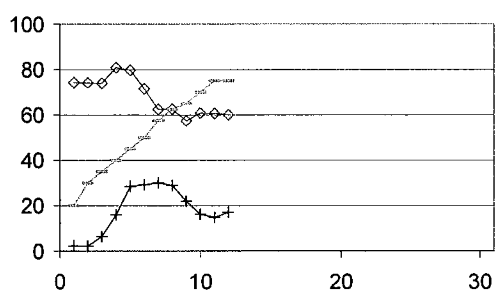
Sample 7



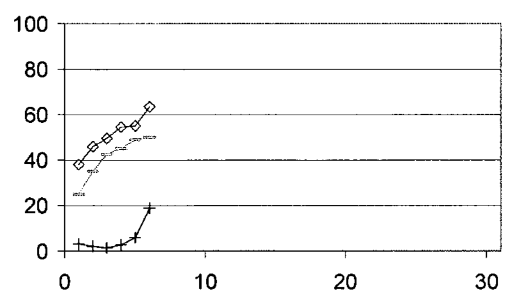
Sample 8



Sample 9



Sample 10



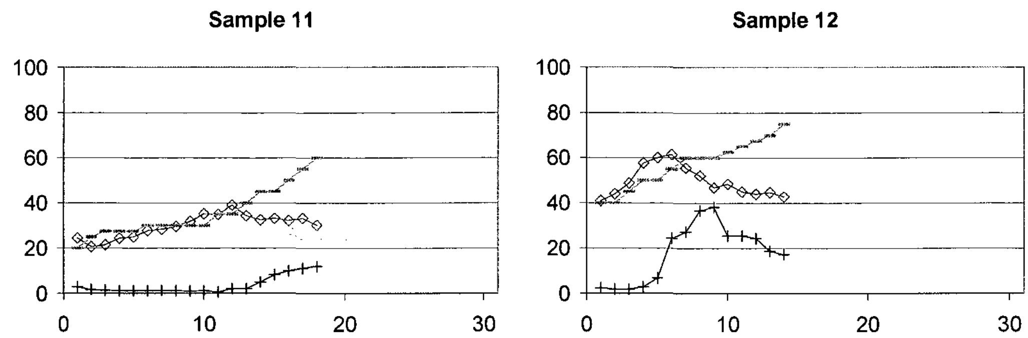


Fig. 5. 8 Ripeness prediction of 12 control samples using $Peak_7$
[- : observed colour (%); ◊: RR (ml/kg/h); +: $Peak_7$ (Cts/kg/h)]
[X axis: time (days)]

After an overview of the 164 fruit-measurements, a $Peak_7$ value of 12 was selected as the criterion: when $Peak_7$ was over 12 (counts/g), the mangoes had mostly begun to ripen. A statistical assessment of prediction accuracy for the 164 measurements with regard to a subjective color criterion of >50% yellow colour development as a sign of ripeness, showed unripe mangoes to be misclassified in 5 of 104 cases, and ripe mangoes misclassified in 11 of 60 cases, resulting in an overall accuracy of 90% (Figure 5.9).

A similar analysis of 82 rot-free regular samples for ripeness evaluation using $Peak_7$ showed 9 of 44 unripe mangoes were misclassified, as were 7 of 38 ripe mangoes, resulting in an overall accuracy of 80% (Figure 5.10).

Lalel *et al.* (2003) reported similar observations for 'Kensington Pride' mangoes. Respiration rate (CO_2 production) reached a peak of 50.75 $mL.kg^{-1}h^{-1}$ on the fourth day of ripening and the ripe skin color occupied 75% of the total fruit surface area by the seventh day, thereafter color change slowed down. With SPME-GC-FID and SPME-GC-MS techniques, Lalel *et al.* (2003) found that the major monoterpenes (*α-Terpinolene*) increased in the first 3 or 4 days and decreased thereafter when the fruit became overripe. This behaviour is very similar to that observed for $Peak_7$ in the current study.

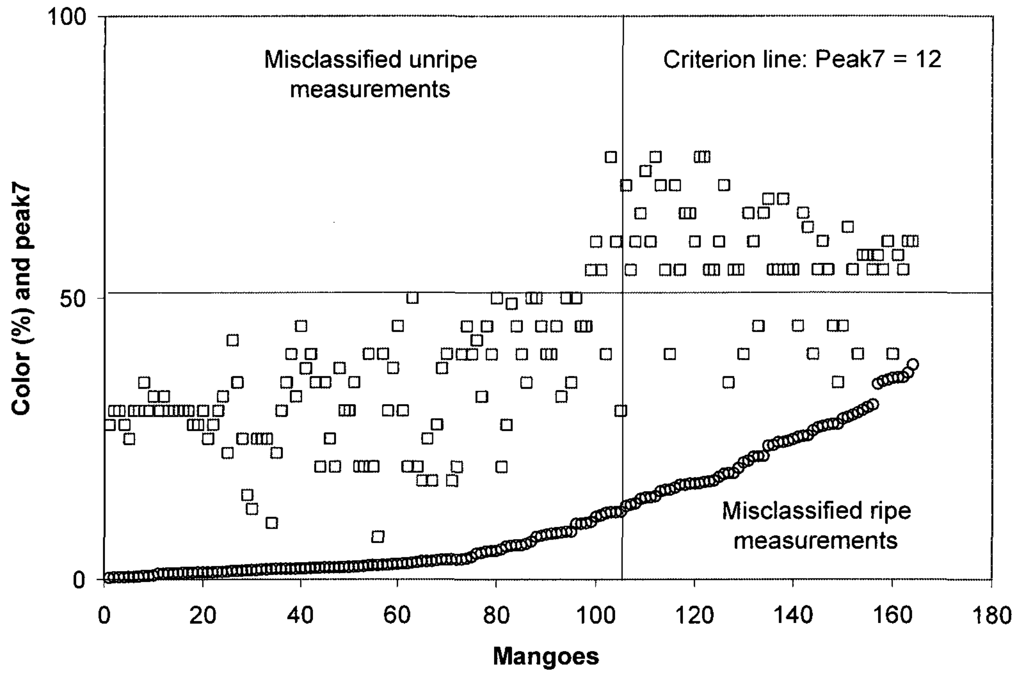


Fig. 5. 9 Ripeness evaluation of 164 rot-free measurements of 12 control samples using Peak$_7$
(○: Peak$_7$; □: color)

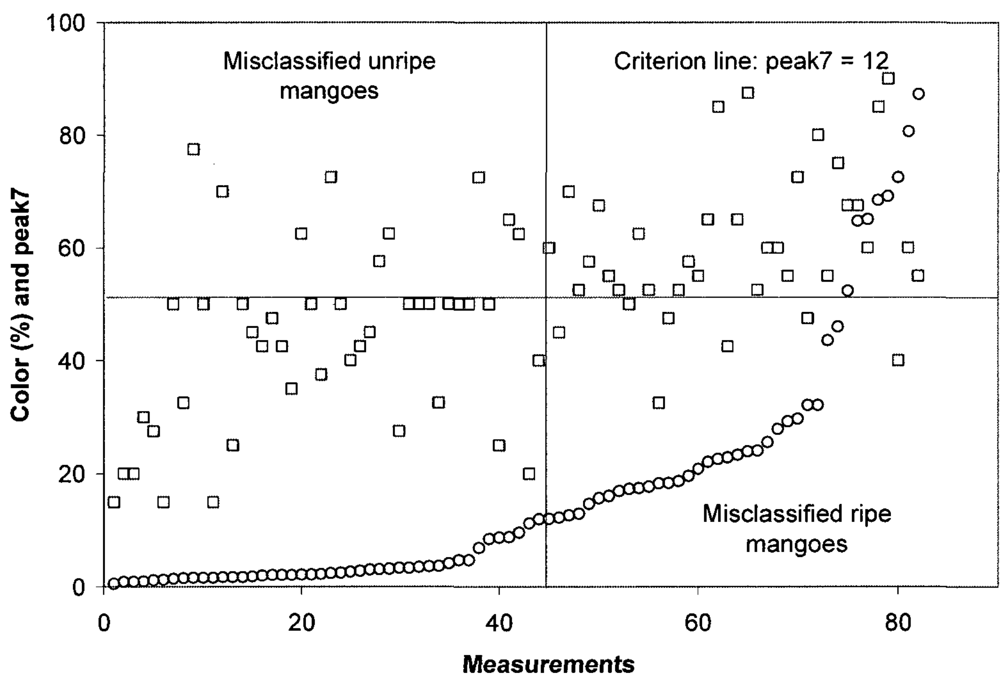


Fig. 5. 10 Ripeness evaluation of 82 rot free regular samples using Peak$_7$
(○: Peak$_7$; □: color)

5.4.4 TSS and objective color

The relationship between TSS and objective color values was investigated for the 82 regular rot-free samples (Fig. 5.11). The "a", "b", and "a*b" values were used in a multiple linear regression model (Jha *et al.*, 2007) to predict TSS contents. Unfortunately, an $R^2 = 0.38$ ($P < 0.0001$) resulted, indicating that TSS cannot be predicted with objective color values. The reason might be that a full development of TSS occurred during 1-2 week transportation from Mexico to Canada, since measured TSS of all 108 regular samples exceeded 8 Brix°. Hence, TSS is not a good index for ripeness evaluation during shelf life. This inversely proved that the evaluation of mango aroma is the only viable method for ripeness estimation.

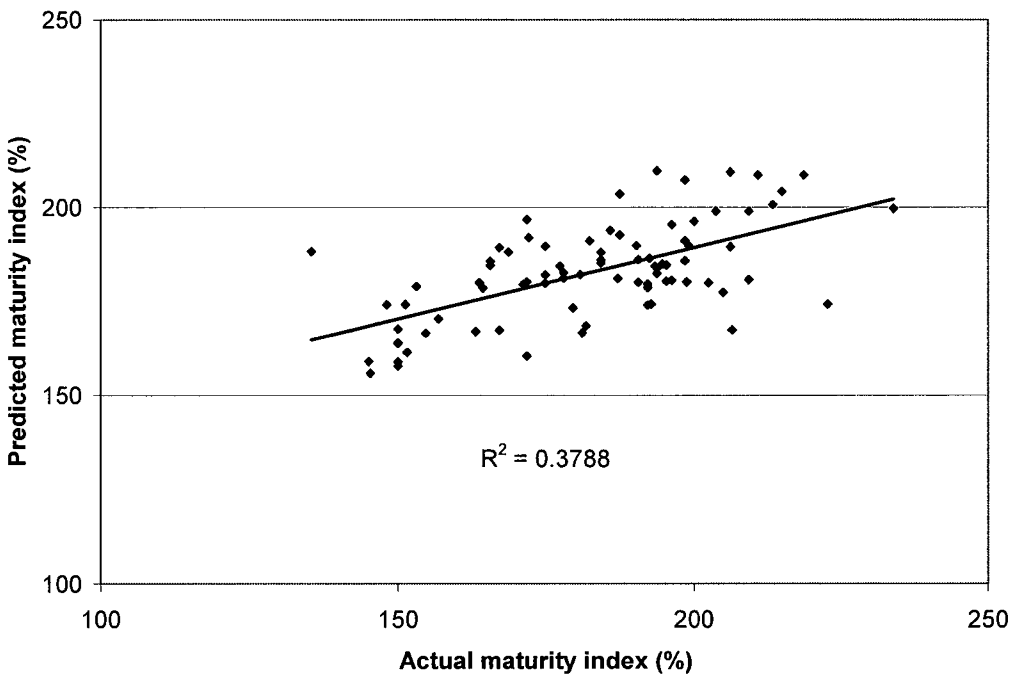


Fig. 5. 11 Relationship of TSS-predicted to actual maturity index

5.5 Conclusions

Aroma signals detected with zNose are useful parameters for mango quality evaluation. Seven marked peaks were identified and three were used in rot prediction and ripeness evaluation. Results showed $Peak_4$ (*limonene*) was the best candidate for rot prediction with a 93% rots prediction accuracy. $Peak_5$ (*Hexyl butanoate*) also achieved a rot prediction accuracy of 88%. Evaluation of ripeness was attempted using $Peak_7$ (*α-terpinolene*). Using subjective color observations as a reference, 80% ripeness evaluation accuracy was achieved. However, the correlation between measured TSS and its predicted counterpart based on colorimetric measurements was poor and cannot be used for ripeness evaluation during shelf life.

The rot prediction methods developed have the potential to be applied in the mango industry for detecting side and stem end rots during mango shelf life in an ultra rapid manner (less than 1 minute). The ripeness evaluation method developed can be used to judge the best consumable period of mango fruits.

5.6 Acknowledgements

The authors are grateful to the NSERC (Natural Sciences and Engineering Research Council of Canada) for financial support.

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CONNECTING STATEMENT 3

Passive (static) aroma detections were conducted in Chapter IV and V. The results revealed that aroma can be effectively measured in an ultra fast way with the zNose technique. This constitutes the basis for design of the work in the following chapters.

To investigate aroma variation during the microwave drying process, a real time aroma detection system was developed. The temperature of food samples during drying was controlled on the basis of variations in aroma components, by automatically adjusting the microwave power level. Different control strategies were tested to achieve the best drying effect.

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Design of a Real Time Aroma Monitoring and Control System for Microwave Drying

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Contributions made by different authors are as follows:

The first author, the PhD student did the experimental work and prepared the manuscript; the second and third authors are the supervisors who guided the overall research work; the fourth author provided guidance during the experimental process.

CHAPTER VI

DESIGN OF A REAL TIME AROMA MONITORING AND CONTROL SYSTEM FOR MICROWAVE DRYING

6.1 Abstract

The purpose of this project was to design a real time aroma monitoring system to control microwave drying processes. Aroma profiles were measured with an ultra fast gas chromatograph (zNose^{TM}, EST) and expressed as retention time and peak area. These signals were then analyzed using a fuzzy logic algorithm to determine adequate drying temperatures to maintain product quality. Phase control was used to adjust the microwave power level. Carrot and apple samples were used to test the system. The system was shown to have the capacity to successfully fulfill the desired control functions and thereby lead to better retention of aromatic compounds in dried materials.

Keywords: Design, aroma, microwave, fuzzy control, electronic nose

6.2 Introduction

The alteration of food aroma during food processing is of great concern to the food industry. Unexpected aroma loss, change, distortion, or even destruction often happen and lead to unacceptable quality of final products. How to preserve the preferred aroma and limit the development of unacceptable aromas is a crucial task for food engineers.

The first step in fulfilling this task is the accurate and rapid detection of food aroma during processing. Unfortunately, most current instruments, either electronic noses or conventional analytical chemistry equipment, are all time-consuming and complex (Li *et al.,* 2006a), which make it almost impossible to detect food aroma in real time and online. Consequently, no literature is so far available regarding online detection of food aromas, and only a few offline aroma measurements have been reported (Kompany and Rene, 1993; Krokida and Philippopoulos, 2006; Samuelsson *et al.*, 2006).

In recent years, a new aroma detection instrument, the zNose^{TM} (7100 Fast GC Analyzer, Electronic Sensor Technology, New Bury Park, CA, USA) has become commercially available. This new instrument is capable of detecting food aroma as fast as in seconds, which makes online detection of food aroma possible for the first time. However, so far, applications of the zNose have remained limited to offline detection (Lammertyn *et al.*, 2004; Gan *et al.*, 2004), with no online employment reported as yet.

The new thermal processing technique of microwave drying, developed in recent years, has the advantages of a higher drying rate, lower energy consumption, and better quality of the dried products (Gerard and Roberts, 2004; Mullin, 1995; Orsat *et al.*, 2006; Raghavan *et al.,* 2005; Sanga et al., 2000; Vadivambal and Jayas, 2007). In the applications of this technique, microwave power and drying temperature applied to the foods were proven to have significant influence on the quality of final products. To optimize microwave drying, researchers have studied various microwave power profiles, including intermittent methods and continuous methods (Venkatachalapathy and Raghavan, 2000; Cheng *et al.*, 2006). Feedback combined with phase control of microwave power achieved a precise and effective control of the drying process (Li *et al.*, 2006b).

Despite numerous attempts at power and temperature control, no aroma evaluation during microwave drying has hitherto been reported. How aroma varies and how product quality can be improved in terms of aroma is unknown. Moreover, the problem of non-uniformity of microwave heating which may result in partial and interior burning of the products remains a critical challenge to microwave researchers.

Fuzzy logic control, which was first introduced by Mamdani (Mamdani, 1974) and is based on Zadeh's fuzzy sets theory (Zadeh, 1965), is a precise, quick, and effective control strategy. It is usually applied in processes where a mathematical model is deficient or complicated, or if the process is nonlinear or probabilistic. As aroma is a nonlinear parameter and there is no available description to directly relate it with human senses, it is better expressed in linguistic variables. Hence, fuzzy logic control is a good choice in the control of drying progress to improve dried food aroma quality.

The objectives of this study were:

1) To design an aroma monitoring and control system for microwave drying of agricultural products;
2) To control the aroma by varying drying conditions with fuzzy logic controllers;
3) To test the system with carrot and apple samples.

6.3 Materials and Methods

6.3.1 Hardware design

An aroma monitoring and control system was designed for microwave drying (Fig. 6.1). The system consists of an aroma detection unit, a microwave drying system, a temperature and power control system, and a PC-based data acquisition unit (DAQ). The aroma detection unit included a zNose, a two-position pneumatic valve, an air flow installation, a moisture condenser, and a flow meter; the microwave drying system included a domestic microwave oven, a Teflon container, and an electrical scale; the temperature and power control system included an optical fiber sensor, a control circuit, a 12 VDC and an 110 VAC power supplies; the data acquisition unit was a PCI 6014 multifunction device (National Instruments, TX, USA) with analog outputs to facilitate MW power control.

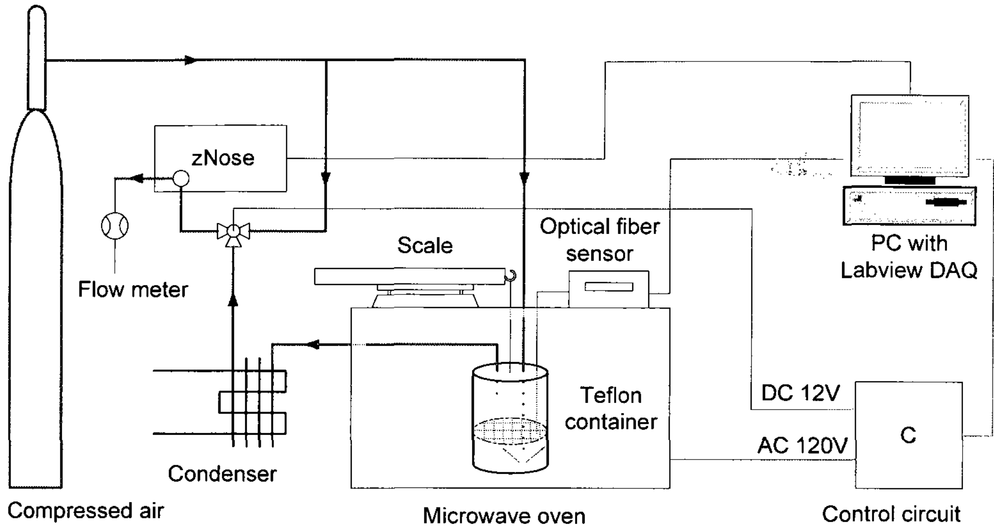

Fig. 6. 1 Schematic diagram of the aroma monitoring and control system

6.3.1.1 Aroma detection unit

A zNose was used for volatile compounds detection in this study. It is actually a miniature, high-speed gas chromatograph (GC) containing a detector, a short separation column, and support electronics.

The detector of the zNose is an uncoated, high quality surface acoustic wave (SAW) crystal. The crystal operates by maintaining high frequency acoustic waves on its surface. The targeted compounds land and stick on the detector and change its frequency. The frequency change is measured with a microcontroller and processed by software, allowing the compounds to be identified and quantified.

Before the volatile compounds reach the detector, they are separated by a short column, which contains an internal coating of a bonded liquid phase. Volatile compounds dissolve in this liquid phase first and then exit at different times when the column is heated. The heating of the column is program-controlled and hence separation is enhanced. The time a compound is retained in the column is recorded as its retention time (RT) and is supposed to be unique for each specific component. The summed frequency change of this compound is expressed as an area of a peak and is considered as a quantitative measurement of its amount.

The zNose employs headspace as its sampling mode. A side Luer needle is used as a sample odour injection tool and a rotating valve is used to switch the machine from a sampling configuration to an injection configuration. A trap is used as pre-concentrator to collect and hold volatile samples, and high grade helium is used as the sample carrier gas.

By calibrating with standard chemicals (n-Alkanes), zNose can convert detected peaks to a Kovats index (Appendix C). Hence, chemical compounds can be identified by checking a library of related volatile compounds.

All aroma measurements in this experiment were conducted in the following operation mode: sampling through side Luer needle for 6-10 s (for different samples), separating different compounds in column for 14 s, acquiring data every 0.02 s for 20 s, and baking the sensor for 30 s. The sensor detection temperature was set at 60°C, column temperature was ramped from 40°C to 180°C at a rate of 10°C/s, sensor baking temperature was 150°C, and the carrier gas flow rate was 3.0 $cm^3\ s^{-1}$.

The zNose can automatically repeat its measurement at a preset time interval. In this study, this interval was set to 2 minutes. A two-position pneumatic valve (M223W1DFR-HT-312, Teqcom, CA, USA) was installed in front of the zNose to alternate clean air and food vapour, so as to clean the zNose and to detect the aroma, respectively. Both cleaning and detection cycles lasted 2 minutes; hence the aroma was measured every 4 minutes.

Compressed air was used to carry the vapour out of the Teflon container and bring it to the zNose. Air pressure was adjusted to 6.9 kPa (10 psi) with a regulator and the flow rate was controlled at 1000 mL min^{-1} with an adjustable flow meter. Moisture was collected in an air-cooled condenser just after the microwave oven and before the zNose. Hence, only dry volatiles were allowed to reach the zNose. All tubes and connectors were made of Teflon so as to not retain aromas and facilitate microwave drying.

6.3.1.2 Microwave drying system

A 600 W domestic microwave oven (Beaumark 02314, Matsushita Electric Ind. Co. Ltd., Yamatokoriyama, Japan) was utilized for food drying in this study. The original control panel was by-passed to facilitate the new power control strategy. The stirrer and fan were modified to be controlled separately and were fully on during the whole drying process. The power supply to the magnetron was phase controlled with the DAQ and control circuit.

Food samples were dried in the Teflon container, which was equipped with a small porous basket to hold samples and allow air flow through it. The mass of the entire container with samples was measured online with an electronic scale (PK 4801, Denver Instrument, Denver, CO, USA). An optical fiber sensor (Nortech Fibronic Inc., Quebec Canada) was inserted in the center of one of the samples for temperature measurements.

6.3.1.3 Temperature and power control

Aromas emanating from food samples during drying are not a factor that can be controlled directly. However, they are influenced by moisture content, air flow rate, drying temperature, etc. In this study, all parameters but temperature were kept as stable as possible. The control of aromas was hence fulfilled by varying drying temperatures. Preliminary experiments showed that aroma signals, i.e., peak areas increased dramatically when the temperature was increased and vice versa. This phenomenon suggested a built-in aroma-temperature relationship where aroma control could be implemented by means of temperature control. This relationship will be investigated in the following sections.

To control the temperature of food samples during microwave drying, one possible means is to control the output power of the magnetron. However, magnetrons work at high voltages (2000-4000 V) and it is not safe to control this high voltage directly. Instead, a control of the primary side of the transformer which supplies the high voltage to the magnetron is a possible choice.

Current power controls applied in MW drying are either intermittent (on/off) or continuous. In this study, these two methods were combined with a phase controller and a continuously automatic power control was implemented. The concept is that a portion of the sine wave is cut and the rest remains. By adjusting the duty cycle, power adjustment is implemented. The advantage of this method is that it is possible to control the power with a DAQ and the energy consumption is minimized.

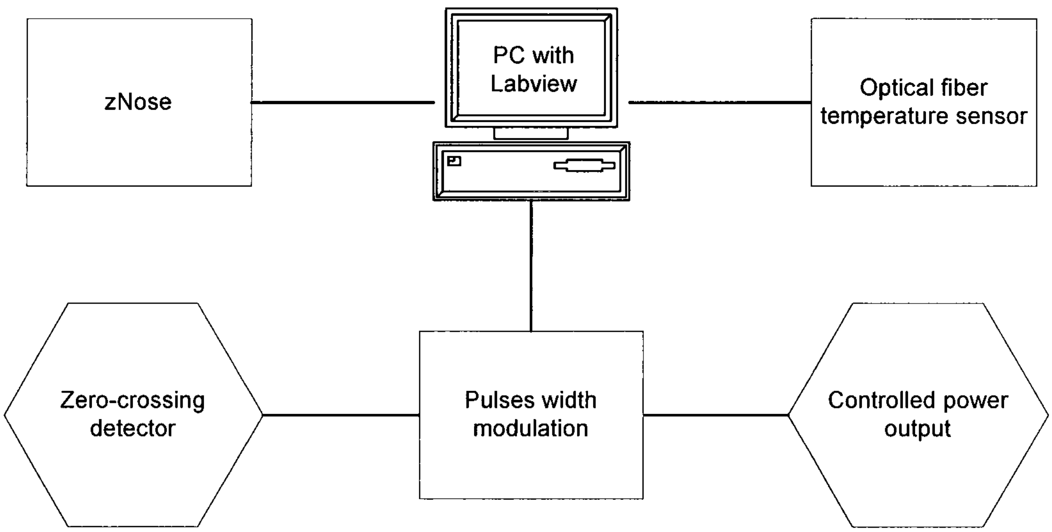

Fig. 6. 2 Diagram of temperature and power control

A diagram of the temperature and power control system is shown in Fig. 6.2 and further details can be found in the Appendix D. The first part of the system is a zero-crossing circuit, which detects the zero points of the sine wave and generates trigger pulses with an opto-isolator (H11L1). The pulses are input to a LM555 and generate output pulses with adjustable duty cycles. The duty cycles of these pulses are dependent on temperature requirement and are adjusted with an analog voltage (1-4 V) output from the DAQ. The output pulses from LM555 further drives a Triac circuit through another opto-isolator (MOS3012) and hence the power is controlled. By both continuous adjustment and on-off control of the MW power, the temperature control was delicately fulfilled. The opto-isolators protect the DAQ and PC from any possible destructive high voltage.

6.3.1.4 Data acquisition unit

The PC utilized in this study housed the Labview program (Labview 7.1, National Instruments, TX, USA) and had a Multifunction Data Acquisition device built-in (PCI 6014, National Instruments, TX, USA). The PCI 6014 is a 16-bit resolution DAQ board with 250 kS/s rate and includes 2 analog outputs, 8 digital I/O lines, and two 24-bit counters. Three ports of the DAQ were used in this study: an analog input for the optical fiber temperature sensor, a DC output to control the two-position pneumatic valve, and an analog output to adjust AC power to the magnetron. All the controls were implemented in the Labview environment.

The PC can also read signals from zNose with the RS-232 port, and process the data with software called MicroSense 4.0 (Electronic Sensor Technology, CA, USA), expressing aroma as retention times (RT) and peak areas. Unfortunately, these processed data cannot be accessed and only the original frequency signal can be read. Hence, a program of calculating peak area and retention time had to be developed separately for aroma monitoring and control.

6.3.2 Software design

The system software includes a program for calculation of aroma peak area and retention time, a program for temperature and power control, and a program for fuzzy logic control. All programs were implemented in the Labview environment.

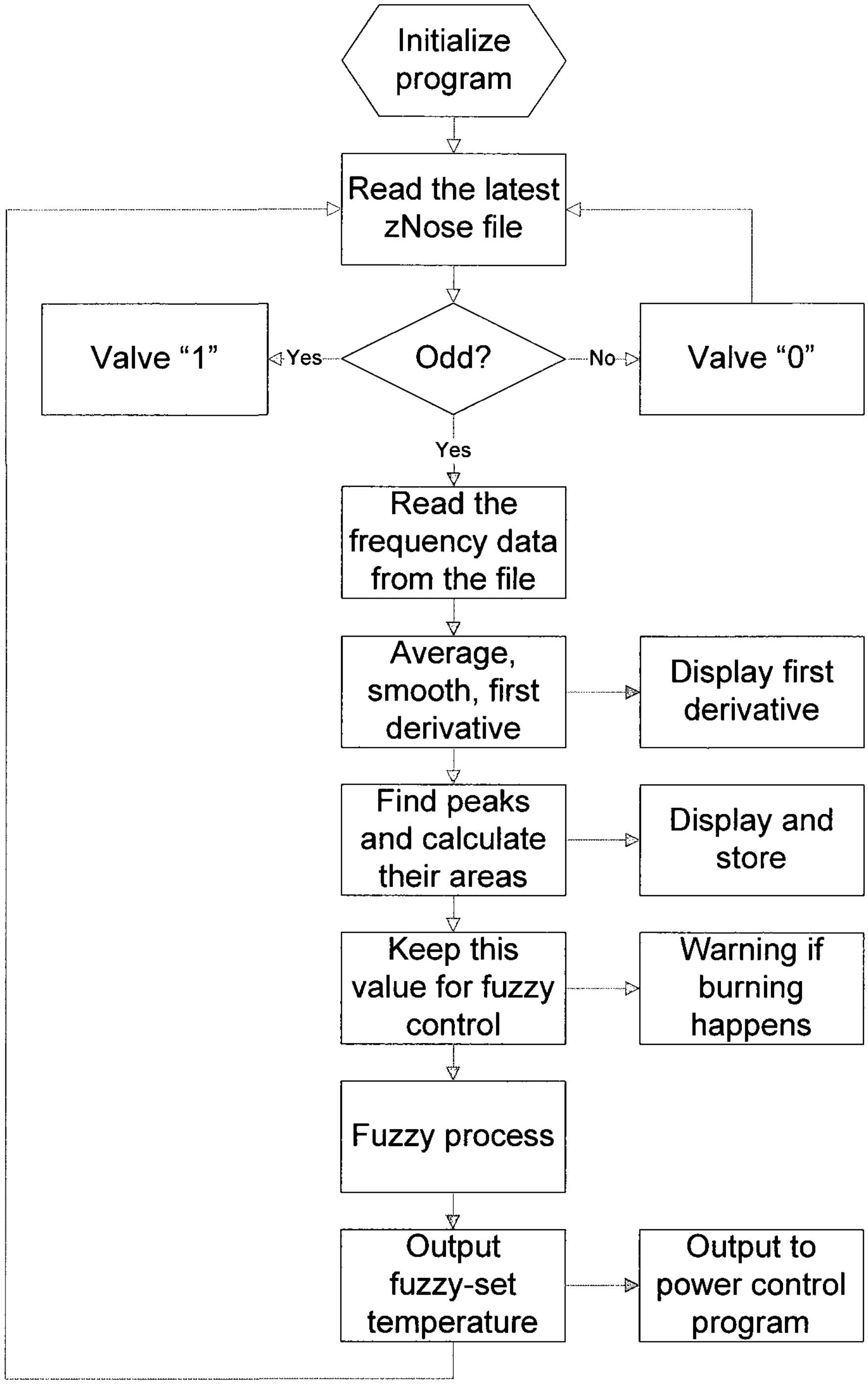

Fig. 6. 3 Calculation of peak area and retention time with Labview

6.3.2.1 Aroma signal processing

A program for calculation of peak areas and retention times was designed. The program first smoothed the original frequency signals, calculated the first derivative, retained its positive portions (as MicroSense), and smoothed them again. The obvious peaks in each specific part of the profile were then identified and their areas calculated. All these steps minimize the possible influence of noise. A flowchart of the program is shown in Fig. 6.3. The detailed Labview program can be found in Appendix E.

6.3.2.2 Temperature and power control

A power and temperature control program was designed and the flowchart is shown in Fig. 6.4. The detailed program can be found in Appendix D. In this program, temperature was measured with the optical fiber sensor and 1-5V analog signal was input to DAQ. After comparing it with a preset temperature, a difference, Δt, was calculated. If the difference is less than "0", the control voltage is "3.4V" and the output power is near "0W". If the difference is larger than "0", the control voltage (hence the output power) will be a value proportional to the temperature difference according the following equation:

$$V = F - \frac{PT - AT}{PT - RT} \times (F - MV) \tag{6.1}$$

where

AT is the actual temperature (°C),

F is the control voltage corresponding to the final drying power (V),

MV is the minimum control voltage corresponding to maximum power (V),

PT is the preset temperature (°C),

RT is room temperature (°C), and

V is the control voltage (V).

What this equation represents is that:

1) When $AT = RT$ (the beginning of the drying process when the food temperature is the same as the room temperature), $V = MV$ (the output power is maximum);
2) When $AT = PT$ (the preset temperature is reached), $V = F$ (the output power is a stable value F when the preset temperature is reached)
3) When $RT < AT < PT$ (actual temperature is between room temperature and the preset temperature), V will vary between MV and F.

F and MV values are dependent on the type of food and were carefully adjusted to ensure that the power was just enough to fulfill the temperature requirements and not too much to overheat the magnetron.

Thus the power was controlled both by "on-off" and continuous control methods, and temperature control was within a feedback loop. This combination greatly improved the drying in terms of temperature control and energy consumption.

To achieve precise power control, the relationship between the control voltage and the output power of the magnetron must be investigated. Different control voltages were applied to heat 500 g water in a 1000ml beaker for 1 minute to calibrate the microwave power levels before experiment. The results were reported and the relationship was used in power control.

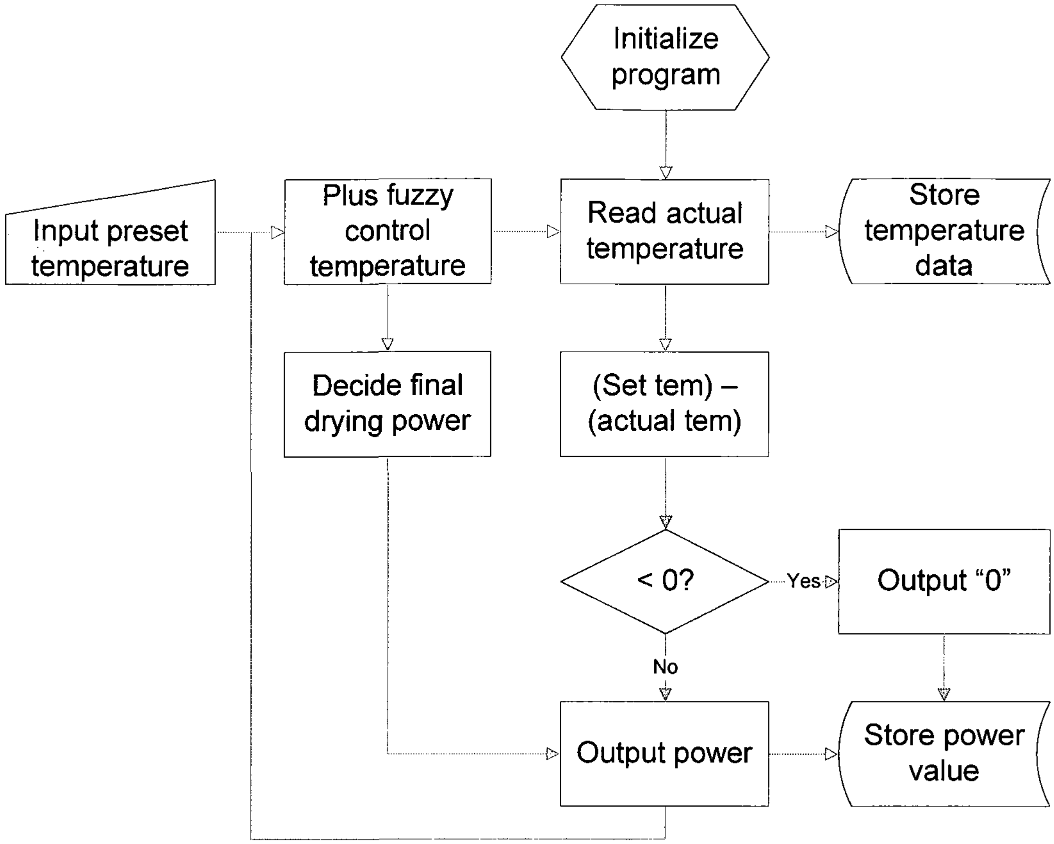

Fig. 6. 4 Flowchart of the temperature and power control program

6.3.2.3 Fuzzy logic control

As aroma is a nonlinear function with respect to other parameters and the relation between these peak values and human senses is unknown, it is difficult to express it with a traditional mathematical model. In this case, a fuzzy logic algorithm is a good choice, as it can vaguely describe the aroma in linguistic variables such as light, medium, or strong. This algorithm also simplifies the control process by reducing complex aroma signals to several linguistic terms. The benefits of the fuzzy logic control are more obvious when more than one variable are considered, such as aroma peaks and burning peaks (where products were charred) used in this study.

After investigating the relationship of aroma variations with respect to drying temperatures, two typical aroma peaks were chosen as input variables to the fuzzy logic controller, one to represent normal aroma and one for burning aroma. The output of the fuzzy logic controller was a temperature within a reasonable range dependent upon the type of product. The purpose of the fuzzy logic controller was to retain normal aroma and prevent occurrences of burning as much as possible. Restricting input variables to two reduces the complexity of the fuzzy controller and also makes the fuzzy rule practical and clear.

Fig. 6.5 shows the triangle function of an aroma variable. All the input aroma variables had a similar function but with different ranges and intervals. Fig. 6.6 represents the singleton temperature output. Again different values were carefully selected for different product types. This temperature plus a preset of 70°C forms the fuzzy set temperature. Fuzzy inference used Mamdani's Max – Min method. The defuzzification method was center of gravity. Table 6.1 shows the rule base of the fuzzy logic controller. The fuzzy controller was implemented in the Labview environment to facilitate integral control in combination with power and temperature control.

Table 6. 1 General fuzzy rule base

Burn \ **Aroma**	**Low**	**Middle**	**High**	**Very high**
Low	P	Z	N+	N+
Middle	N	N	N+	N+
High	N+	N+	N+	N+
Very high	N+	N+	N+	N+

* P: positive, Z: zero, N: negative, N+: strongly negative

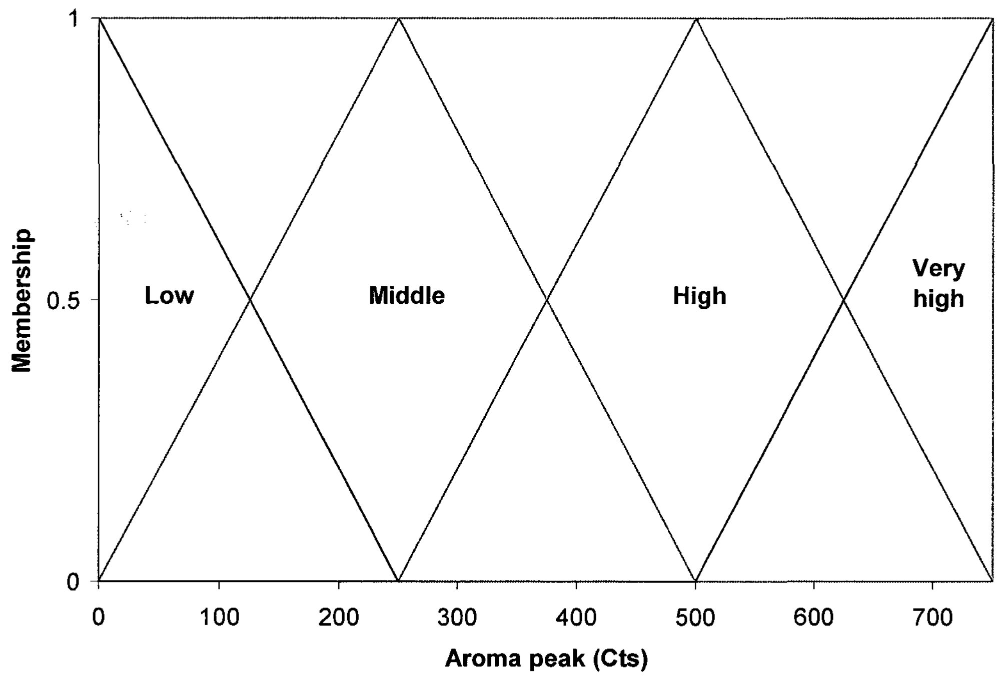

Fig. 6. 5 Fuzzification of one of the aroma functions

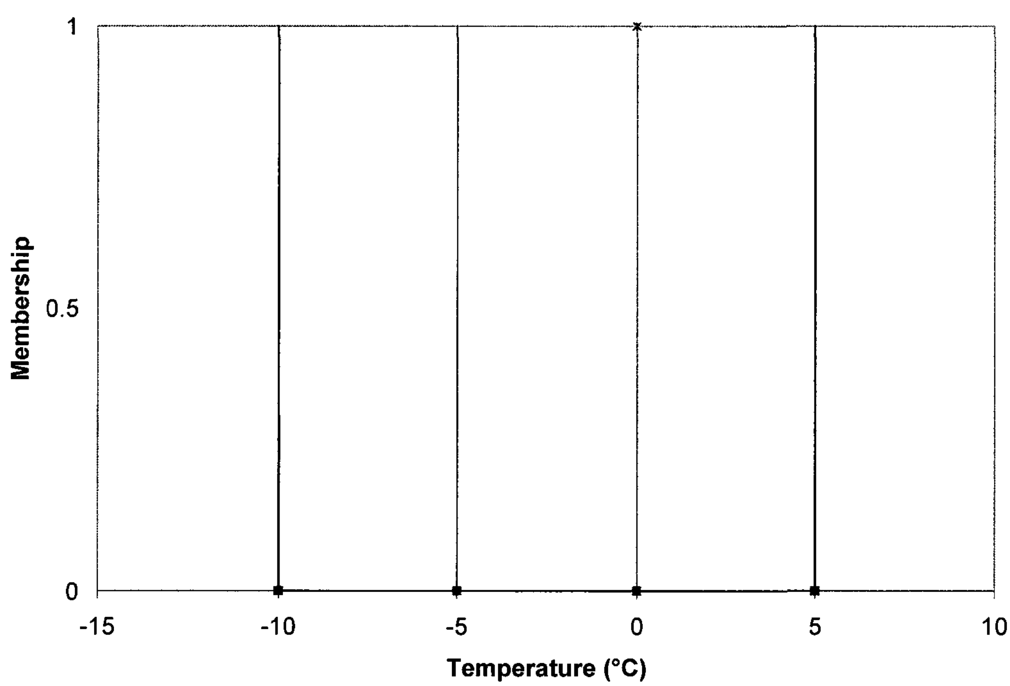

Fig. 6. 6 Singleton output temperature

6.3.3 System test

The developed hardware and software were tested first and the system was then tested by drying carrot and apple as representatives of vegetables and fruits, respectively. Carrot has a rich aroma complement and apple is susceptible to burning because of its high sugar content. Both experiments represented a challenge to the system in terms of aroma detection and control capacity.

6.3.3.1 Hardware and software tests

The software for aroma signal processing and hardware for power control were tested and reported, as they are the basis of the whole system. The rest parts were integrally tested in carrot and apple drying.

6.3.3.2 Carrot drying

Carrots of an unknown cultivar were purchased from a local market and stored at 4°C. Initial moisture content was roughly 88%. Peeled carrot samples in $10 \times 10 \times 10$ mm cubes (25±1g) were used in this test. All samples were dried to 10% moisture content (wet basis). Both fixed temperature drying and fuzzy controlled drying were tested.

6.3.3.3 Apple drying

The system was further tested with apple cubes. Granny Smith apples were purchased from a local market and stored at 4°C. Initial moisture content was 85.17%. Peeled apple cubes of $10\times10\times10$ mm (20±1g) were blanched in 80°C water for 1 minute before drying to prevent color changes and enzymatic reaction (Nieto *et al.*, 1998). All samples were dried to 10% moisture content (wet basis). Both fixed temperature drying and fuzzy controlled drying were tested.

6.4 Results and Discussion

6.4.1 Hardware and software tests

6.4.1.1 Aroma signal processing

The program developed in Labview to calculate aroma peak area and retention time was tested and its accuracy was compared with the commercial MicroSense 4.0 program. Fig. 6.7 shows the first derivative profile of n-Alkanes standard solution and Fig. 6.8 shows its positive portions. A comparison of these two methods for calculation of retention time and peak areas is shown in Table 6.2. The difference of retention times was within 0.05 second and the difference in peak areas was within 10%, indicating a sufficient accuracy for aroma monitoring and fuzzy logic control.

Table 6. 2 Retention time (RT) and peak area (PA) comparison for data processing by MicroSense (Mic.) *vs.* Labview

Peak	Mic. RT	Labview RT	RT diff. (sec.)	Mic. PA	Labview PA	PA diff. (%)
1	0.78	0.76	-0.02	685	716.13	+4.54%
2	1.58	1.56	-0.04	447	482.91	+8.03%
3	2.2	2.18	-0.02	1267	1348.01	+6.39%
4	2.94	2.92	-0.02	2658	2471.74	-7.01%
5	3.74	3.71	-0.03	4400	4430.85	+0.70%
6	4.54	4.52	-0.02	4224	4582.52	+8.49%
7	5.38	5.36	-0.02	5700	5274.74	-7.46%
8	6.24	6.22	-0.02	11223	11094.2	-1.15%
9	7.1	7.07	-0.03	7230	7509.14	+3.80%

* "+": a higher Labview calculated value; "-": a lower Labview calculated value

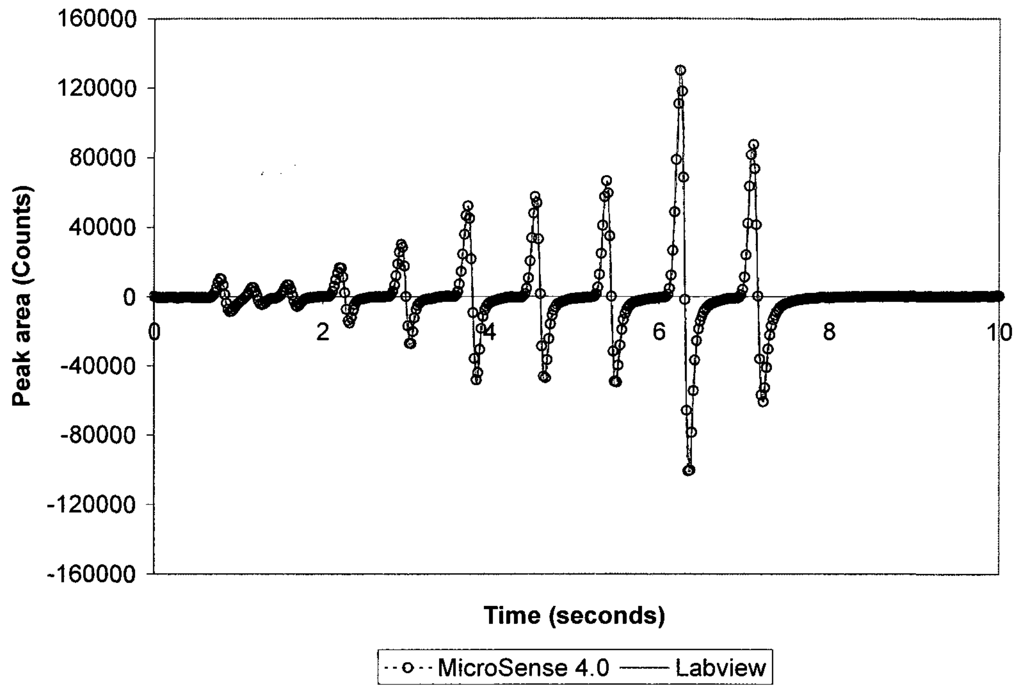

Fig. 6. 7 The first derivative profile of n-Alkanes

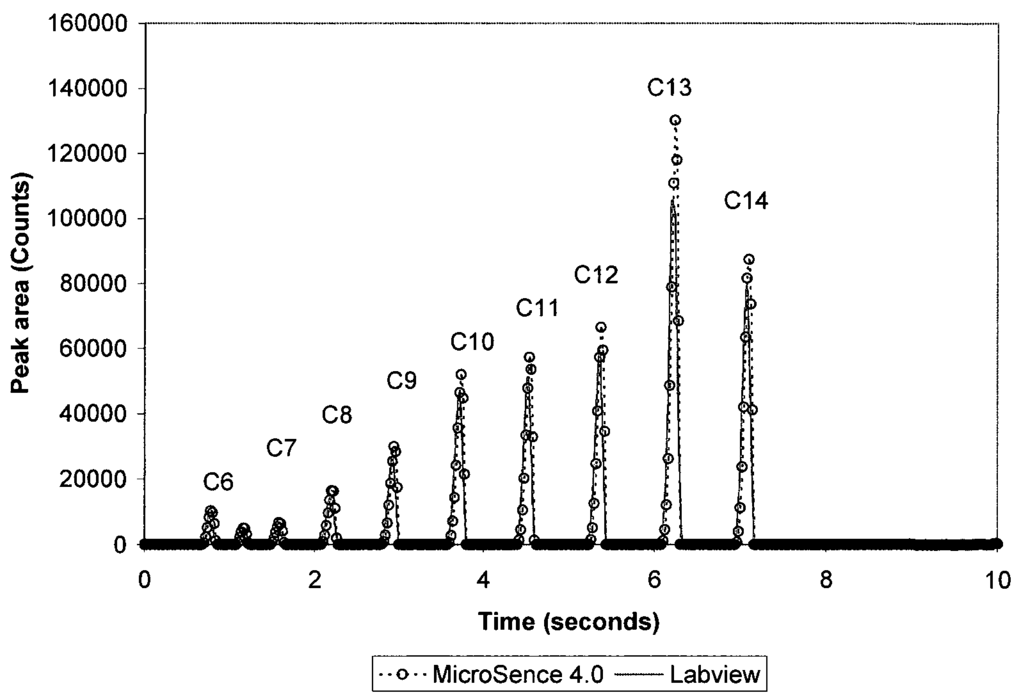

Fig. 6. 8 Positive portion of the first derivative after smoothing

6.4.1.2 Power control

The trigger pulses and LM555 generated pulses were monitored with an oscilloscope (Fig. 6.9) and the circuit was tested with a resistive load (a lamp) and a conductive load (microwave oven) (Fig. 6.10).

The calibration result of microwave power levels is shown in Fig. 6.11. By close observation one can find the nearly linear parts in the middle of this curve. By increasing the control voltage from 1.7V to 2.5V the output power of the magnetron decreased almost linearly from 475W to 15W with a value of R^2 = 0.99. Hence, this portion of the power relationship was used in the microwave power control experiments. Another point worth emphasizing here is that the "power-off" status was to be implemented with a control voltage of "3.4V", which corresponds to a near "0W" power output. This arrangement avoids the vibration which occurs in the magnetron when a complete power shutdown is applied and an immediate pulse is again generated to bring the power back.

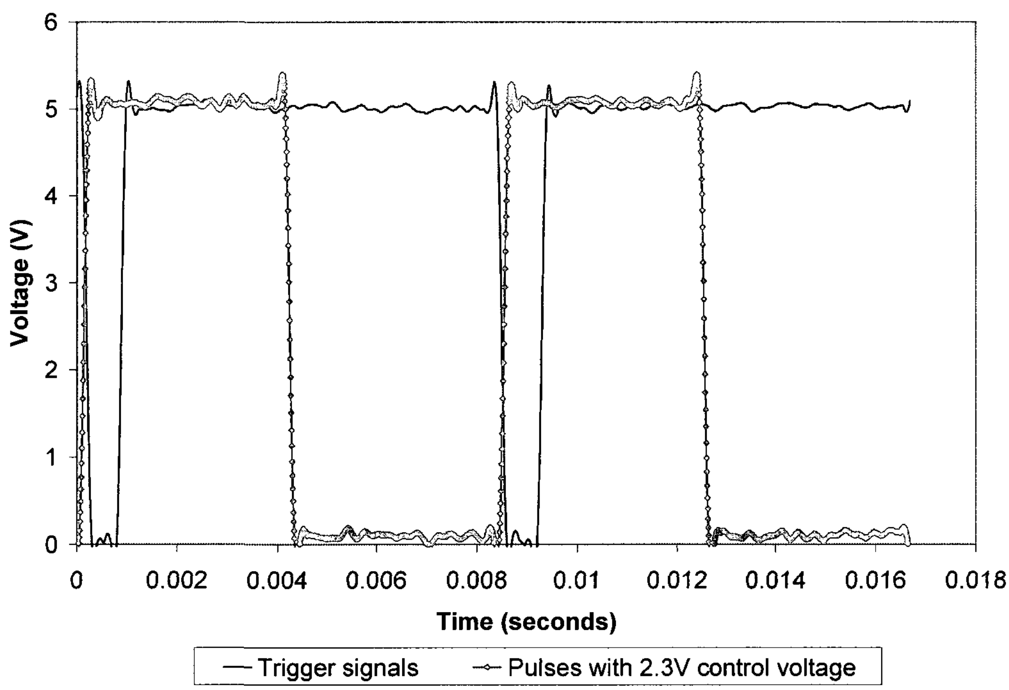

Fig. 6. 9 Trigger signals and generated pulses

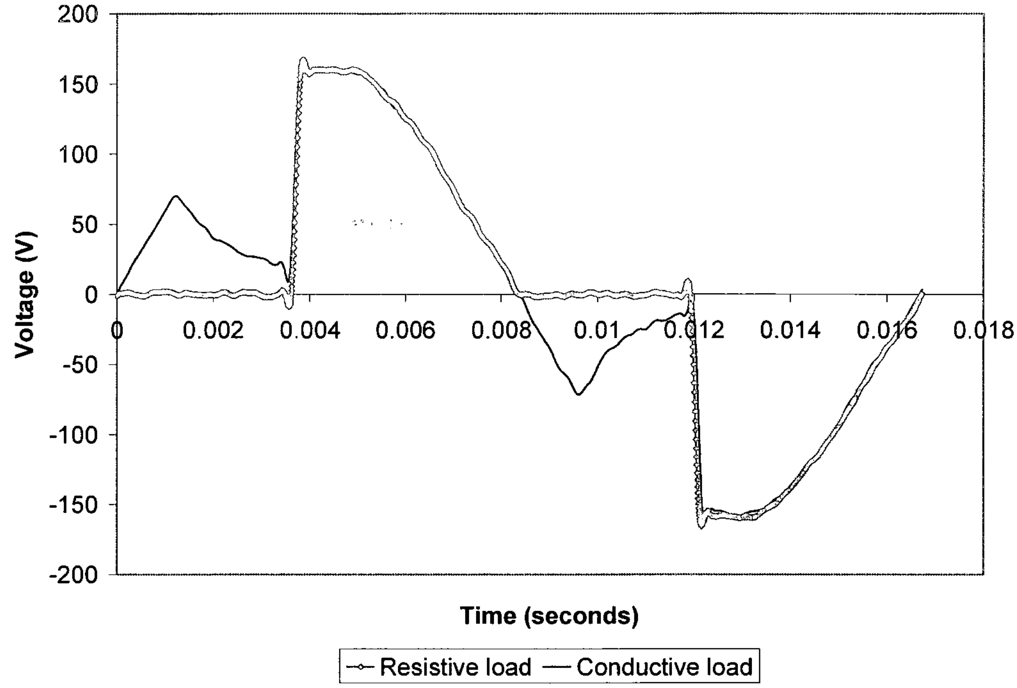

Fig. 6. 10 Phase control tests

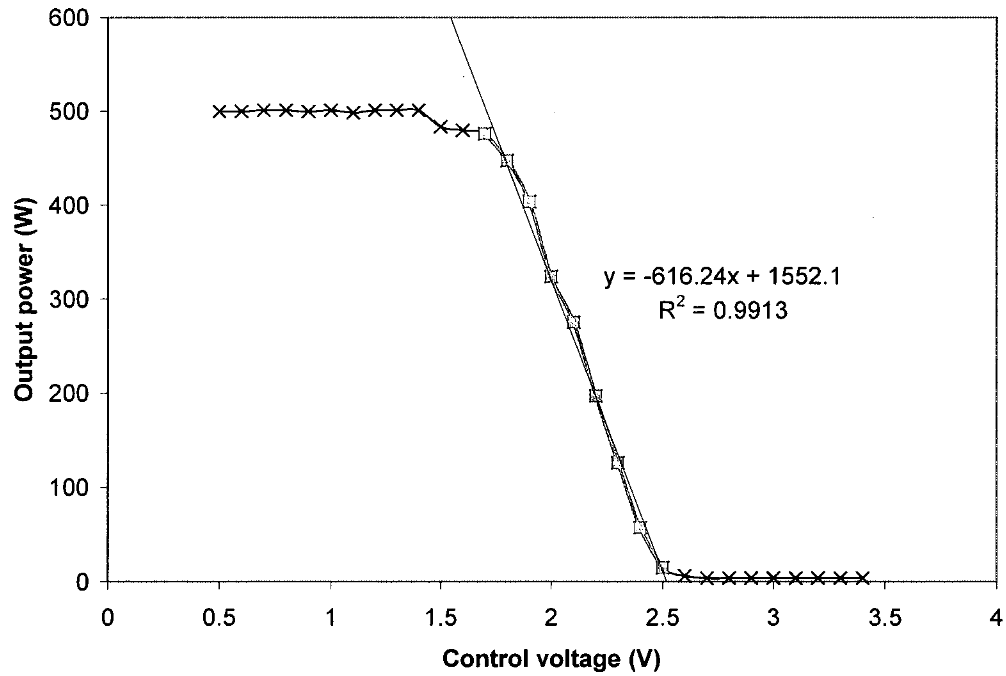

Fig. 6. 11 Microwave power calibration

6.4.2 Carrot drying

6.4.2.1 Carrot drying at fixed temperatures

Carrot cubes were first dried at four fixed temperatures: 50°C, 60°C, 70°C, and 80°C to investigate variations in aroma. Aroma profiles were detected and analyzed every 4 minutes, and important peaks were identified online and recorded. Temperature and power were also controlled and recorded automatically every second.

By employing the feedback system and phase controller, carrots temperatures were successfully controlled (Fig. 6.12). From the initial ambient temperature, preset temperatures were reached in a short time (1-3 minutes). The maximum deviation of temperature was ±5°C. The drying times required for 50°C, 60°C, 70°C, 80°C were roughly 400, 310, 210, and 150 minutes, respectively.

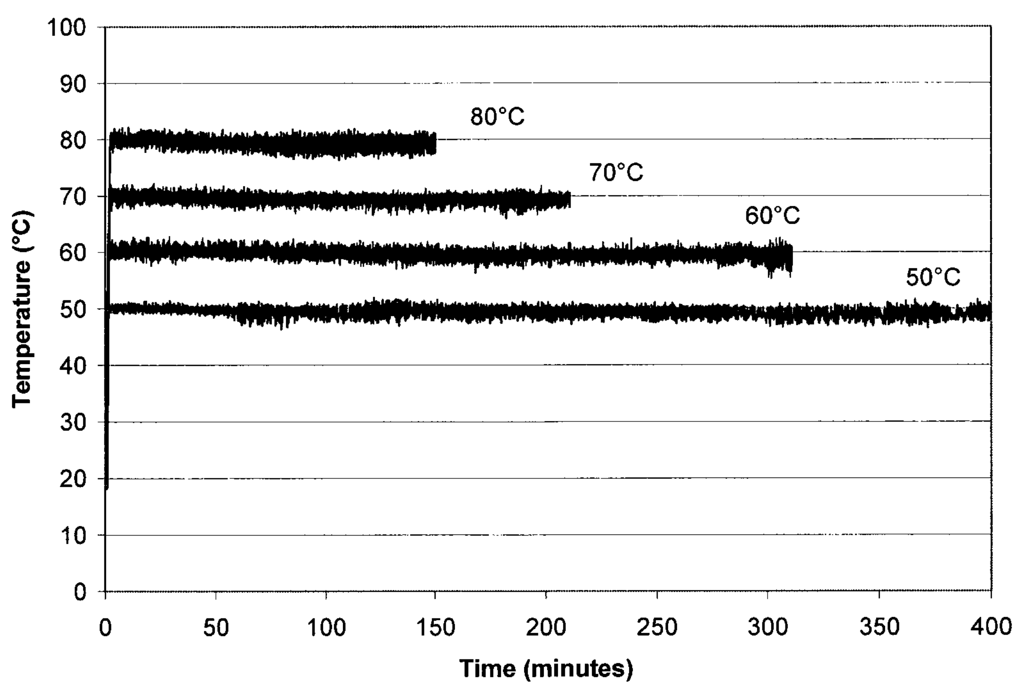

Fig. 6. 12 Carrot drying at fixed temperatures

Six marked peaks were identified with zNose and analyzed with the Labview program. However, only $Peak_4$ and $Peak_5$ will be discussed in this test as they were included in the fuzzy logic control. By comparing the Kovats index with the literature (Alasalvar *et al.,* 1999; Varming *et al.*, 2004), $Peak_4$ may represent caryophyllene. $Peak_5$ only appeared when the carrot was burned; hence it is an indicator of a burning aroma.

To facilitate visibility, only the signals of the first 60 minutes of $Peak_4$ were shown (Fig. 6.13), as thereafter signals were very small and little difference was found between the different drying temperatures. In contrast, burning $Peak_5$ is fully shown for the full drying period (Fig. 6.14).

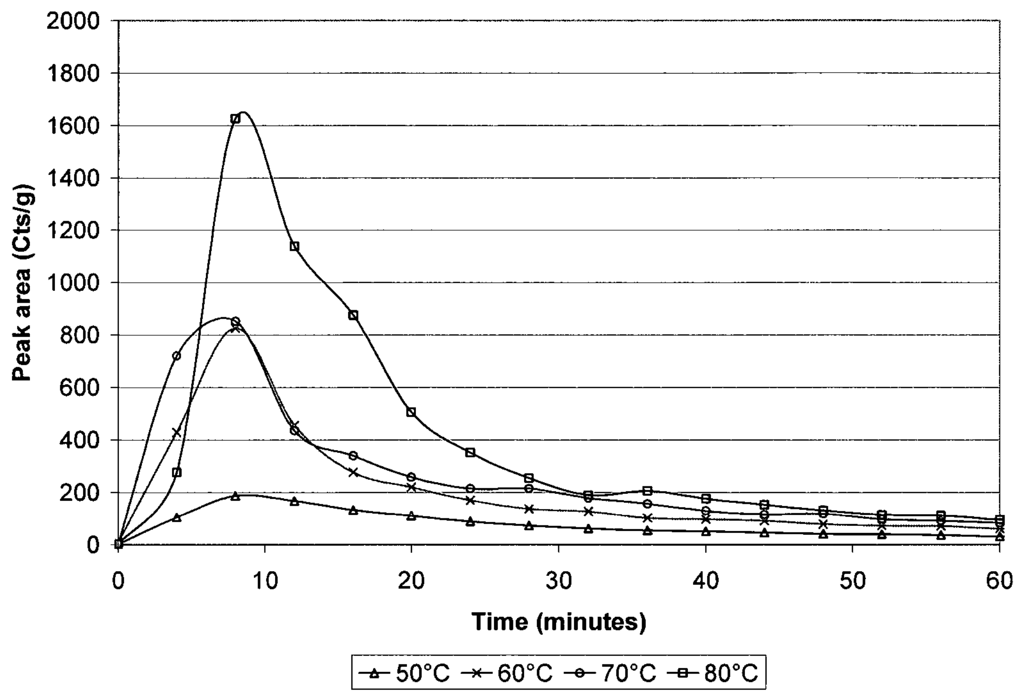

Fig. 6. 13 Normal carrot aroma as represented by $Peak_4$

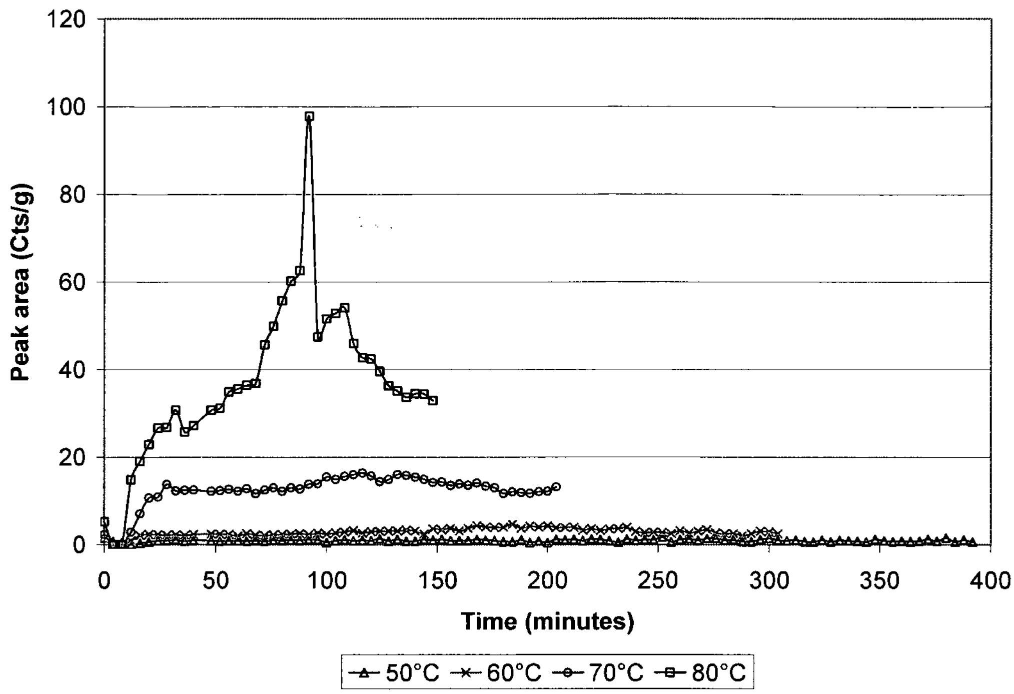

Fig. 6. 14 Burning as detected with $Peak_5$

A great deal of aroma compounds were lost and, as one might expect, more burning occurred at high temperatures (Figs 6.13 and 6.14). Greater $Peak_4$ signals appeared during the early stages of drying, while burning ($Peak_5$) occurred mainly in the middle drying stages. During the final stages, both $Peak_4$ and $Peak_5$ were very small.

6.4.2.2 Fuzzy control drying for carrots

The previous section showed that $Peak_4$ and $Peak_5$ both increased with an increase in temperature. Hence, low temperature drying is a good choice for aroma retention and to avoid burning. However, low temperature drying took longer and required more energy, hence there was a need to develop a method to shorten drying time, retain aroma, avoid occurrences of burning, and save energy. To fulfill this requirement, a fuzzy logic control was attempted. $Peak_4$ and $Peak_5$ were used as the two input variables of the fuzzy logic controller. The purpose of the controller was to reduce $Peak_4$ and suppress $Peak_5$ as much as possible. The output of the fuzzy controller was a temperature between 50°C and 80°C.

The controlled temperature profile is shown in Fig. 6.15. All three replicates gave similar results with little fluctuations (the remaining two are not shown). Carrot temperature increased from 60°C to 75°C in roughly 60 minutes and remained at 75°C until the end. The actual temperature followed the preset temperature tightly indicating an efficient temperature and power control. Total drying time was around 210 minutes, which was the same as that for 70°C carrot drying.

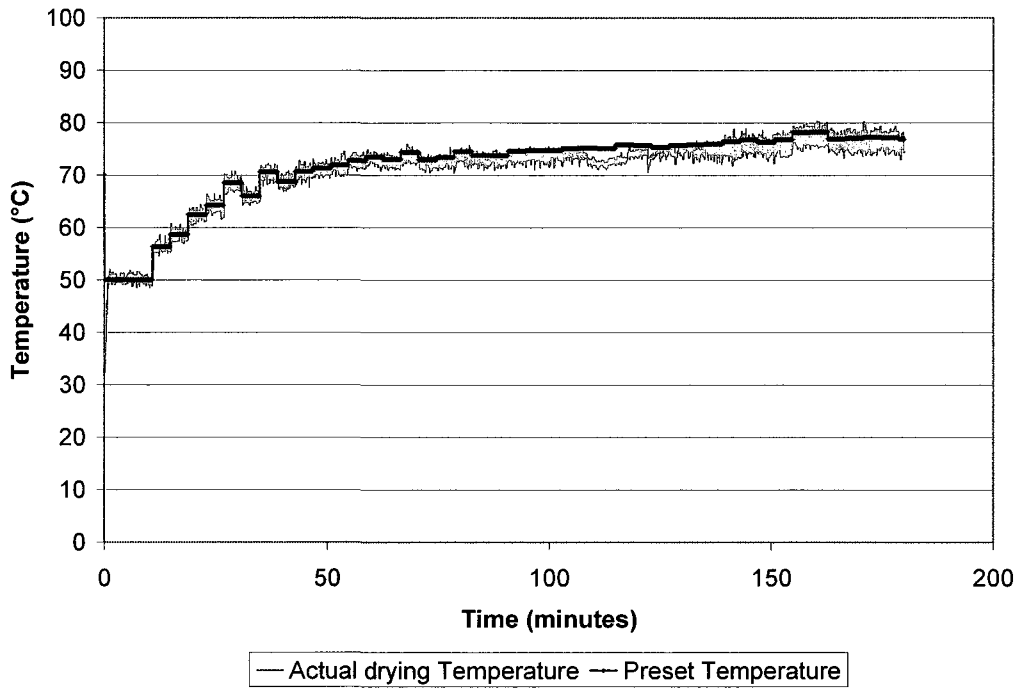

Fig. 6. 15 Fuzzy control of drying temperature for carrot cubes

Variations in $Peak_4$ and $Peak_5$ during fuzzy control drying (Fig. 6.16) showed that both peak values remained low, indicating the fuzzy logic control method was effective in terms of aroma retention. Burning suppression was also obvious with the new control strategy.

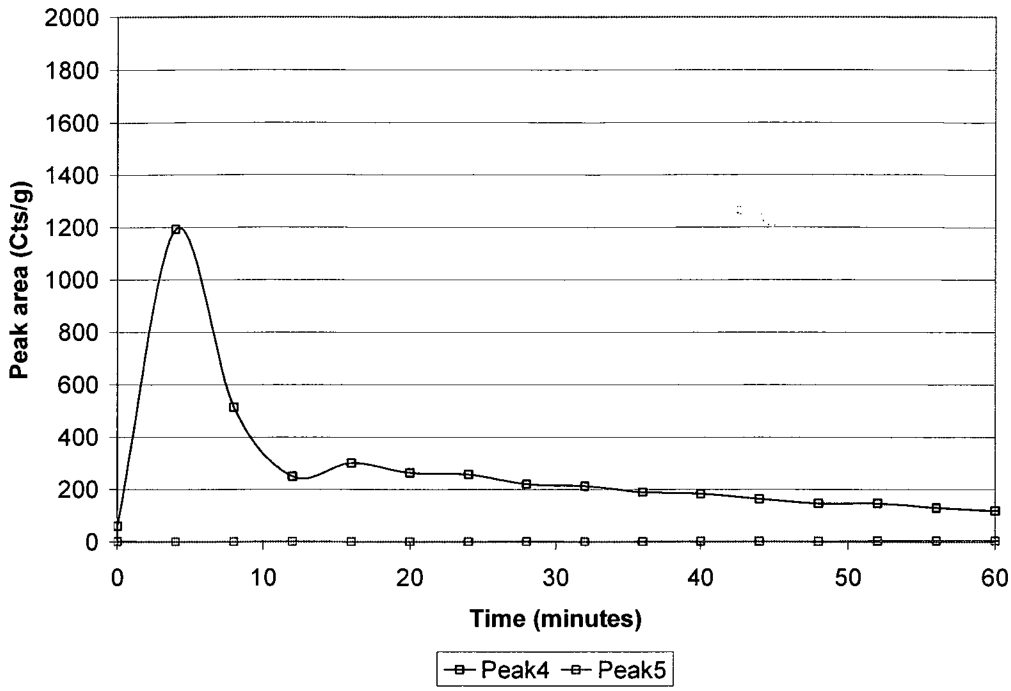

Fig. 6. 16 Normal carrot $Peak_4$ and burning $Peak_5$ levels during fuzzy controlled drying

6.4.3 Apple drying

6.4.3.1 Apple drying at fixed temperatures

Apple cubes were first dried at 60°C, 70°C, 80°C, and 90°C to investigate aroma variations. Aroma signals were also processed and analyzed with the program developed in Labview. Important peaks were identified online and recorded every 4 minutes during the drying processes. Temperatures and power were controlled and recorded every second.

With automatic power adjustments apple temperatures were successfully controlled. From the initial ambient temperature, preset temperatures were rapidly reached (1-2 minutes). The drying times for 90°C, 80°C, 70°C, 60°C were roughly 70, 85, 100, and 215 minutes, respectively (Fig. 6.17).

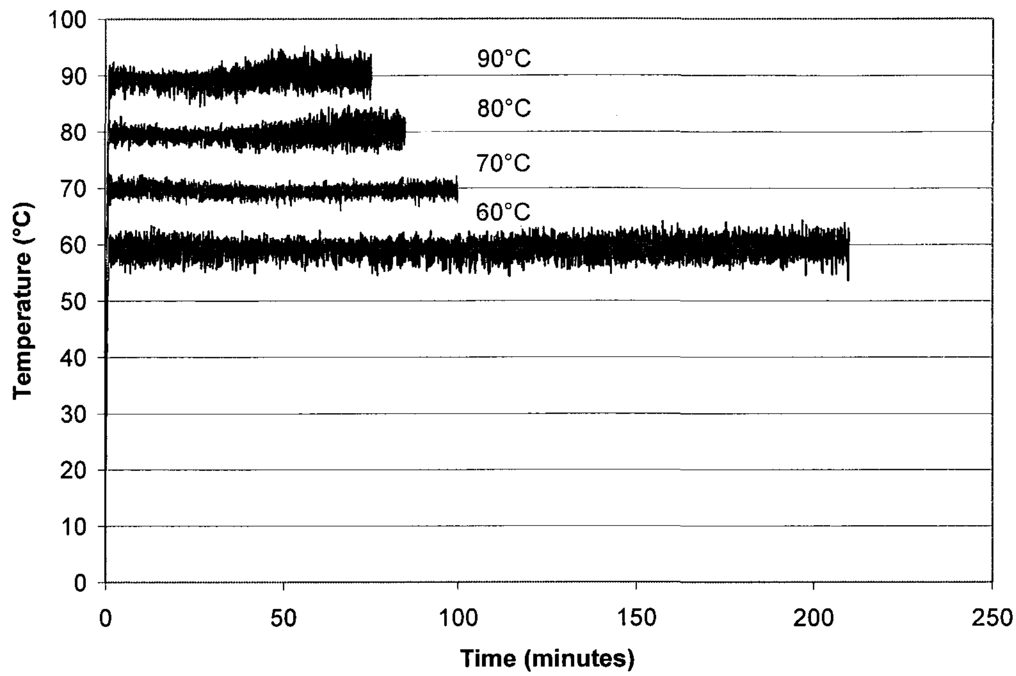

Fig. 6. 17 Apple cube temperature during apple drying at fixed temperatures

The highest preset temperature used was 90°C because above this, serious burning occurred, such as to make the final product quality totally unacceptable. The lowest temperature was 60°C because apple aroma would deteriorate if lower drying temperatures was used, i.e., longer drying times led to greater aroma loss.

Four significant peaks were detected with zNose during the microwave drying of apples, but only $Peak_2$ and $Peak_3$ will be discussed because they were included in the fuzzy logic control. $Peak_2$ was chosen as a representative of normal apple aroma and $Peak_3$ indicated the occurrence of burning.

$Peak_2$ and $Peak_3$ arose in the middle drying stages, and were not obvious at the beginning or at the end of the drying process (Figs. 6.18, 6.19). A temperature increase led to increases in both normal apple aroma and burning peaks. Burning $Peak_3$ was much larger than $Peak_2$. This result provided the preliminary observation that a low temperature was preferred in terms of aroma retention and to avoid burning.

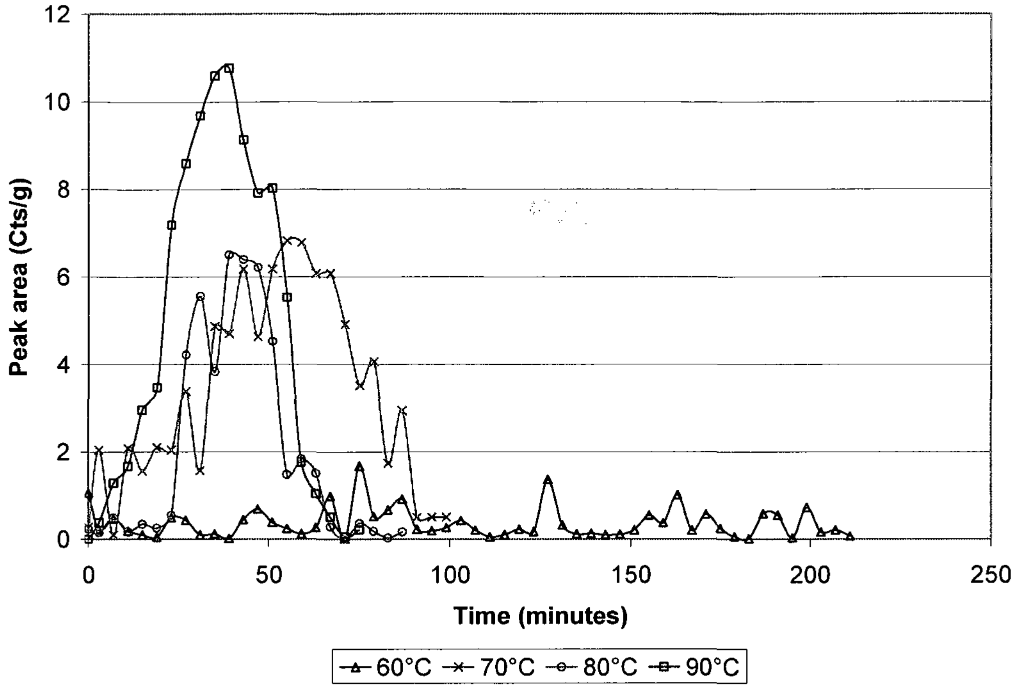

Fig. 6. 18 Apple $Peak_2$ at fixed temperatures

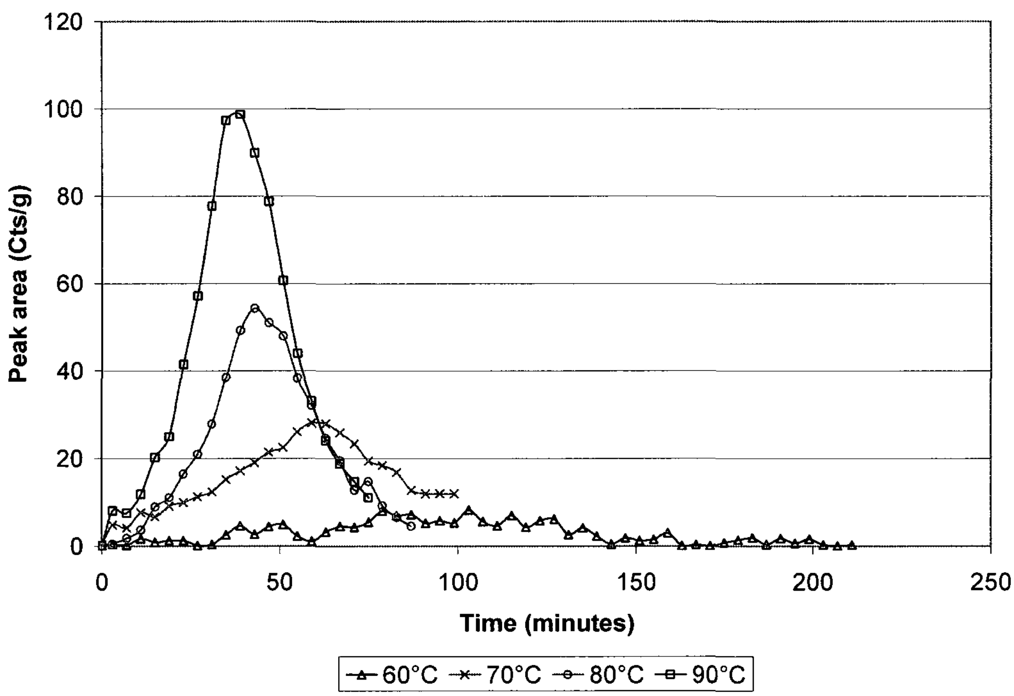

Fig. 6. 19 Apple burning $Peak_3$ at different temperatures

6.4.3.2 Fuzzy control drying for apples

Similar to fuzzy logic control for carrot cubes, $Peak_2$ and $Peak_3$ were used as two input variables to the fuzzy logic controller for apple drying. The output of the fuzzy controller was a temperature from 60°C to 75°C.

The controlled temperature profile is shown in Fig. 6.20. All three replicates had similar temperature curves with few fluctuations (the remaining two are not shown). Apple temperature decreased from 75°C to 65°C and then again increased to 75°C gradually, if the fluctuations are neglected. The actual temperature rapidly followed that set by the fuzzy control, indicating an efficient power control for the whole control process. Total drying time was around 120 minutes.

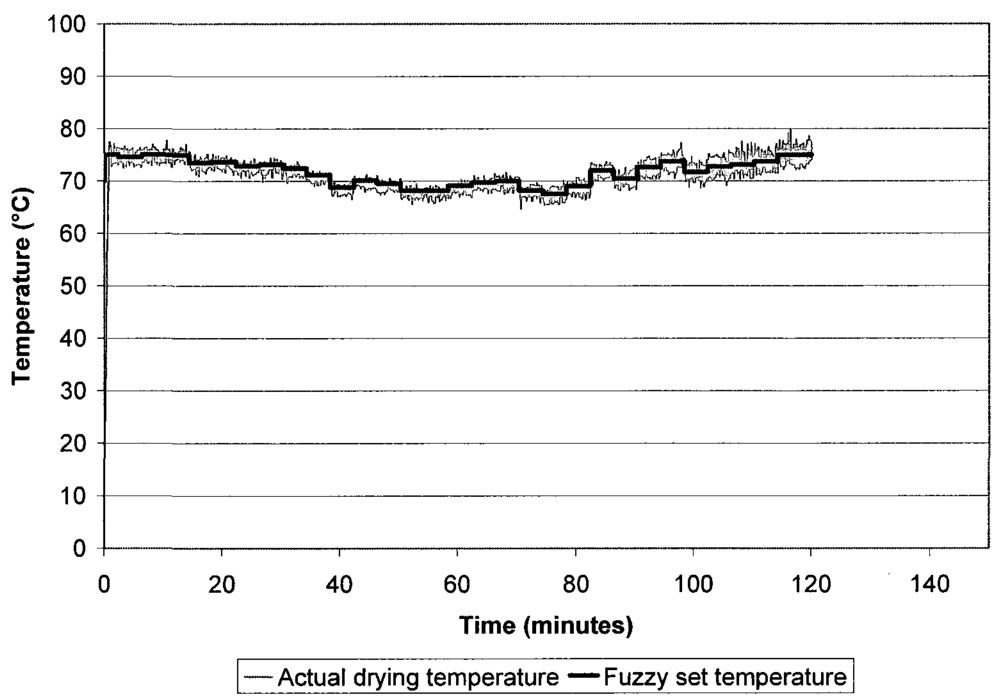

Fig. 6. 20 Fuzzy control of apple drying

Aroma peaks under fuzzy logic controlled drying (Fig. 6.21) were quite low indicating a satisfactory control in terms of aroma retention and burning suppression.

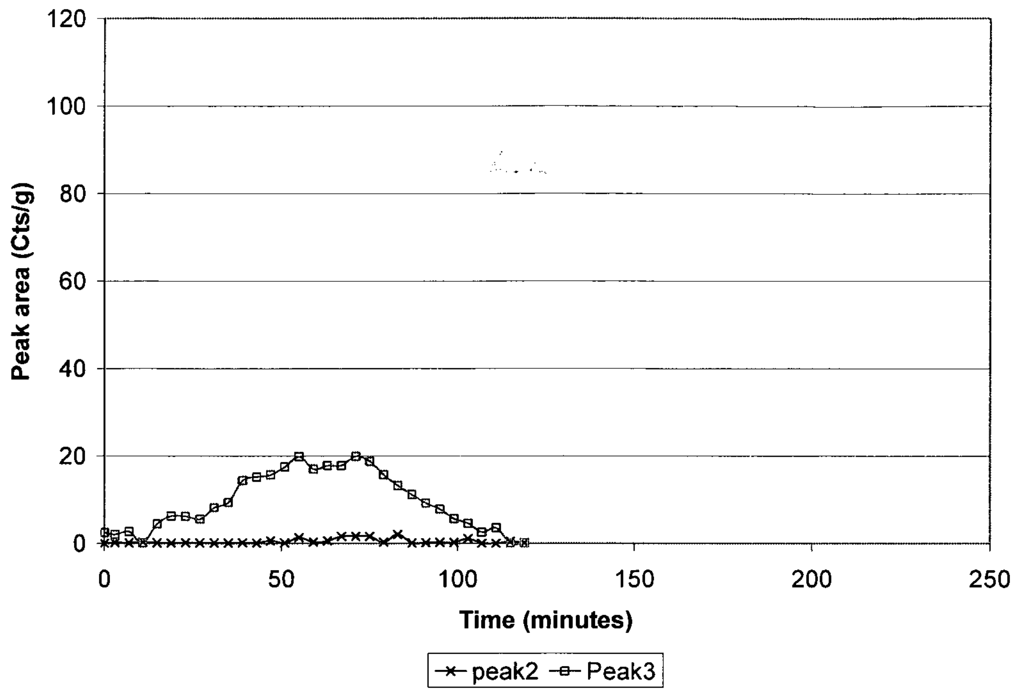

Fig. 6. 21 Normal apple peak and burning peak in fuzzy control drying

Comparing the detected aroma signals from carrot and apple drying, it was found that carrot aroma signals were much larger than those of apple, even at the same drying temperatures. This might be because that some apple aroma signals are beyond the trapping ability of the DB-5 column in the zNose, which can only collect C_5 to C_{22} substances. Further study needs to be conducted by GC/MS to confirm the major volatile compounds emitted from carrot and apple during microwave drying.

The resulting fuzzy logic control profiles from carrot and apple drying are quite different, indicating different drying requirements for different products. Therefore, specific studies should be conducted for different products to investigate their distinct control needs.

Another important but easy-to-neglect issue is the drying temperature in the final stages. Fuzzy controlled profiles of both carrot and apple revealed that in the final drying stages the temperature should be kept high. But this may not represent the best control pattern. The lesser aroma emission of samples under low moisture contents might lead to the high temperature output from the fuzzy logic controller in this stage, which might be erroneous. A low temperature may be better in the final stages in terms of aroma retention. Therefore, variations in samples' moisture contents during drying should be considered as another factor in the development of control strategies. However, this requires an automatic measurement of moisture contents, which was not included in this system. An advanced electrical scale would be highly recommended to allow such measurement.

6.5 Conclusions

A real time aroma monitoring and control system was developed and applied to microwave drying. Aroma was detected with a zNose and the signals were analyzed online with a program developed for this purpose. Important peaks were recorded automatically and used online for aroma control during drying.

Aroma control was implemented by varying drying temperatures. A phase controller was developed for microwave power adjustment. Actual temperatures followed the preset temperatures tightly for both fixed temperature drying and fuzzy controlled drying, indicating a successful power control.

Drying tests showed drying fresh produce at fixed temperatures cannot achieve ideal results when food aroma retention, burning prevention, and time efficiency were all considered. Fuzzy logic control could improve aroma retention, reduce burning occurrence, and shorten drying time.

For carrot drying at fixed temperatures, intensive aroma loss occurred during the early stages and burning occurred in the middle stages. For apple drying, however, all aroma signals were high in the middle stages. Drying apple took a shorter time than drying carrot, and their fuzzy logic controlled profiles were quite different. The results implied that different temperature profiles are required for different product types in terms of aroma control.

6.6 Acknowledgements

The authors are grateful to the NSERC (Natural Sciences and Engineering Research Council of Canada) for financial support.

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CONNECTING STATEMENT 4

In this chapter, an in-depth controlled drying experiment was conducted. Carrot aroma during microwave drying was detected and controlled with the system developed in the previous chapter. Different control strategies were employed to improve the product quality. Drying efficiency in terms of time and energy cost was also investigated.

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Optimal Control strategy for Microwave Drying through Aroma Monitoring

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Contributions made by different authors are as follows:

The first author, the PhD student did the experimental work and prepared the manuscript; the second and third authors were the supervisors who guided the research work.

CHAPTER VII

OPTIMAL CONTROL STRATEGY FOR MICROWAVE DRYING THROUGH AROMA MONITORING

7.1 Abstract

Carrot cubes were dried in a microwave oven and the aroma was monitored and controlled. An electronic nose was used to detect carrot aroma. Drying temperatures were first set to 50°C, 60°C, 70°C, and 80°C. Six typical peaks were recorded every 4 minutes for all the three replicates. Color, aroma and sensory evaluation were performed after drying. An untrained taste panel was included in the sensory evaluation. Samples could be dried in a short time at high temperatures but the interior of some cubes was burnt. Drying time at low temperatures was much longer and much more aroma was lost. For fixed temperatures, the best results were achieved at 60°C. A fuzzy logic control system was then designed and employed to control the whole drying process, using carrot aroma peaks as variables. Based on a fuzzy logic control profile, a simple linear control method was developed. It was shown that the carrot color and aroma were intact with these new control strategies and less time and power were consumed.

Keywords: Carrot, aroma, microwave, drying, fuzzy control

7.2 Introduction

Aroma is one of the most important quality aspects of food products. This characteristic, to a great extent, determines consumers' preference and acceptance. However, in most food processing, aroma is often lost, changed, distorted, or even destroyed. Preserving preferred aromas and avoiding unacceptable aroma occurrence is a crucial task in the food industry. To fulfill this task, food aroma must be correctly and rapidly detected online during food processing. Unfortunately, most current instruments, either electronic noses or conventional analytical chemical equipment, are all time-consuming and complex (Li *et al.,* 2006), which makes it almost impossible to detect food aroma online. So far, no literature is available regarding online detection of food aroma, and only a few offline aroma measurements have been reported (Kompany and Rene, 1993; Krokida and Philippopoulos, 2006; Samuelsson *et al.*, 2006). This crucial task still remains unsolved.

In recent years, a new aroma detection instrument called zNose^{TM} (7100 Fast GC Analyzer, Electronic Sensor Technology, New Bury Park, CA, USA) has become commercially available. This new instrument has the capacity of detecting food aroma as fast as in seconds, which, for the first time, makes online detection of food aroma possible. However, the application of the zNose has remained in offline detection (Lammertyn *et al.*, 2004; Gan *et al.*, 2004) and no online employment has been reported.

Carrot is one of the most commonly consumed vegetables for its nutritional benefits, given its high vitamin and mineral content. To enhance their preservation, a number of drying techniques have been developed for carrot dehydration (Doymaz, 2004). Amongst these methods, microwave drying is considered time and energy efficient. However, while some advantages of this dehydration technique have been broadly estimated, no online aroma evaluation during microwave drying process is hitherto reported. How the aroma is altered and how to improve the microwave drying technique to achieve a better product quality in terms of aroma is still unknown.

The objectives of this study were:

1) To evaluate carrot aroma variations during microwave drying processes;
2) To improve microwave drying in terms of aroma quality with new control strategies;
3) To compare drying kinetics, energy consumption, and product quality under different microwave drying conditions.

7.3 Materials and Methods

7.3.1 Aroma monitoring and control system

An aroma monitoring and control system was designed for microwave drying (Fig. 7.1). The system consisted of an aroma detection unit, a microwave drying system, a temperature and power control system, and a PC based data acquisition unit (DAQ). The aroma detection unit included a zNose, a two-position pneumatic valve, an air flow installation, a moisture condenser, and a flow meter; the microwave drying system included a domestic microwave oven, a Teflon container, and an electronic scale; the temperature and power control system included an optical fiber sensor, a control circuit, a 12 VDC and an 110 VAC power supplies; the data acquisition unit was a PCI 6014 multifunction device (National Instruments, TX, USA) with analog outputs to facilitate the MW power control.

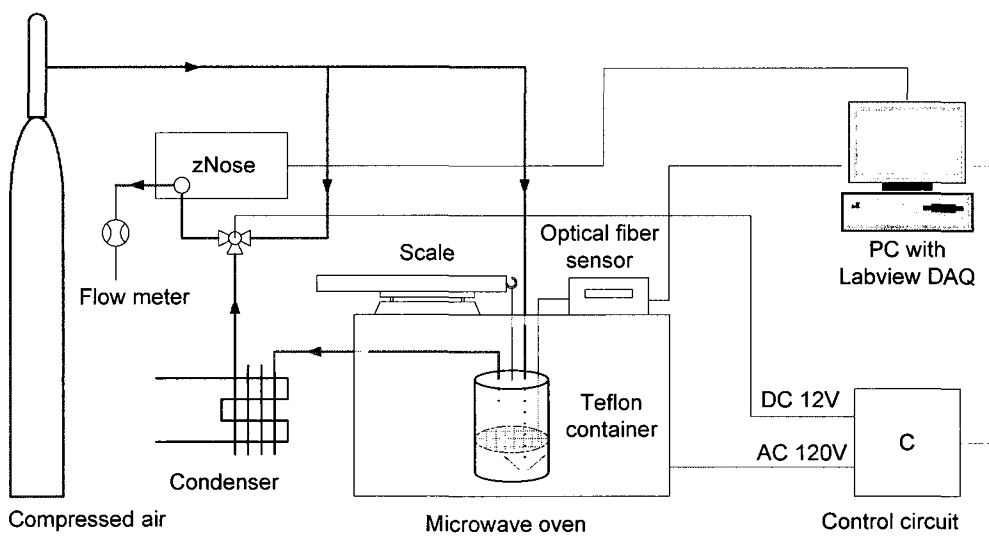

Fig. 7. 1 Schematic diagram of the aroma monitoring and control system

7.3.1.1 Aroma detection unit

The zNose is actually a fast GC. It can detect aroma in as little as 1 minute and automatically repeat the measurement procedures. Detailed description of the instrument can be found elsewhere (Lammertyn *et al.*, 2004). Just as a regular GC, detected volatiles are expressed in terms of retention times and peak areas, which represent different compounds and their quantities, respectively. The temperature of the sensor was set to 60°C and sampling time was 6 seconds.

A two-position pneumatic valve (M223W1DFR-HT-312, Teqcom, CA, USA) was installed in front of the zNose to alternately detect aromas and clean air for detection of sample aroma and cleaning the zNose, respectively. Both detection and cleaning cycles were set to 2 minutes; hence sample aroma was measured every 4 minutes. Different peaks were assumed to represent different volatiles. By calibrating with standard chemicals (n-Alkanes), zNose converted detected peaks' retention times to a Kovats index (Appendix C). Hence, chemical compounds could be tentatively identified by checking a library of related volatile compounds.

Compressed air was used to carry the vapour from the Teflon container and microwave oven and bring it to the zNose. Air pressure was adjusted to 10 psi with a regulator and flow rate was controlled at 1000 mL min^{-1} with an adjustable flow meter. Moisture was collected in an air-cooled condenser just after the microwave oven and before the zNose. Hence, only dry volatiles were allowed to reach the zNose.

All tubes and connectors were made of Teflon so as not to retain any aroma and to facilitate microwave drying. The air-cooled condenser made of 6 slim glass bottles was carefully cleaned after each experiment.

7.3.1.2 Microwave drying system

A 600 W domestic microwave oven (Beaumark 02314, Matsushita Electric Ind. Co. Ltd., Yamatokoriyama, Japan) was used for drying. The stirrer and fan were modified to be controlled separately and were fully on during the whole drying process. Power supply to the magnetron was phase-controlled with the DAQ and a control circuit.

Samples were dried in a Teflon container, equipped with a small porous basket to hold samples and to allow air to go through. The mass of the container with the sample was monitored manually every 30 min with an electronic scale (PK 4801, Denver Instrument, Denver, CO, USA). An optical fibre sensor (Nortech Fibronic Inc., Quebec Canada) was inserted in the center of one of the samples for temperature measurements.

7.3.1.3 Temperature and power control system

There were two main parts to the temperature and power control system: a phase controller to adjust the power level, which could automatically and continuously adjust AC power according to the temperature requirements, and a relay and its driver for on/off control of the pneumatic valve. The low voltage part of the circuit, which connected to the DAQ and PC, was protected with optical-electrical coupling devices.

7.3.1.4 Data acquisition unit

A PC housed the Labview program (Labview 7.1, National Instruments, TX, USA) and a Multifunction Data Acquisition device (PCI 6014, National Instruments, TX, USA) for data collection and system control. The PCI 6014 is a 16-bit resolution DAQ board operating at a 250 kS/s rate and includes 2 analog outputs, 8 digital I/O lines, and two 24-bit counters. Three ports of the DAQ were used in this study: an analog input for the optical fiber temperature sensor, a DC output to control the two-position pneumatic valve, and an analog output to adjust AC power to the magnetron. All the controls were implemented in the Labview environment.

7.3.2 Experiment design

7.3.2.1 Sample preparation

Carrots of unknown cultivar were purchased from a local market and stored at 4°C. Initial moisture content was determined by drying fresh carrots at 70°C for 12 hours in a hot air oven (Ranganna, 1986). Peeled carrot samples in 10×10×10 mm cubes (25±1g) were used. All samples were dried to a final moisture content of 10% (wet basis).

7.3.2.2 Carrot drying at fixed temperatures

Carrot cubes were first dried at four fixed temperatures: 50°C, 60°C, 70°C, and 80°C. Temperatures were automatically recorded every second by the PC. Aroma signals were processed and analyzed with a programme developed in Labview. Important peaks were identified online and recorded during the drying process.

Mass variations of the carrot samples during MW drying were monitored and recorded manually every 30 minutes and the moisture contents were calculated. Power rates were automatically recorded every second by the PC.

7.3.2.3 Control strategies for microwave drying

Based on the observation and analysis of aroma variation in the fixed temperature drying processes, a fuzzy logic controller was designed. The purpose of the fuzzy control was to decrease aroma loss, avoid the occurrence of burning, and increase drying speed. Aroma and burning peaks were used as input variables and the output was temperature adjustment. The Fuzzy rules are outlined in Table 7.1. Input variables are restricted to two in order to reduce the complexity of the fuzzy controller and allow the fuzzy rule to be practical and clear.

Table 7. 1 Fuzzy rule base for carrot aroma control

Burn \ Carrot	Low	Medium	High	Very high
Low	P	Z	N+	N+
Medium	N	N	N+	N+
High	N+	N+	N+	N+
Very high	N+	N+	N+	N+

* P: positive, Z: zero, N: negative, N+: more negative

The functions of input variables were triangles. Fuzzy inference was Max – Min, while the defuzzification method was center of gravity, and the output function was singleton. All these designs simplified the fuzzy logic controller and also smoothed the temperature outputs.

Depending on the fuzzy controlled temperature profiles, a simple linear control was further designed to make it possible to popularize the control methodology. All parameters were recorded as in the previous section.

The drying results of the two strategies were compared to each other and to fixed temperature drying as well. A comparison of aroma profiles, moisture contents, and power consumption was conducted.

7.3.3 Quality evaluation

Quality of rehydrated carrot products from the above six drying methods was evaluated and compared, because in most cases, dehydrated carrots are to be consumed in their rehydrated form. The rehydration procedure was conducted according to USDA standards (Anon, 1944). Distilled water (150 mL) was brought to the boiling point in a 500 ml beaker and allowed to boil for 3 minutes before a 5g sample of dried carrots was added and boiled for another 5 minutes. Samples were drained and excess water was removed with a filter paper, surface color was measured with a colorimeter (Model CR-300X, Minolta Camera Co. Ltd., Japan). Each measurement of the colorimeter provided three values: L*, a*, b*, representing lightness, purple-red and blue-green coefficients, and yellow-blue coefficients, respectively. The ratio (a/b) is a convenient way of indicating redness of the products and was employed in this study (Changrue, 2006).

Aroma of the rehydrated samples was then measured with the zNose. Rehydrated samples (10 g) were sealed in a 40ml vial for 30 minutes before measurement. Fresh carrot cubes (10g) were also measured for comparison.

A taste panel of ten untrained judges was used to assess overall appearance and taste of the rehydrated carrot samples. A balanced 9-points hedonic rating was employed, where 9 denoted "like extremely" and 1 indicated "dislike extremely" (Resurreccion, 1998; Lin *et al.*, 1998).

7.3.4 Statistical analysis

All experiments were conducted with three replicates and mean values were reported. Error bars representing standard deviations for moisture contents were also included. SAS software (SAS 9.1, SAS Institute Inc, Cary, NC, USA) was used to perform the ANOVA procedure for power consumption, redness, aroma peaks of rehydrated carrots, overall appearance and taste. Duncan's Multiple Range Test was carried out for each of the above factors. Typical standard deviations of aroma signals are listed in Appendix F.

7.4 Results and Discussion

7.4.1 Carrot drying at fixed temperatures

Carrot was first dried at four fixed temperatures: 50°C, 60°C, 70°C, and 80°C. Temperatures and drying time were recorded, as well as moisture contents and power rate.

7.4.1.1 Temperatures and drying times

Carrots' temperatures were successfully controlled with appropriate power levels. Starting at the initial ambient temperature, preset temperatures of 50°C, 60°C, 70°C, and 80°C were reached within 1-3 minutes. The maximum deviation in temperature was ±5°C. The drying times required for 80°C, 70°C, 60°C, and 50°C were roughly 150, 210, 310, and 400 minutes, respectively (Fig. 7.2). This result indicated that drying at lower temperatures required a longer time.

The highest temperature used was 80°C since above this temperature serious burning occurred, making the final product quality totally unacceptable. The lowest temperature was 50°C, as the product quality in terms of flavour would deteriorate if a lower drying temperature was applied due to longer drying time.

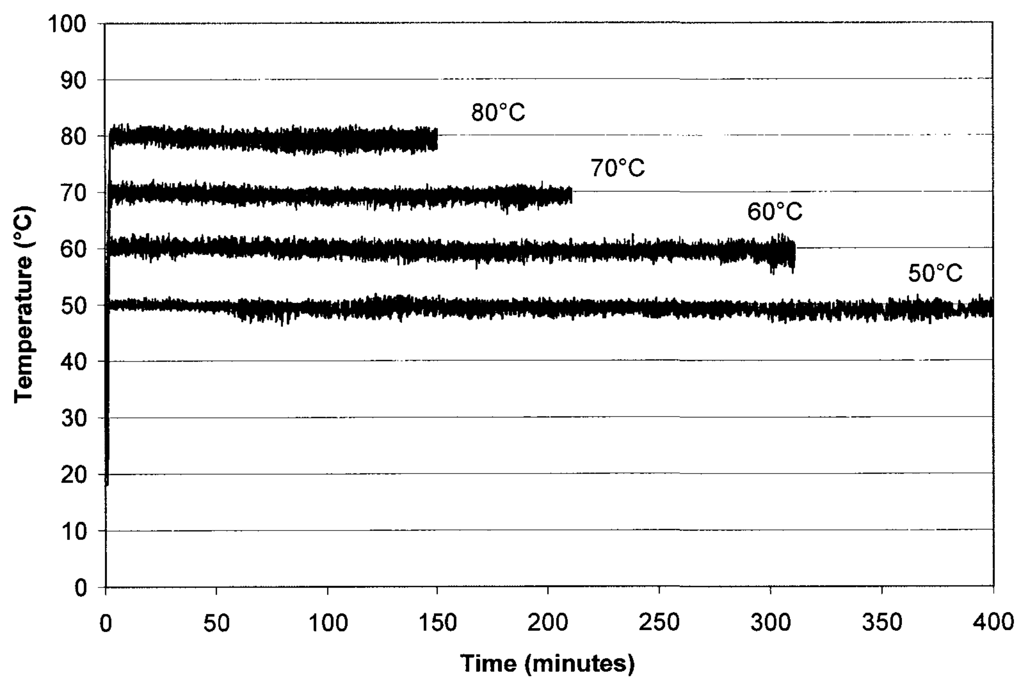

Fig. 7. 2 Temperature regulation during carrot drying at fixed temperatures

7.4.1.2 Aroma profiles

Six distinct peaks were detected with zNose. By comparing their Kovats index with literature (Alasalvar *et al.,* 1999; Varming *et al.*, 2004), it was found that the first four may represent α-pinene, β-pinene, α-terpinolene, and caryophyllene, respectively. These four peaks were also detected from fresh carrot cubes and hence will hereafter be termed "carrot peaks". The remaining two peaks, corresponded to the burning of carrots, were only detected when carrots were burned and were not present in fresh carrots. They will be hereafter termed "burning peaks".

To facilitate visibility, only the first 60 minutes of carrot peaks are shown (Fig. 7.3a-d), thereafter values were small and showed few differences amongst themselves. In contrast, the burning peaks are shown for the full period (Fig. 7.3e-f).

More aroma was lost and more burning occurred at high temperatures (Fig. 7.3). $Peak_3$ and $Peak_4$ were much larger than other peaks (note the scale of the figures) and were maintained for a longer time. The intensive aroma of carrot was mainly lost in the early stages, while burning occurred mainly in the middle drying stages. In the final stages, both carrot peaks and burning peaks were very small.

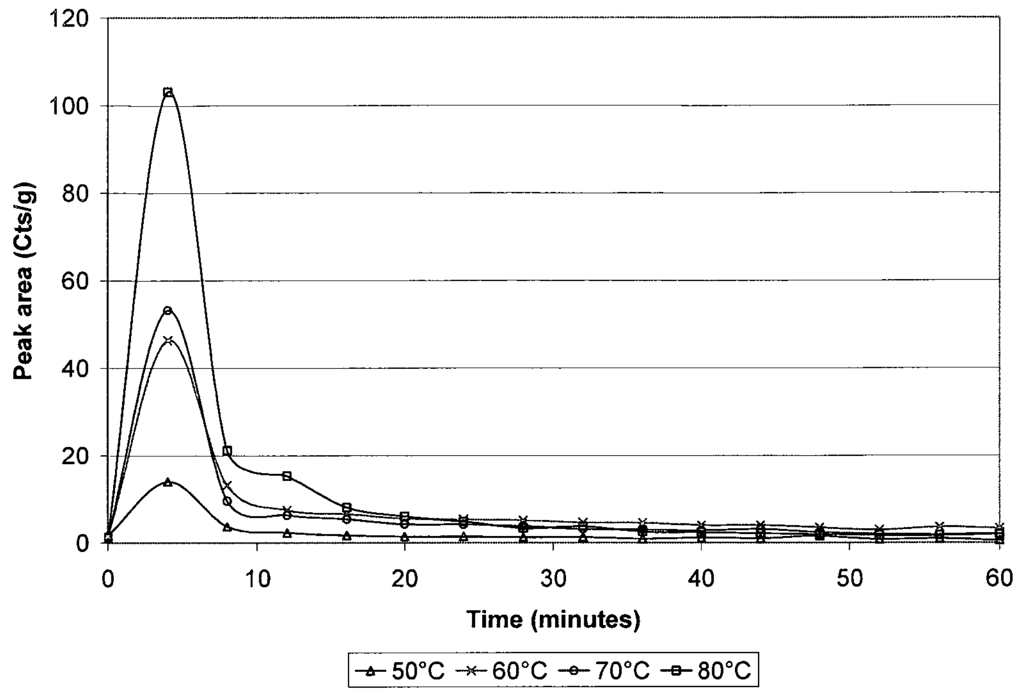

Fig. 7.3a Carrot $Peak_1$ at fixed temperatures

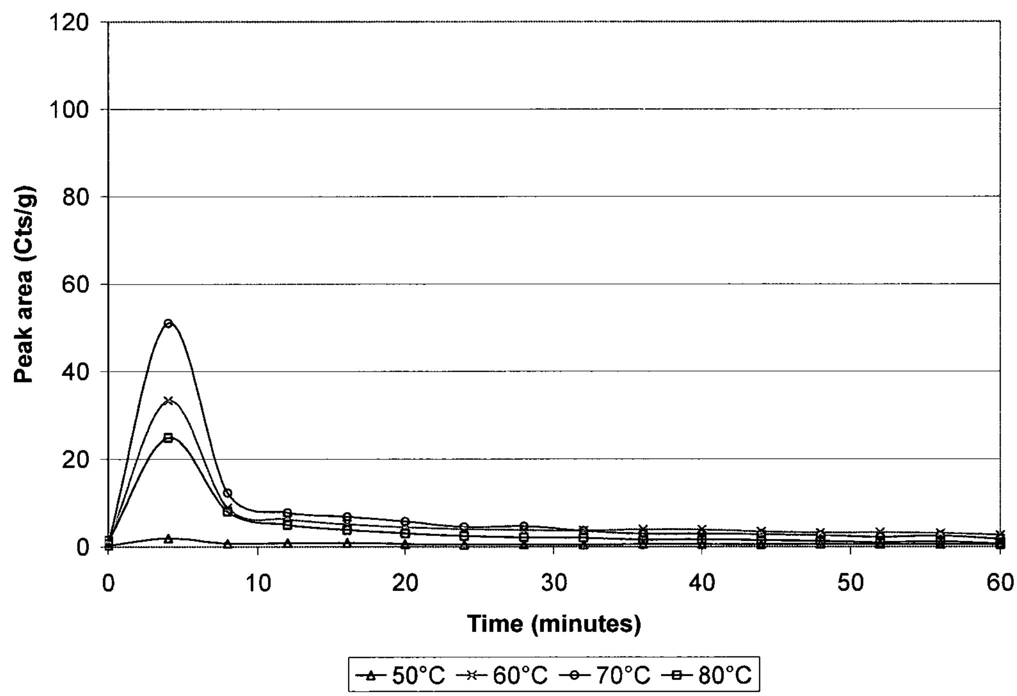

Fig. 7.3b Carrot $Peak_2$ at fixed temperatures

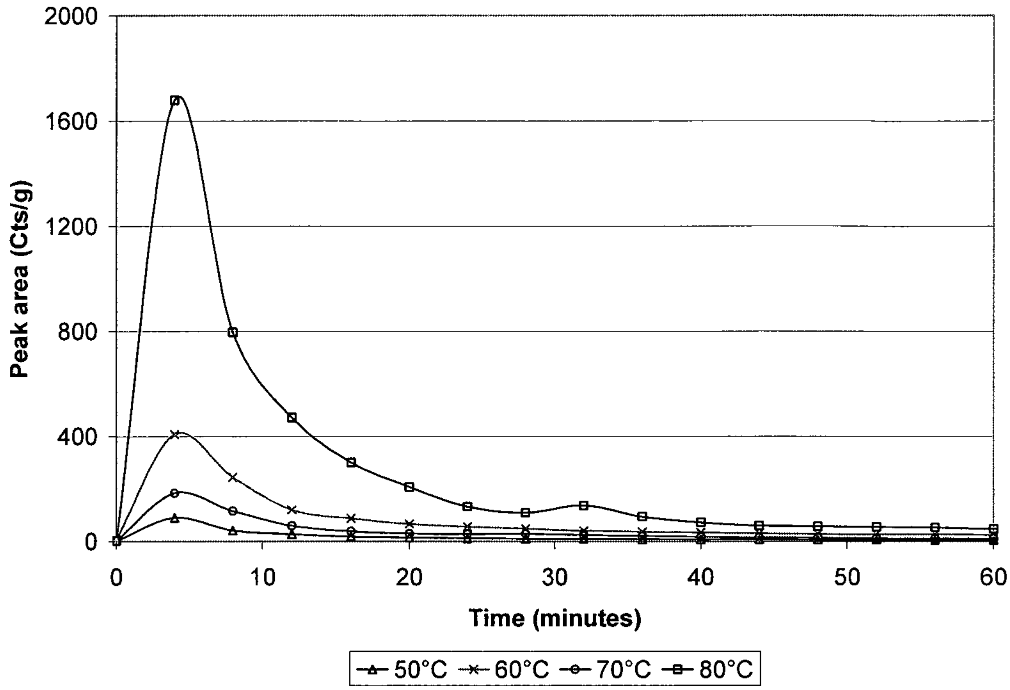

Fig. 7.3c Carrot Peak$_3$ at fixed temperatures

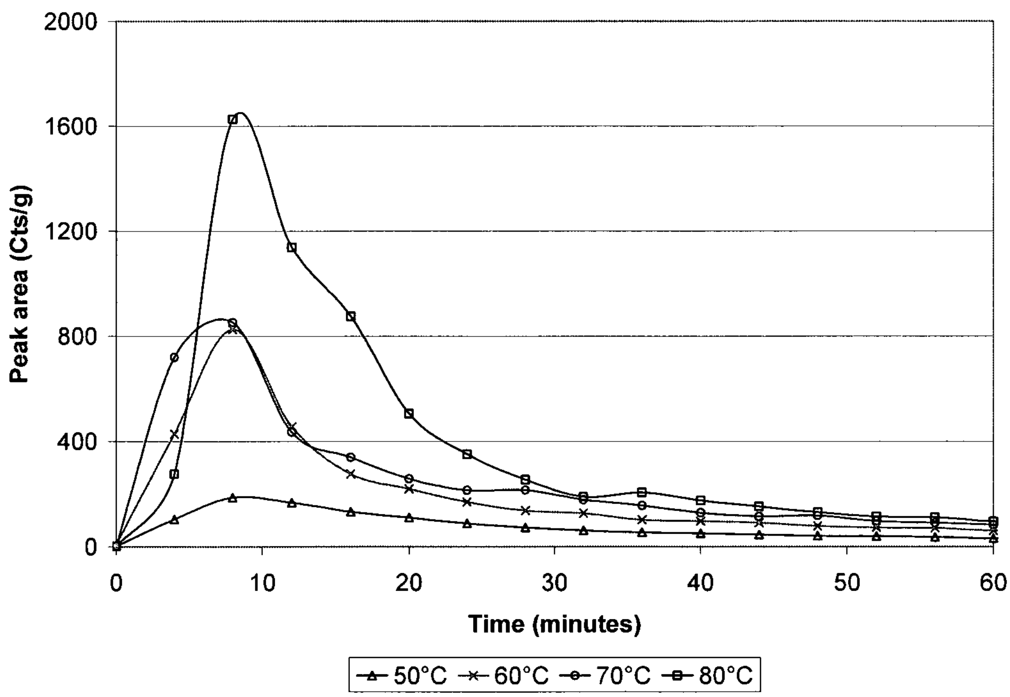

Fig. 7.3d Carrot Peak$_4$ at fixed temperatures

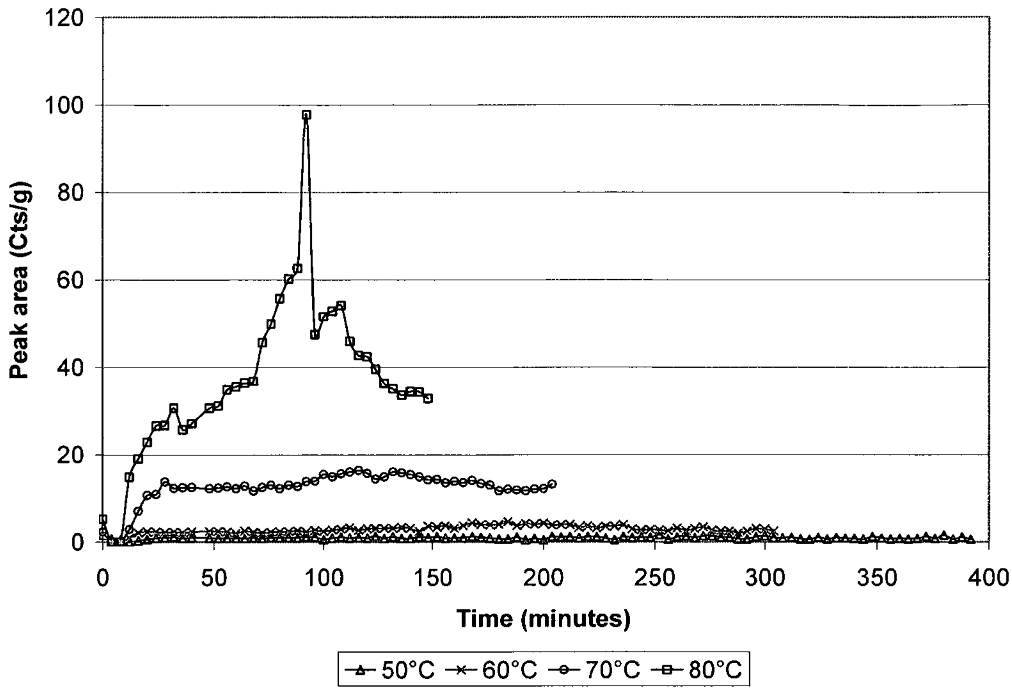

Fig. 7.3e Carrot 'burn' $Peak_5$ at fixed temperatures

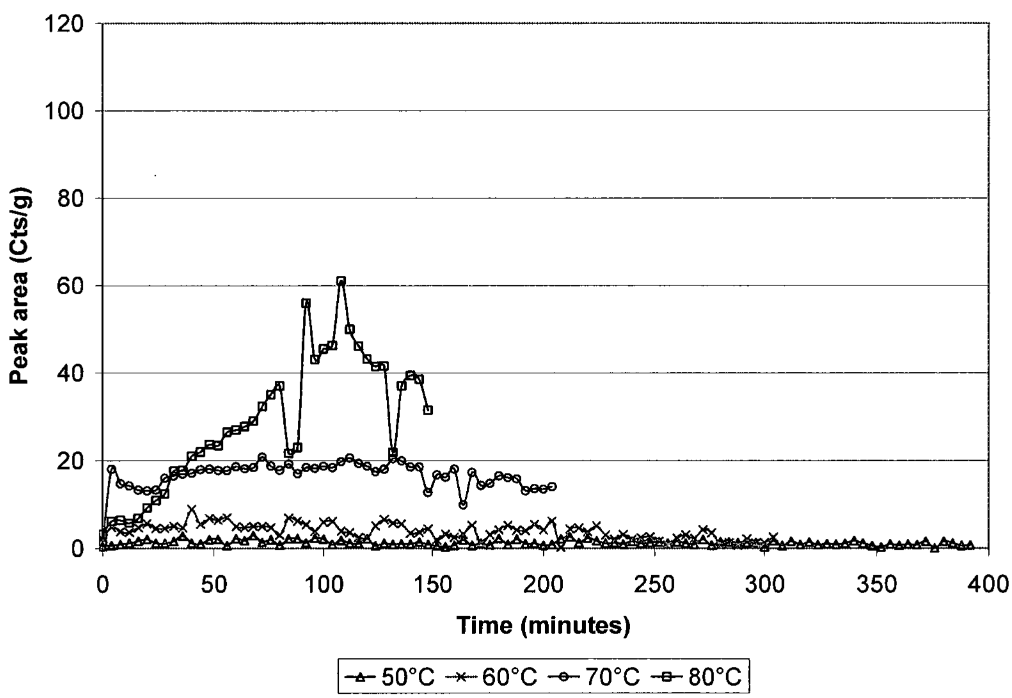

Fig. 7.3f Carrot 'burn' $Peak_6$ at fixed temperatures

Fig. 7. 3 a-f Carrot $Peaks_{1-6}$ at fixed temperatures

7.4.1.3 Moisture contents

The initial moisture content of fresh carrots was 88.26%. The curves of moisture contents at four fixed drying temperatures (Fig. 7.4) show that drying speeds increased with the increase in drying temperatures, with fastest drying rate being achieved at 80°C. Curves were not smooth at some stages; this might be because of moisture condensation in the container. However, the moisture was completely removed in the final drying stages and did not influence the final mass measurement.

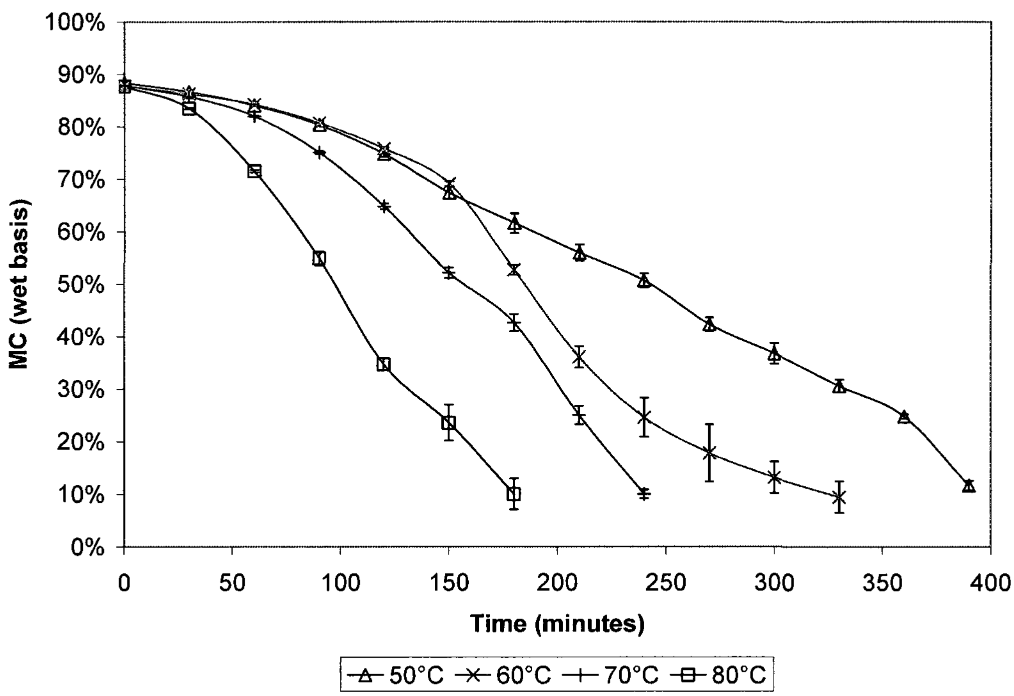

Fig. 7. 4 Moisture contents of carrot cubes at fixed temperatures (Error bars represent standard deviations)

7.4.1.4 Energy consumed

Power rates applied to the magnetron were adjusted and recorded every second. For high temperature drying, higher power rates were required.

Appropriate power levels were carefully selected to be just sufficient to maintain the temperature requirements but not sufficient to overheat the magnetron.

By summing the power rate over time a total quantity of energy consumed was calculated. Mean power rate and total energy consumed at different drying temperatures is shown in Fig. 7.5. It is worth emphasizing that although the power rate applied at high temperatures were high, the total consumed energy was lower due to the shorter drying time. Hence, higher temperatures' drying would be preferred in terms of energy efficiency.

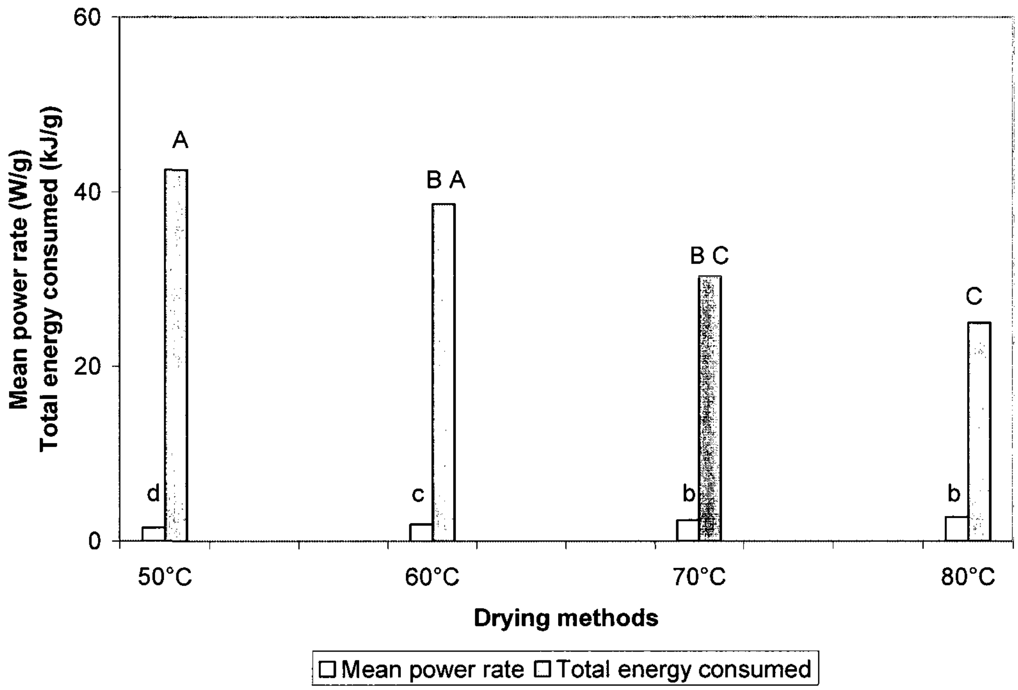

Fig. 7. 5 Mean power rate and total energy consumed when drying carrots at fixed temperatures
(Mean values, values with different letter are significantly different, $\alpha = 0.05$, letters in lower case: mean power rate; letters in uppercase: total energy consumed)

Considering drying times, energy consumed, and aroma variations at four fixed temperatures, it was clear that at higher temperatures, drying times were shorter, energy consumed was less, but more aroma was lost and more burning occurred. The phenomenon inspired one to search for a compromise between drying time, carrot aroma, burning occurrence, and energy consumption requirements, which would result in a better microwave drying methodology.

7.4.2 Fuzzy logic and linear control

Carrot was dried using fuzzy and linear controlled methods. Temperatures and drying time were recorded, as well as moisture contents and power rate.

7.4.2.1 Fuzzy control

Analysis of aroma peaks in previous experiments showed that $Peak_3$ and $Peak_4$ had large value and were maintained for a significant portion of the drying process. They both had similar trends in their variation. Hence, $Peak_4$ was chosen as a representative of the four "carrot peaks," while $Peak_5$ was chosen as representative of "burning peaks" because $Peak_6$, as a large retention time peak, tended to persist in the system even after cleaning cycles. As a result, $Peak_4$ and $Peak_5$ were used as two input variables to a fuzzy logic controller. The output of the fuzzy controller was a temperature between 50°C and 80°C.

An aroma-basis controlled temperature curve is shown in Fig. 7.6. All three replicates gave similar results with little fluctuations (the remaining two are not shown). The actual temperature followed the preset temperature tightly indicating an efficient power control. Total drying time was around 210 minutes, which was similar to that for a set temperature of 70°C. Aroma peaks, moisture content, and power consumed were compared.

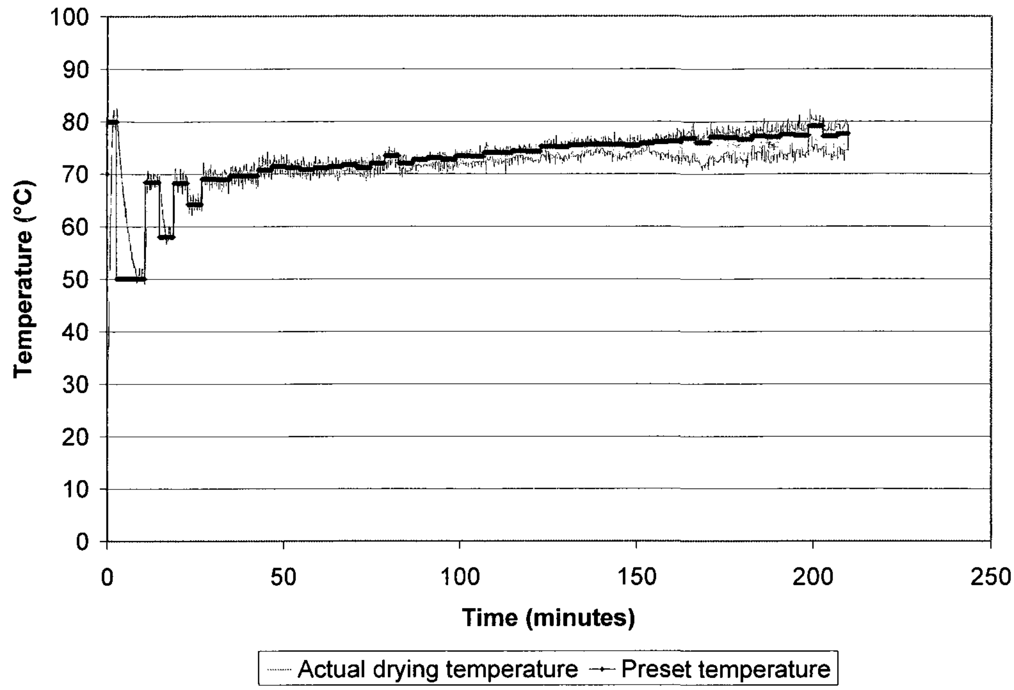

Fig. 7. 6 Fuzzy controlled drying temperatures

7.4.2.2 Linear control

Neglecting fluctuations in temperature in the early stages, observations of the temperature profile of fuzzy controlled drying showed that according to the aroma control requirement, temperature should be increased in the beginning stages from 50°C to 75°C within roughly 60 minutes and then maintained at around 75°C. A linear controller was developed based on these observations. This method simply increased the temperature from 50°C to 75°C in exactly 60 minutes and then kept the temperature at 75°C until the end of drying. With this control strategy, aroma detection was not required. Total drying time was 180 minutes (Figure 7.7), intermediate between that of 80°C and 70°C and shorter than fuzzy control drying.

Another benefit of the linear control is that it does not require the assistance of zNose and can stand alone. As the zNose is a costly instrument, the linear control is a more practical and cost-efficient drying control method.

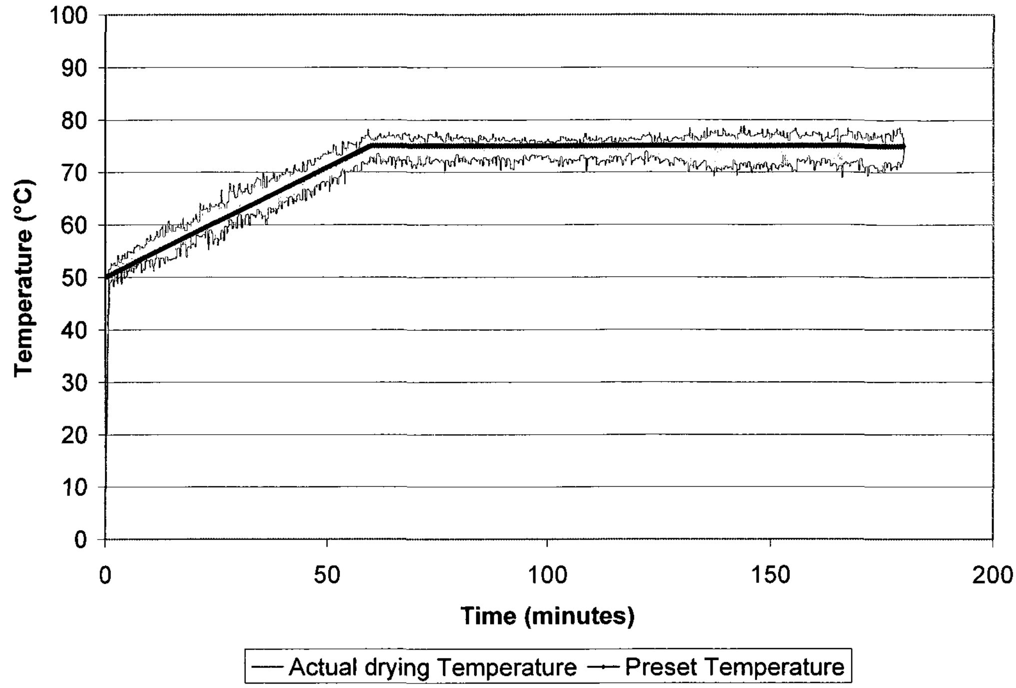

Fig. 7. 7 Temperatures during linear controlled drying

7.4.2.3 Aroma profiles

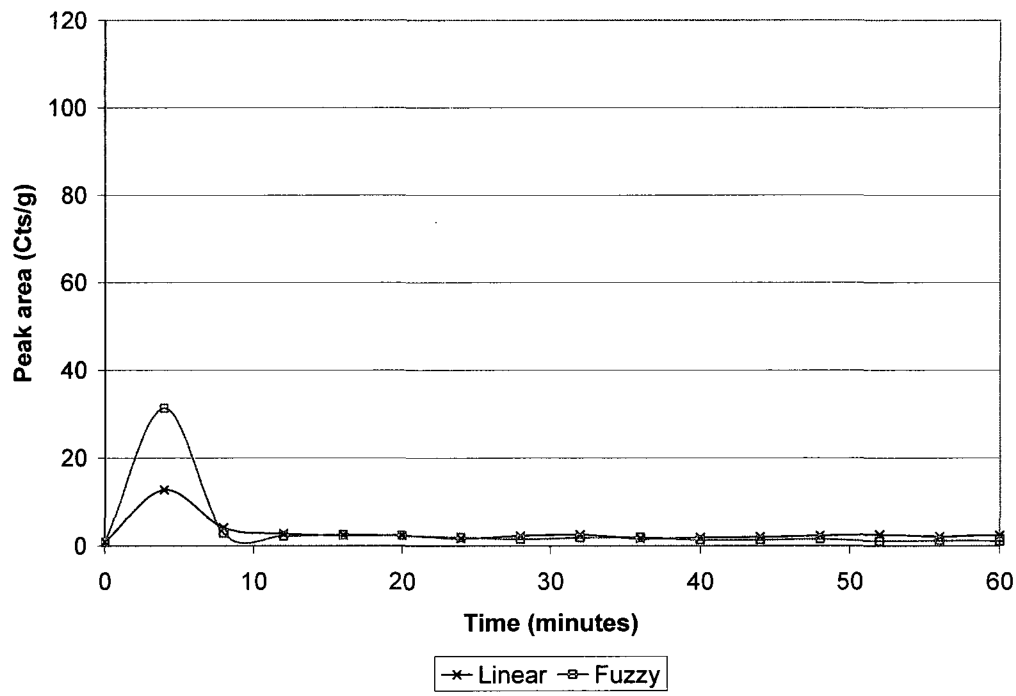

Fig. 7.8a Carrot $Peak_1$ in fuzzy and linear controlled drying processes

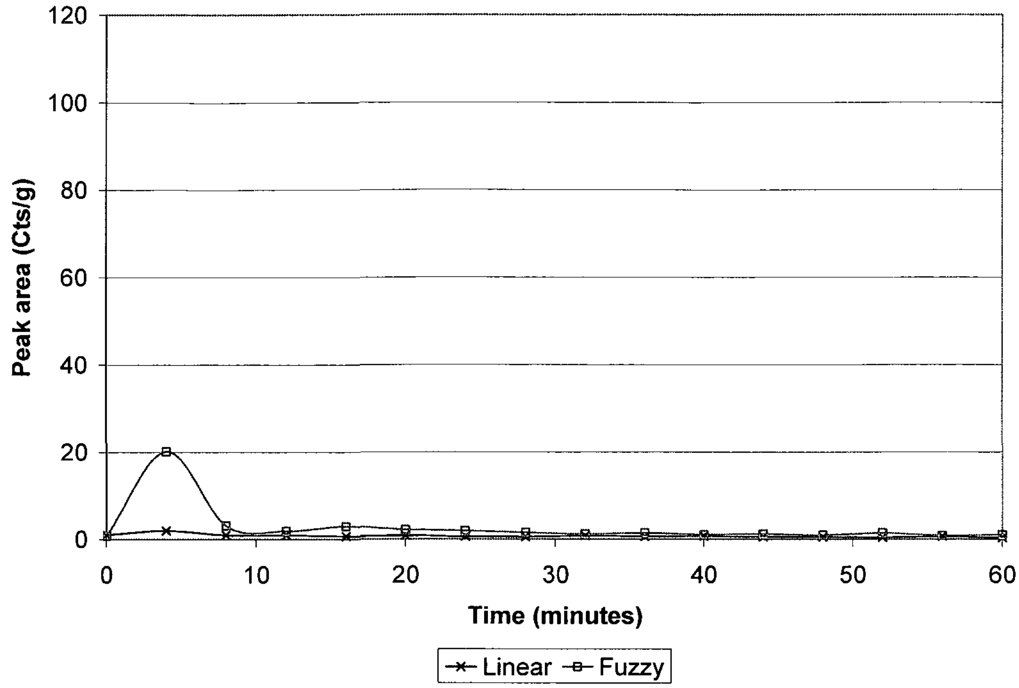

Fig. 7.8b Carrot $Peak_2$ in fuzzy and linear controlled drying processes

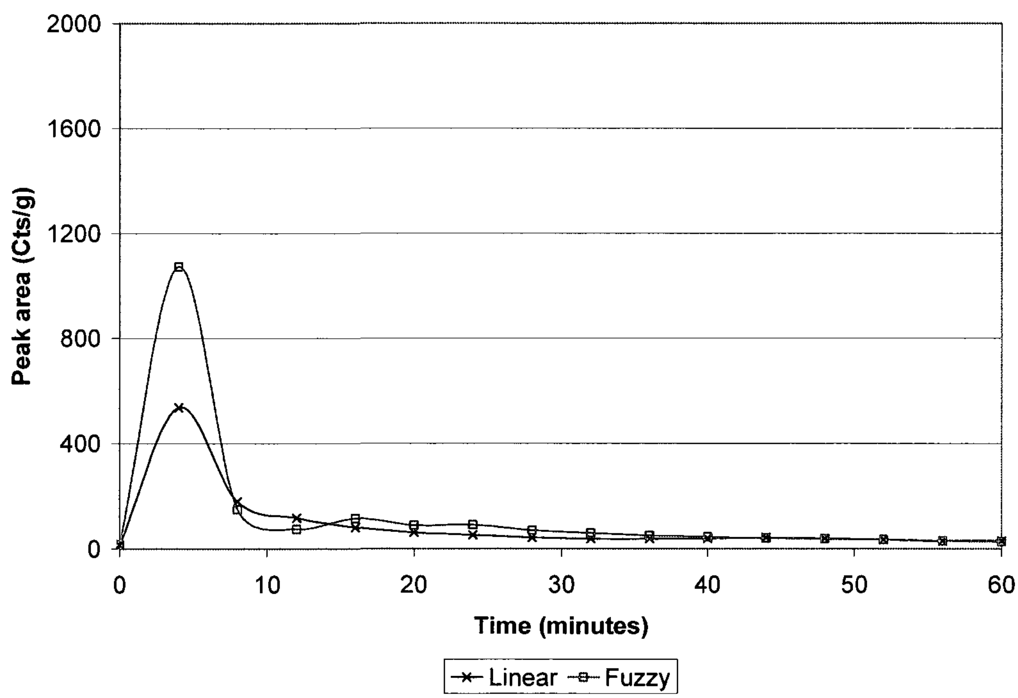

Fig. 7.8c Carrot $Peak_3$ in fuzzy and linear controlled drying processes

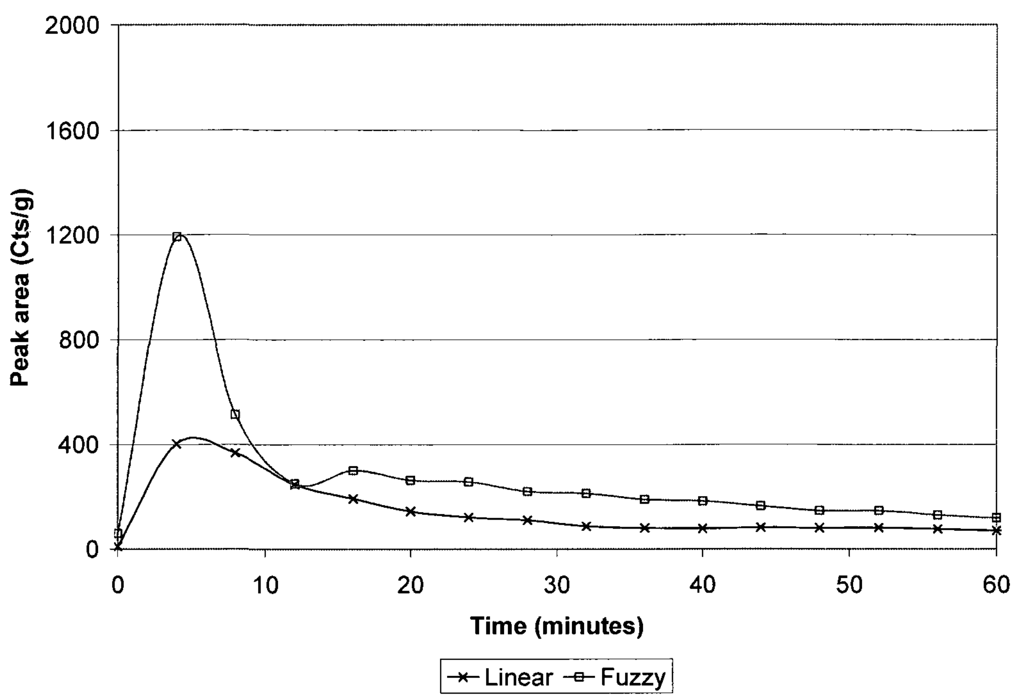

Fig. 7.8d Carrot $Peak_4$ in fuzzy and linear controlled drying processes

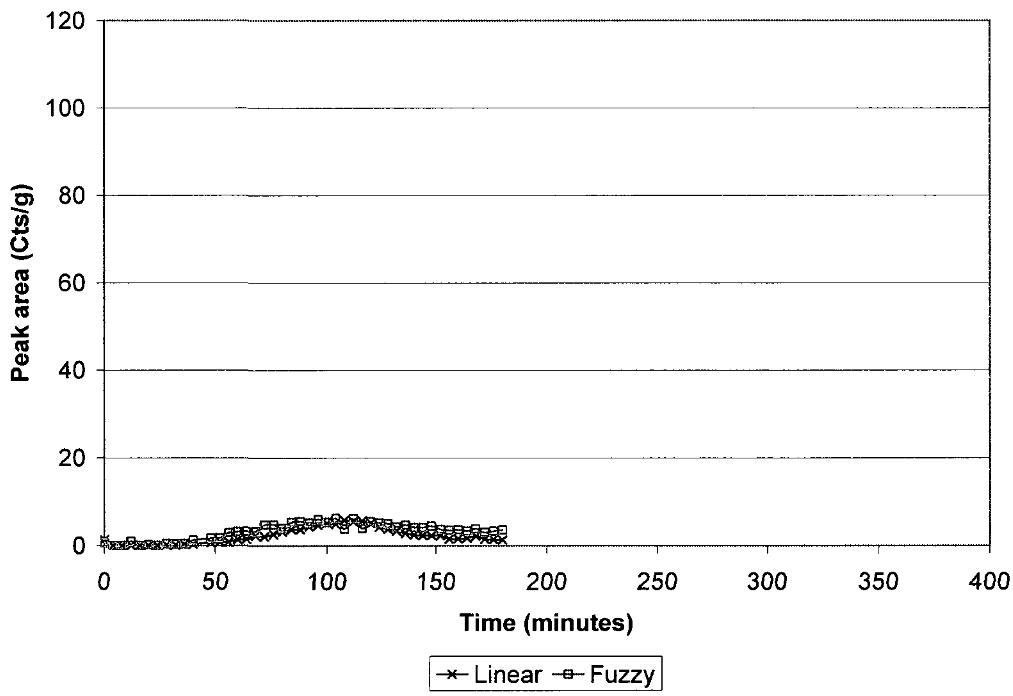

Fig. 7.8e Carrot 'burn' $Peak_5$ in fuzzy and linear controlled drying processes

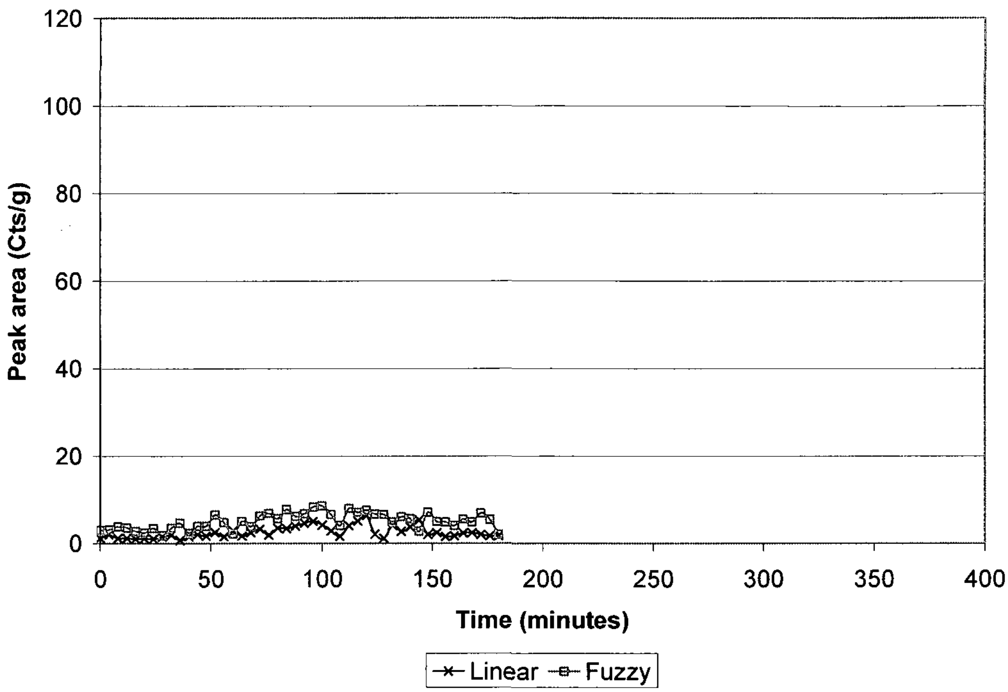

Fig. 7.8f Carrot 'burn' $Peak_6$ in fuzzy and linear controlled drying processes

Fig. 7. 8 a-f Carrot $Peaks_{1\text{-}6}$ in fuzzy and linear controlled drying process

Aroma peaks for fuzzy and linear controlled drying are shown in Fig. 7.8a-f, and were much smaller than those under fixed temperature drying shown in Fig. 7.3a-f. This indicates that these two control methods were effective in terms of aroma retention. Burning suppression was also obvious with these new control strategies, where linear control performed better than fuzzy control.

7.4.2.4 Moisture contents

Moisture contents under fuzzy and linear controlled drying show that drying rates under fuzzy logic control were a little lower than under linear control, and both curves were much smoother than under fixed temperature drying (Fig. 7.9).

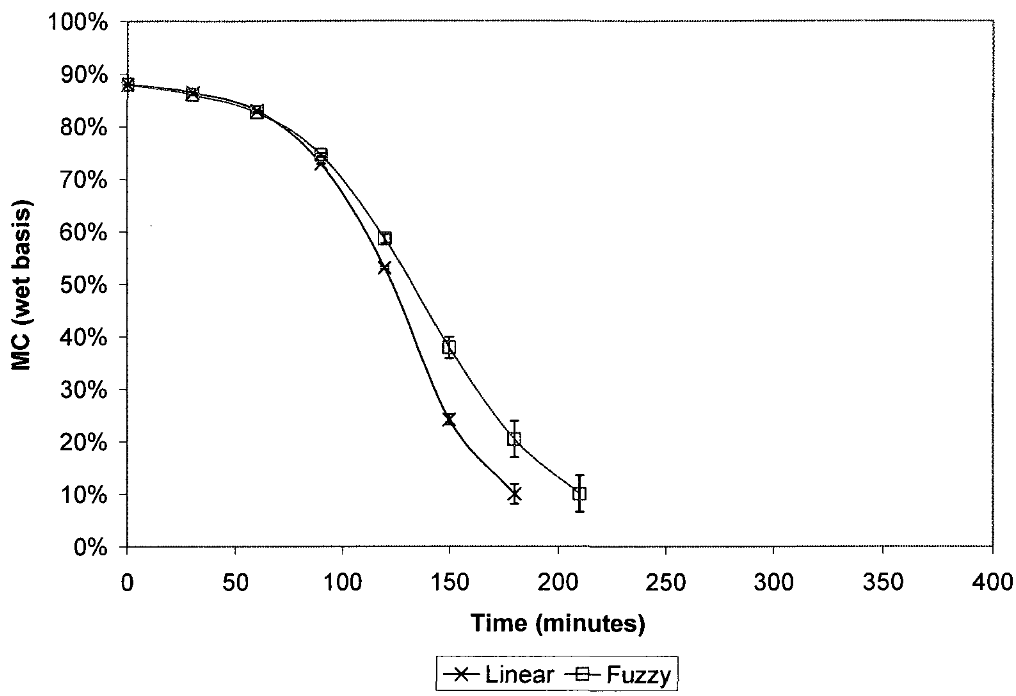

Fig. 7. 9 Moisture contents in fuzzy and linear controlled drying
(Error bars represent standard deviations)

7.4.2.5 Energy consumed

Energy consumed under fuzzy and linear controlled drying is shown in Fig. 7.10 with the results of fixed temperature drying included to facilitate comparison. Fuzzy control consumed almost the same as 60°C drying, while energy consumption of linear control was between that of 70°C and 80°C. It appears that linear control is an energy efficient drying method and even better than fuzzy control.

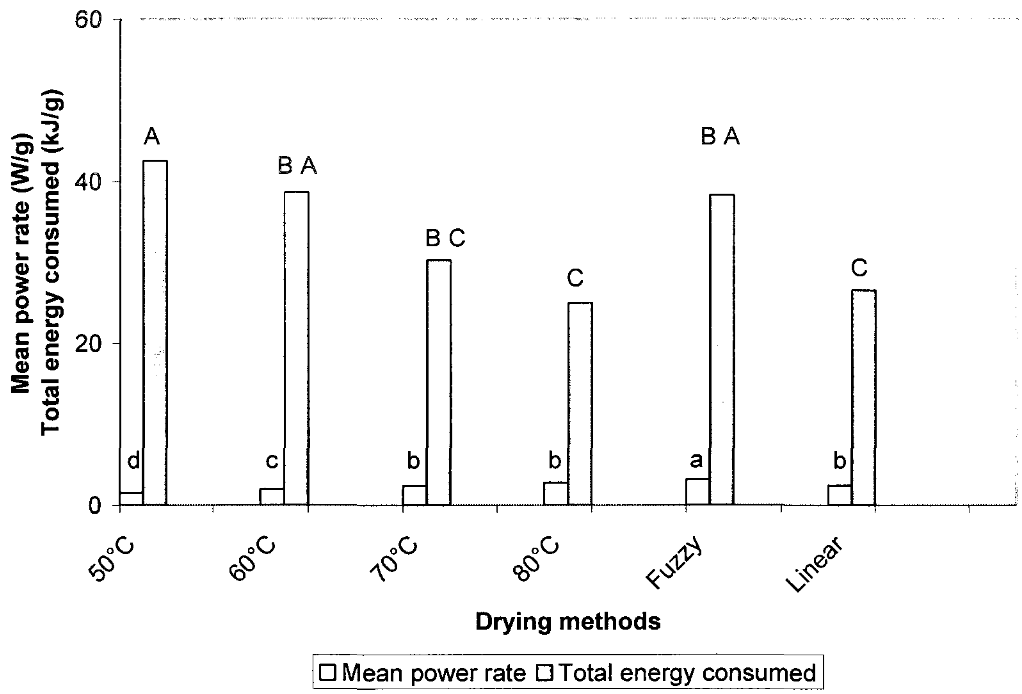

Fig. 7. 10 Energy consumed
(Mean values, values with different letter are significantly different, $\alpha = 0.05$, letters in lower case: mean power rate; letters in uppercase: total energy consumed)

7.4.3 Quality evaluation

7.4.3.1 Color character

Color of the final product is an important quality aspect. The closer it is to the fresh carrot, the better it is preferred. As the fresh carrot was orange with an a/b ratio of "1", a higher a/b value of the rehydrated products would represent more redness and be considered of better quality (Changrue, 2006). On this basis, the best color was achieved at a constant temperature of 60°C, followed by fuzzy logic controlled drying (Fig. 7.11).

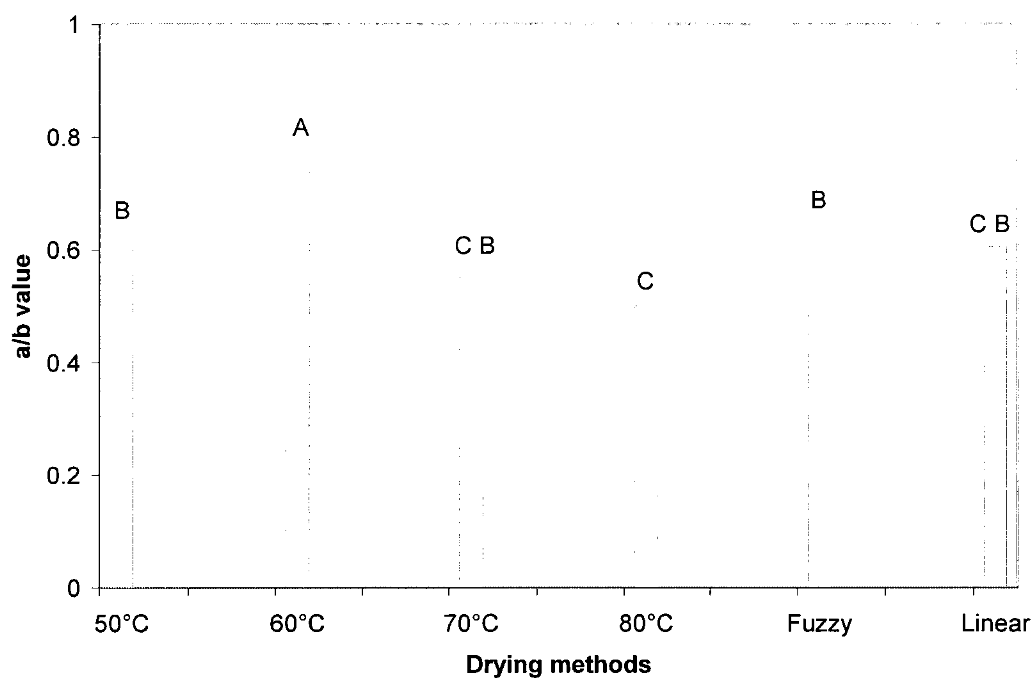

Fig. 7. 11 Redness of rehydrated carrots
(Mean values, values with different letter are significantly different, $\alpha = 0.05$)

7.4.3.2 Aroma measured with zNose

The aroma profiles of rehydrated samples are shown with baselines increased (Fig. 7.12) to facilitate visibility. The top profile is a measurement of fresh carrot cubes and the rest are rehydrated samples. It is obvious that fresh carrots had a much stronger aroma than processed samples. This indicates that during the drying process a large volume of aroma was lost.

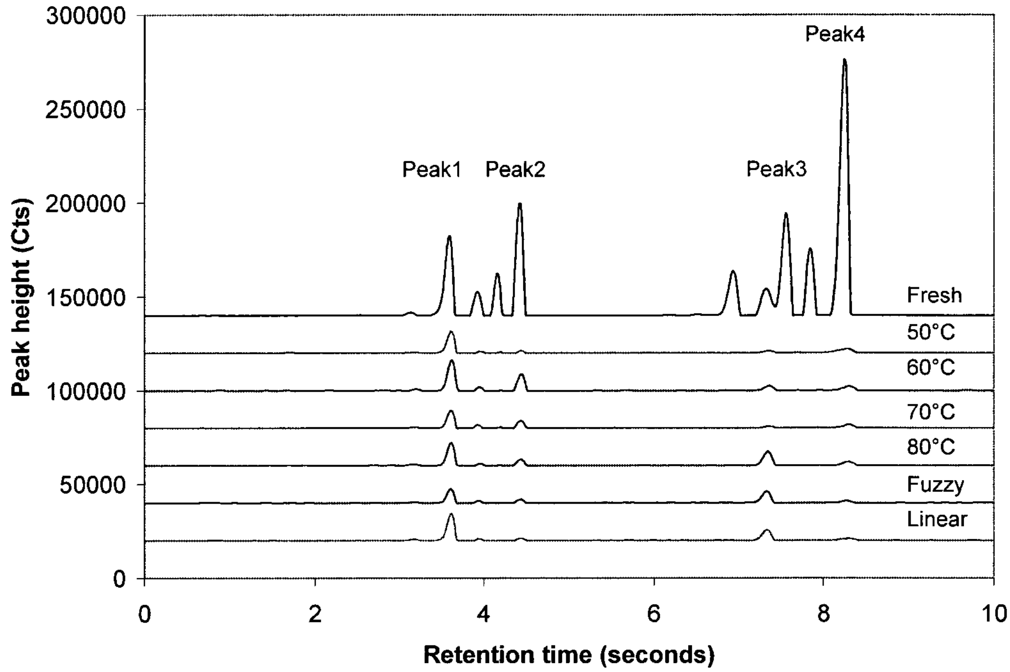

Fig. 7. 12 zNose measured aroma profiles of rehydrated carrots

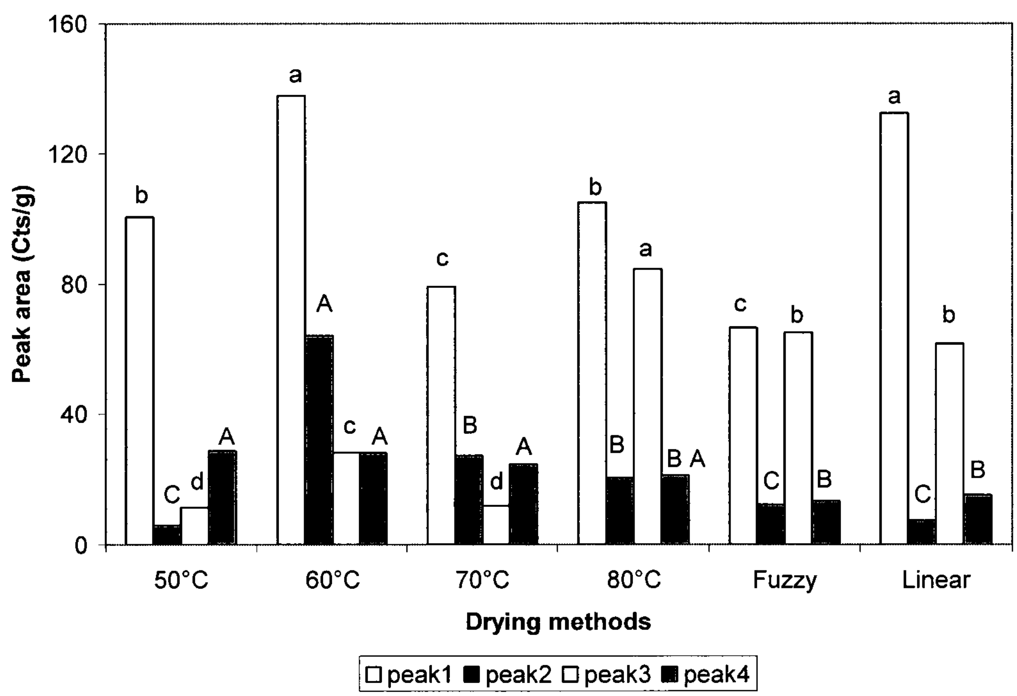

Fig. 7. 13 Four aroma peaks measured in rehydrated carrots
(Mean values, values with different letter are significantly different, $\alpha = 0.05$)

The four "carrot peaks" of rehydrated carrot samples having undergone drying by different methods are shown in Fig. 7.13. Burning peaks could not be detected with zNose after rehydration. Different peaks behave differently and it is hard to judge their aroma quality with these values. Hence, a taste panel assessment became necessary for flavour quality evaluation.

7.4.3.3 Taste panel assessment

Taste panel assessment of the products having undergone dehydration and rehydration revealed that best result was also achieved by 60°C drying method for both appearance and taste (Fig. 7.14), followed by the fuzzy control method.

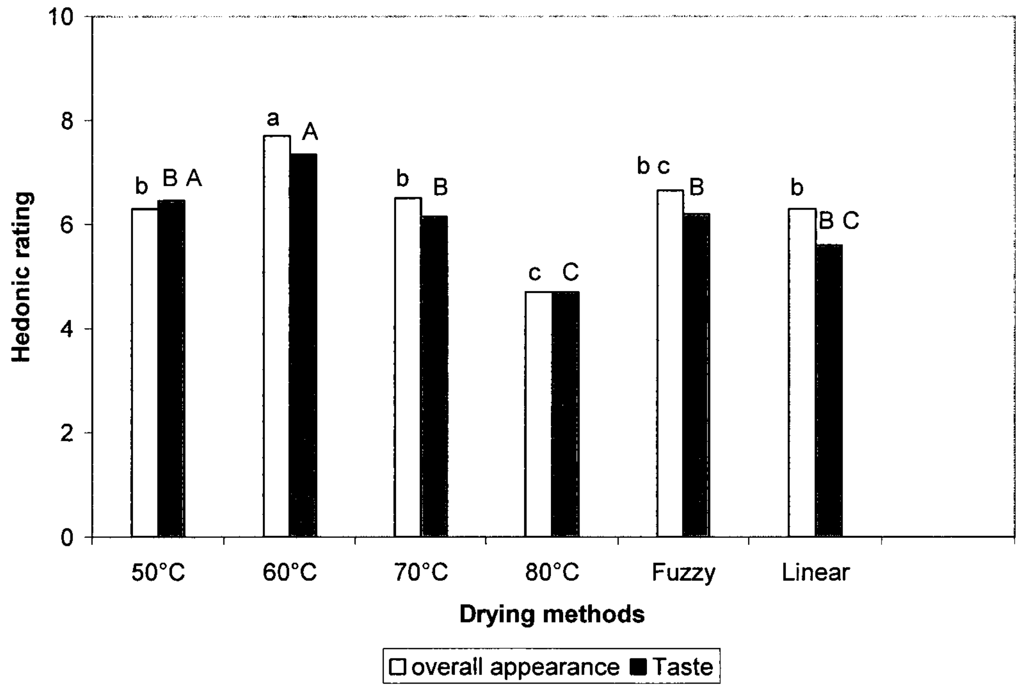

Fig. 7. 14 Overall appearance and taste
(Mean values, values with different letter are significantly different, $\alpha = 0.05$, letters in lower case: overall appearance; letters in uppercase: taste)

Concluding all the quality evaluation, a surprising phenomenon was observed: the lowest drying temperature (50°C) did not result in the best quality. The longer drying time at 50°C might have been responsible for its poor quality.

The quality of carrots having undergone fuzzy and linear control were comparable to 50°C and 70°C fixed temperatures, better than 80°C, but worse than the best quality obtained in 60°C drying. Considering time and energy efficiency, fuzzy and linear controls were still acceptable drying control methodologies.

Many studies have investigated the effects of microwave drying on dried carrot's final products quality. Lin *et al.* (1998) compared vacuum microwave drying of carrot slices to air drying and freeze drying on the basis of rehydration potential, color, density, nutritional value, and texture properties. Changrue (2006) conducted microwave vacuum drying of carrot cubes in different power modes, with prior osmotic treatment, and compared drying effects on rehydration properties, color, and sensory evaluation. However, no study has reported on aroma variations during carrot drying. The current study is an important contribution to this field and also gives practical ways to improve product quality with sophisticated control strategies.

7.5 Conclusions

Intensive aroma loss occurred during the early stages of carrot drying. In contrast, burning of carrot samples happened in the middle of drying process. Much more aroma was lost at the lowest drying temperatures due to the longest drying time, and quality of carrots having undergone high temperature drying were unacceptable due to the burned flavour. Best product quality was achieved at a fixed drying temperature of 60°C. Drying at higher temperatures cost less time and energy, although the mean power rate was higher than that at lower temperatures.

Results showed a fixed temperature cannot achieve best drying effect when carrot aroma, burning occurrence, time and energy efficiency were all considered. Fuzzy and linear control methods could retain aroma and reduce drying time as well as energy consumption. Fuzzy control needed online detection of aroma as variables, while linear method did not. Linear control is a simplification of fuzzy control. Acceptable product quality was also achieved with fuzzy and linear control in terms of color, taste, and overall appearance.

7.6 Acknowledgements

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CONNECTING STATEMENT 5

In the previous chapter, carrot was chosen as a representative of vegetables for aroma study. A similar study was conducted on apple as a representative of fruits and is presented in this chapter. However, a different linear control method was tested to prove that different foodstuffs need different control strategies.

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Apple Aroma Monitoring and Control During Microwave Drying

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Contributions made by different authors are as follows:

The first author, the PhD student did the experimental work and prepared the manuscript; the second and third authors are the supervisors who guided the research work.

CHAPTER VIII

APPLE AROMA MONITORING AND CONTROL DURING MICROWAVE DRYING

8.1 Abstract

Apple aroma was monitored online with a zNose and controlled based on levels of aroma constituents during microwave drying. Apples were first dried at fixed temperatures of 60°C, 70°C, 80°C, and 90°C. Four typical aroma peaks were recorded every 4 minutes for all the three replicates. Samples could be dried in a shorter time at high temperatures but were more susceptible to burning. A fuzzy logic controller was then designed and employed to control the drying processes. The controller used apple aroma and burning signals as variables and tried to retain apple aroma, avoid burning, shorten drying time, and save energy. Based on the temperature profile achieved under fuzzy logic control, a simple linear control method was developed where zNose online assistance could be omitted. Color and sensory evaluation by an untrained taste panel were performed after rehydration. With the new control strategies, apple color, aroma, and overall appearance were intact and less time and power were consumed.

Keywords: Apple, aroma, zNose, microwave drying, fuzzy control

8.2 Introduction

Since the early studies of Powers and Chestnut (1920; 1922) more than 300 volatile compounds have been identified as aroma substances from various cultivars of apples (Dimick and Hoskin, 1983; Crouzet, 1986). Within these numerous compounds, only a few, between 20-40 compounds, have been determined to have a decisive impact on the sensory quality (Cunningham *et al.*, 1986). Despite exhaustive studies of apple fruit aroma, knowledge of aroma variations during the drying process is not abundant. Krokida and Philippopoulos (2006) recently analyzed by GC-MS three representative components of Delicious Red apple during air and freeze drying. Timoumi *et al.* (2007) dried Red Chief apples with an IR moisture analyzer and compared the aroma loss after different drying conditions. However, these analyses are all restricted to off line detection of apple aroma and no online measurement has been reported up to present. Although the offline aroma detection can provide some useful information about aroma variation, it is inconvenient and time-consuming. Moreover, it is impossible to provide control parameters to optimize drying conditions in real time and *in situ* to improve aroma retention with offline measurement.

The first step in accomplishing an online measurement and control is a rapid aroma detection. Most current instruments, either electronic noses or conventional analytical chemistry equipments, are time-consuming and complex (Li *et al.,* 2006), which make it almost impossible to detect food aroma instantly. Consequently, no literature is so far available regarding online detection of food aroma. Fortunately, a new aroma detection instrument called a zNose^{TM} (7100 Fast GC Analyzer, Electronic Sensor Technology, New Bury Park, CA, USA) has recently become available. This new instrument can detect food aroma as fast as in seconds, which for the first time makes online detection possible. However, the application of the zNose has remained in offline detection (Lammertyn *et al.*, 2004; Gan *et al.*, 2004) and no online employment is reported.

Microwave drying is considered time and energy efficient. On one hand, many advantages of this technique have been cited (Orsat *et al.,* 2006; Venkatesh and Raghavan, 2004; Vadivambal and Jayas, 2007); however, on the other hand, just as with other food processing methods, no online aroma evaluation has been hitherto reported. How the aroma is altered and how to improve the microwave drying conditions to retain more favourable aroma is still unknown. Moreover, the problem of non-uniformity of heating which may result in partial and interior burning of products with high sugar content is still a critical challenge to microwave researchers.

The objectives of this study were:

1) To investigate apple aroma variations during microwave drying processes;
2) To improve microwave drying in terms of aroma retention with new control strategies;
3) To compare drying effects, energy consumption, and product quality for apples having undergone different microwave drying methods.

8.3 Materials and Methods

8.3.1 Sample preparation

Granny Smith apples were purchased from a local market and stored at 4°C. Initial moisture content was determined by drying fresh apple cubes at 70°C for 24 hours in a hot air oven. Peeled apple cubes of 10×10×10 mm (20±1g) were blanched in 80°C water for 1 minute before drying. All samples were dried to 10% moisture content (wet basis).

8.3.2 Aroma monitoring and control system

An aroma monitoring and control system was designed for microwave drying (Fig. 8.1). The system consisted of an aroma detection unit, a microwave drying system, a temperature and power control system, and a PC based data acquisition unit (DAQ). The aroma detection unit included a zNose, a two-position pneumatic valve, an air flow installation, a moisture condenser, and a flow meter; the microwave drying system included a domestic microwave oven, a Teflon container, and an electrical scale; the temperature and power control system included an optical fiber sensor, a control circuit, a 12 VDC and an 110VAC power supplies; the data acquisition unit was a PCI 6014 multifunction device (National Instruments, TX, USA) including analog outputs to facilitate the MW power control.

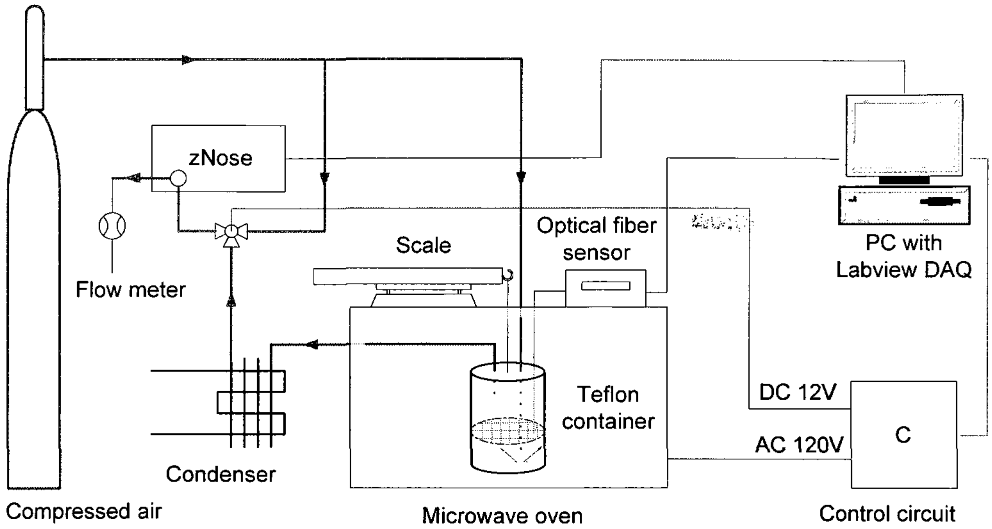

Fig. 8. 1 Schematic diagram of the aroma monitoring and control system

8.3.2.1 Aroma detection unit

The zNose is actually a fast GC. It can detect aroma in as little as 1 minute and automatically repeat the measurement procedures. Detailed description of the instrument can be found elsewhere (Lammertyn *et al.*, 2004). Just as a regular GC, detected volatiles are expressed as retention times and peak areas, which represented different compounds and their quantities, respectively. The temperature of the sensor was set to 60°C and sampling time was 6 seconds.

A two-position pneumatic valve (M223W1DFR-HT-312, Teqcom, CA, USA) was installed in front of the zNose to alternately detect volatiles and clean air for detection of sample aroma and cleaning the zNose, respectively. Both detection and cleaning cycles were set to 2 minutes; hence sample aroma was measured every 4 minutes. Different peaks were assumed to represent different volatiles. By calibrating with standard chemicals (n-Alkanes), zNose converted detected peaks' retention times to a Kovats index (Appendix C). Hence, chemical compounds could be tentatively identified by checking a library of related volatile compounds.

Compressed air was used to carry the vapour from the Teflon container and microwave oven and bring it to the zNose. Air pressure was adjusted to 10 psi with a regulator and flow rate was controlled at 1000 mL min^{-1} with an adjustable flow meter. Moisture was collected in an air-cooled condenser just after the microwave oven and before the zNose. Hence, only dry volatiles were allowed to reach the zNose.

All tubes and connectors were made of Teflon so as not to retain any aroma and to facilitate microwave drying. The air-cooled condenser made of 6 slim glass bottles was carefully cleaned after each experiment.

8.3.2.2 Microwave drying system

A 600 W domestic microwave oven (Beaumark 02314, Matsushita Electric Ind. Co. Ltd., Yamatokoriyama, Japan, 1986) was used. Stirrer and fan were modified to be controlled separately and were fully on during the whole drying process. Power supply to the magnetron was phase controlled by the DAQ and a control circuit.

Apples were dried in a small porous basket inside a Teflon container, which could hold apple samples and allow air to flow through. Apple cubes were carefully separated from each other to avoid microwave arcing. Mass of the container with samples was measured with an electronic scale (PK 4801, Denver Instrument, Denver, CO, USA) and read visually. An optical fibre sensor (Nortech Fibronic Inc., Quebec Canada) was inserted in the center of one of the sample cubes for temperature measurement.

8.3.2.3 Temperature and power control system

There were two main parts to the temperature and power control system: a phase controller to adjust the power level, which could automatically and continuously adjust AC power according to the temperature requirements, and a relay and its driver for on/off control of the pneumatic valve. The low voltage part of the circuit, which connected to the DAQ and PC, was protected with optical-electrical coupling devices.

8.3.2.4 Data acquisition unit

A PC used in this study housed the Labview program (Labview 7.1, National Instruments, TX, USA) and had a Multifunction Data Acquisition device built-in (PCI 6014, National Instruments, TX, USA). The PCI 6014 is a 16-bit resolution DAQ board with 250 kS/s rate and includes 2 analog outputs, 8 digital I/O lines, and two 24-bit counters. Three ports of the DAQ were used in this study: an analog input for the optical fiber temperature sensor, a DC output to control the two-position pneumatic valve, and an analog output to adjust AC power to the magnetron. All the controls were implemented in the Labview environment. The PC also read signals with RS-232 port from zNose, processed the data within the Labview program, and used them in the control processes.

8.3.3 Experiment design

8.3.3.1 Apple drying at fixed temperatures

Apple cubes were first dried at four fixed temperatures: 60°C, 70°C, 80°C, and 90°C. Aroma signals from each run were processed and analyzed using the Labview program. Important peaks were identified online and recorded during the drying process.

Temperatures and power rates were automatically recorded every second by the PC. By contrast, mass was read and recorded manually and moisture contents were calculated.

8.3.3.2 Control strategies for MW drying

Based on the observation and analysis of aroma variation under fixed temperature drying, a fuzzy logic controller was designed. The purpose of the fuzzy logic control was to decrease aroma loss, avoid the occurrence of burning, and increase the drying rate. Peaks related to aroma and burning were used as input variables, and the output was a temperature adjustment parameter. Fuzzy rules were based on Table 8.1.

Table 8. 1 Fuzzy rule base for apple aroma control

Burn \ Carrot	Low	Medium	High	Very high
Low	P	Z	N+	N+
Medium	N	N	N+	N+
High	N+	N+	N+	N+
Very high	N+	N+	N+	N+

* P: positive, Z: zero, N: negative, N+: more negative

The functions of input variables were triangles. Fuzzy inference was Max – Min. The defuzzification method was center of gravity, and the output function was singleton. Input variables were restricted to two in order to reduce the complexity of the fuzzy controller and also make the fuzzy rule practical and clear.

Based on the fuzzy logic control method, a linear control was further designed to make it possible to improve apple drying without the zNose. A simple temperature profile was developed to simulate the temperature profile resulting from fuzzy logic control. All parameters were recorded as in the previous section.

8.3.4 Quality evaluation

Quality parameters of rehydrated apples were evaluated and compared, since in most cases dehydrated products will be consumed in their rehydrated forms. The rehydration procedure was conducted according to USDA standards (Anon, 1944). Distilled water (150 mL) was first brought to boiling point in a 500 ml beaker, allowed to boil for 3 minutes, 5g of dried samples were added and these were boiled for another 5 minutes. After draining water with filter paper, the surface color was measured with a colorimeter (Model CR-300X, Minolta camera Co. Ltd., Japan). Each measurement of the colorimeter provided three values: L*, a*, b*, representing lightness, a purple-red and blue-green coefficient, and a yellow-blue coefficient, respectively. For rehydrated apple samples, lightness was found to be an important index because burning often resulted in a darker product. Hence, the L* value is presented and compared in this study.

Sensory evaluation in terms of overall appearance and taste was then conducted by a taste panel of ten untrained judges. The judges were asked to indicate their preference for each sample based on the color, appearance, and overall acceptance, and then taste its flavour. A balanced 9-points hedonic rating was employed, where 9 denoted "like extremely" and 1 indicated "dislike extremely" (Resurreccion, 1998; Lin *et al.*, 1998).

8.3.5 Statistical analysis

All experiments were conducted with three replicates and mean values were reported. Error bars of standard deviations were included for moisture contents. SAS software (SAS 9.1, SAS Institute Inc, Cary, NC, USA) was used to perform the ANOVA procedure for power consumption, lightness, overall appearance and taste. The Duncan's Multiple Range Test was carried out for each of the above factors. Typical standard deviations of aroma signals are listed in Appendix F.

8.4 Results and Discussion

Preset and actual drying temperatures, drying times, detected aroma profiles, variations of moisture contents, mean power rates, total power consumed, and quality evaluation are reported in this section for both fixed temperature drying and drying with new control strategies.

8.4.1 Apple drying at fixed temperatures

Apple was first dried at four fixed temperatures: 60°C, 70°C, 80°C, and 90°C. Temperatures and drying time were recorded, as well as moisture contents and power rate.

8.4.1.1 Temperatures and drying times

Apple temperatures of 60°C, 70°C, 80°C, and 90°C were successfully controlled with appropriate power levels. The preset temperatures were reached in a short time (1-2 minutes) from initial ambient temperature. The drying time for 90°C, 80°C, 70°C, and 60°C were around 70, 85, 100, 215 minutes, respectively (Fig. 8.2), indicating that, as expected, drying at low temperatures took longer.

The highest temperature was set to 90°C because beyond that serious burning occurred making the final product quality totally unacceptable. The lowest temperature was 60°C, as the product quality in terms of flavour deteriorated when a lower drying temperature was applied due to the longer drying time which led to more aroma loss.

Comparing the drying time with that under convective drying and freeze drying, which was 7-9 hours and 20 hours respectively (Krokida and Philippopoulos, 2006), it is again clear that microwave drying is a time-efficient drying approach.

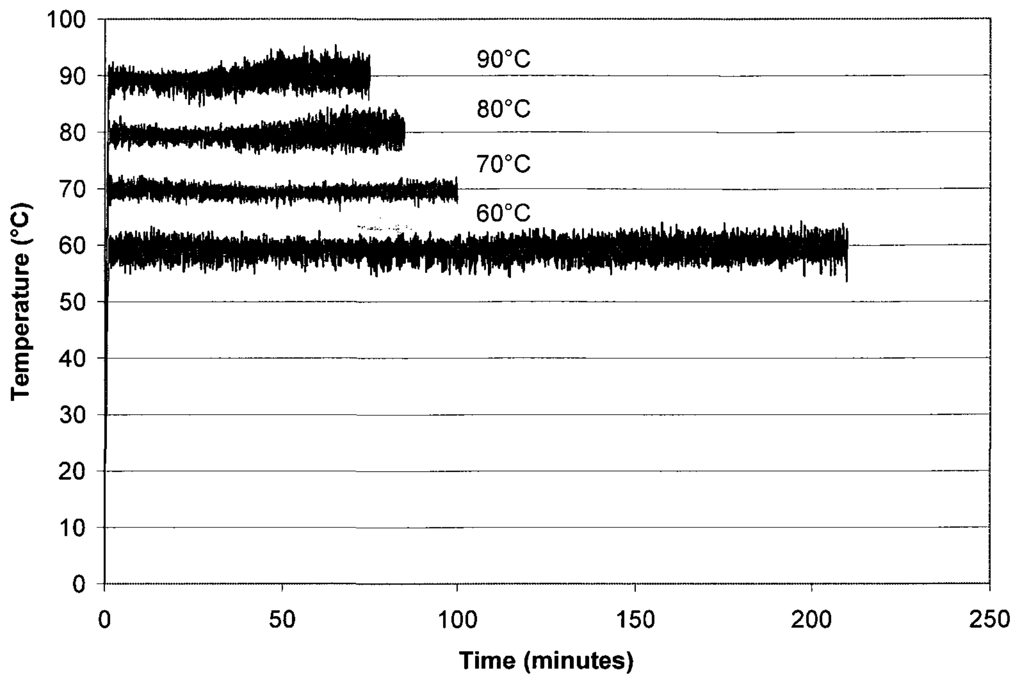

Fig. 8. 2 Apple drying at fixed temperatures

8.4.1.2 Aroma profiles

Four significant peaks were detected with zNose during microwave drying of apple. By comparing their Kovats index with literature (Lopez *et al.*, 1998), $Peak_1$ was identified as 2-heptanol, but $Peak_2$ was unknown. These two peaks were also detected when the aroma of fresh apples was measured. Hence, they could be considered as representative of natural apple aroma and will be termed "apple peaks" in this study. $Peak_3$ and $Peak_4$ only appeared when apples were burned and will be termed as "burning peaks."

All peaks arose during the middle drying stages (Fig. 8.3a-d). An increase in temperature has an adverse effect on the retention of apple peaks ($Peak_1$ and $Peak_2$), whereas the burning peaks $Peak_3$ and $Peak_4$, were increased. This indicated that low temperature was preferable in terms of aroma retention and to avoid burning. However, this conclusion conflicted with that of the final quality evaluation (see section 8.4.3).

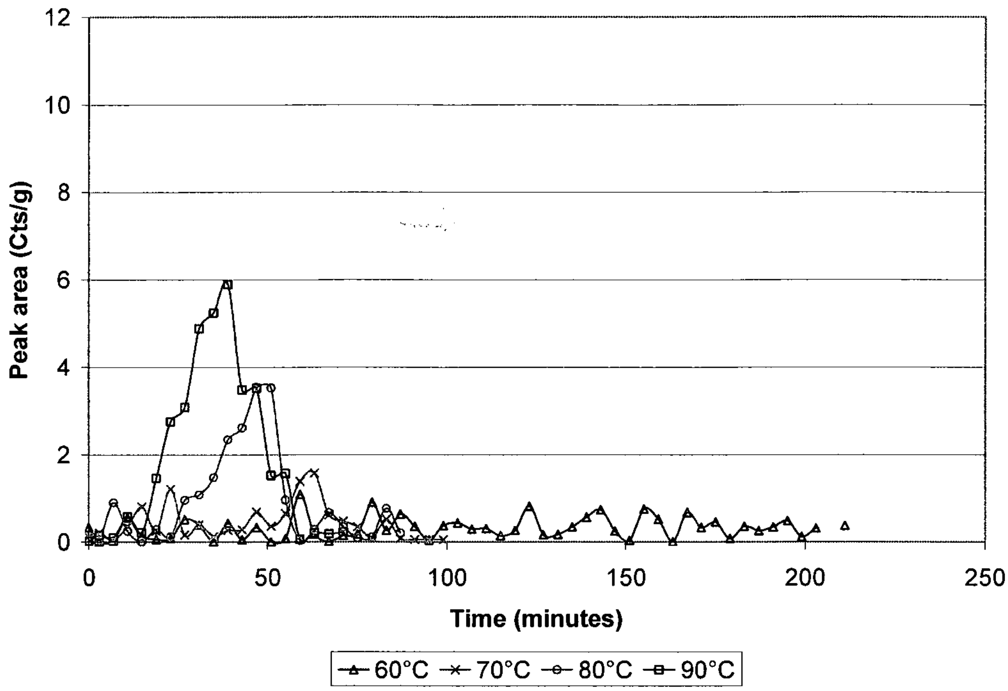

Fig. 8.3a Apple $Peak_1$ under microwave drying at fixed temperatures

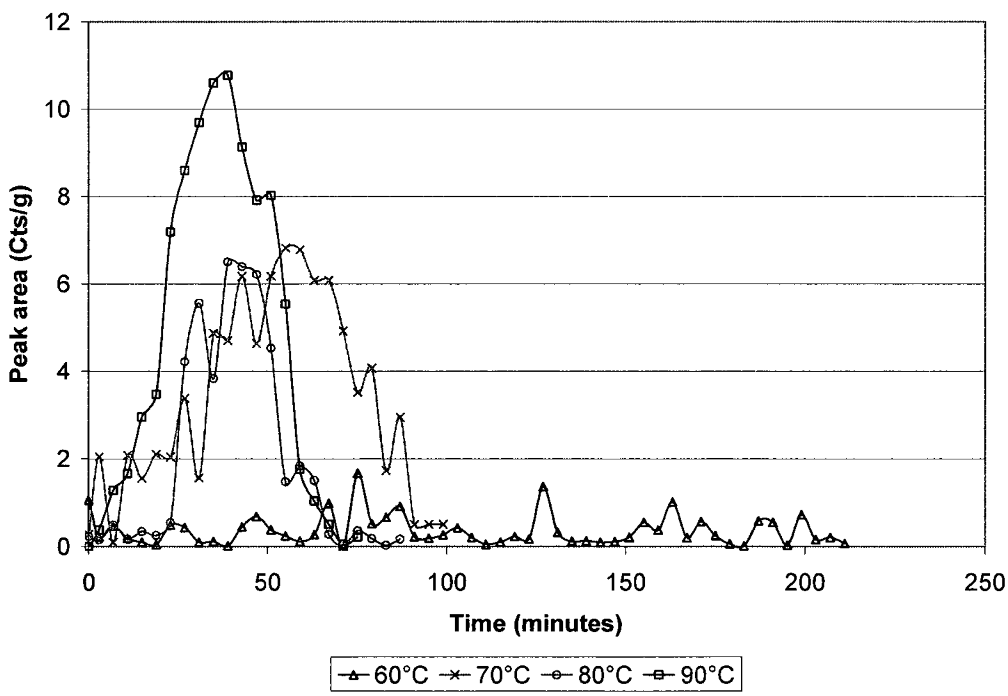

Fig. 8.3b Apple $Peak_2$ under microwave drying at fixed temperatures

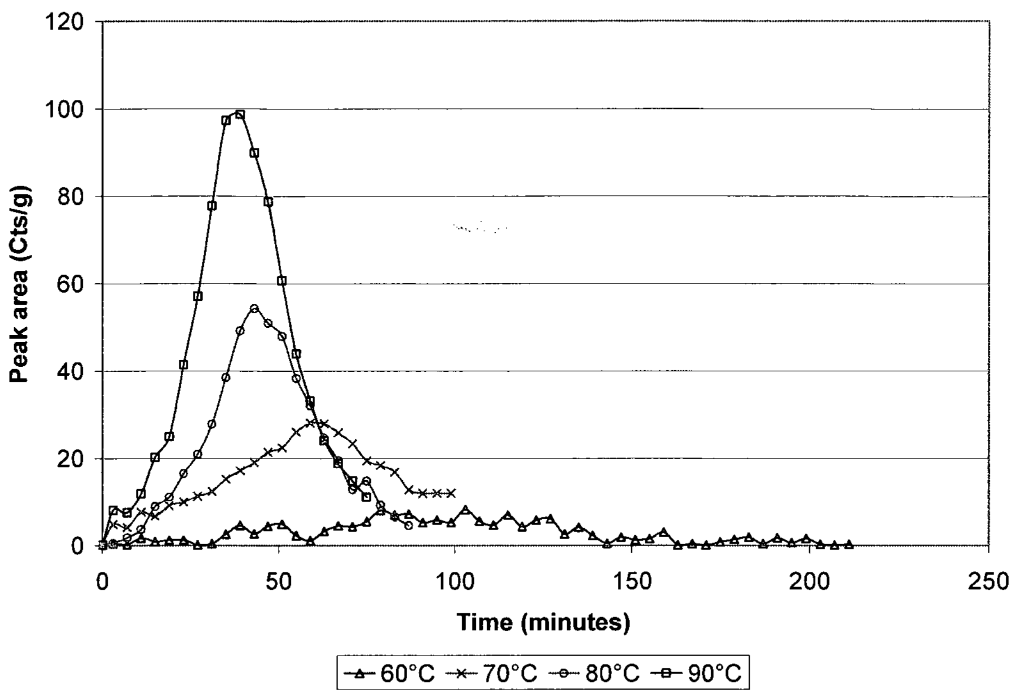

Fig. 8.3c Burn Peak$_3$ under microwave drying at fixed temperatures

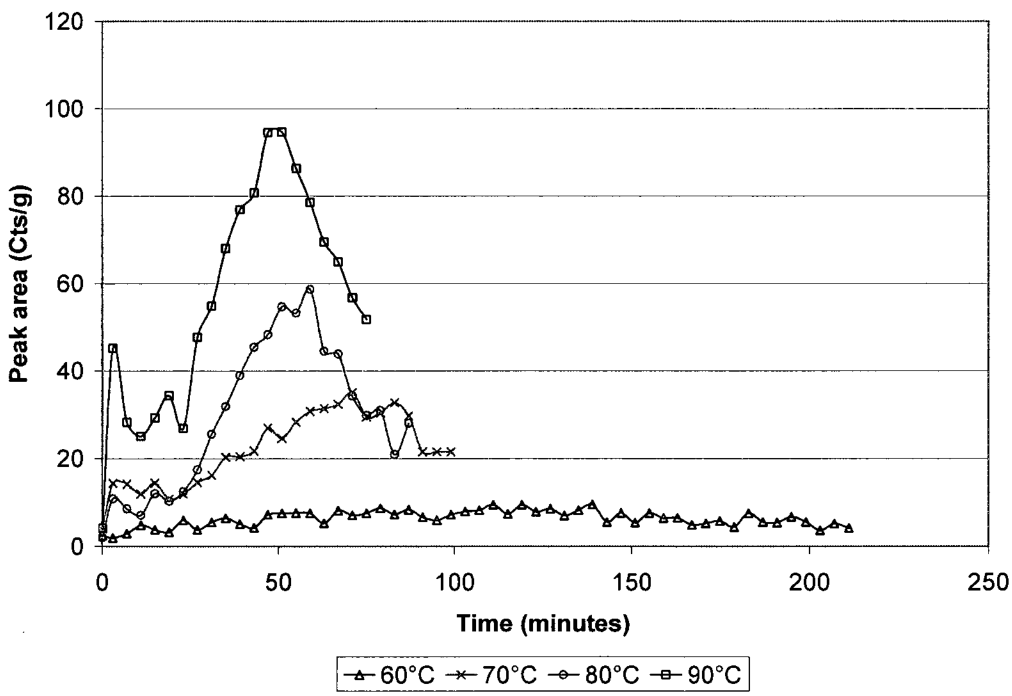

Fig. 8.3d Burn Peak$_4$ under microwave drying at fixed temperatures

Fig. 8. 3 a-d Apple Peaks$_{1\text{-}4}$ under microwave drying at fixed temperatures

8.4.1.3 Moisture contents

The initial moisture content of apples was 85.17%. The curves of moisture contents at four fixed drying temperatures show that drying rates increased with an increase in drying temperature and that the fastest drying rate was achieved at 90°C (Fig. 8.4). It was noticed that the drying curves were not smooth during the middle drying stages; this might be attributable to moisture condensation in the Teflon container. However, the moisture was completely removed in the last stages of drying and did not influence the final mass measurement.

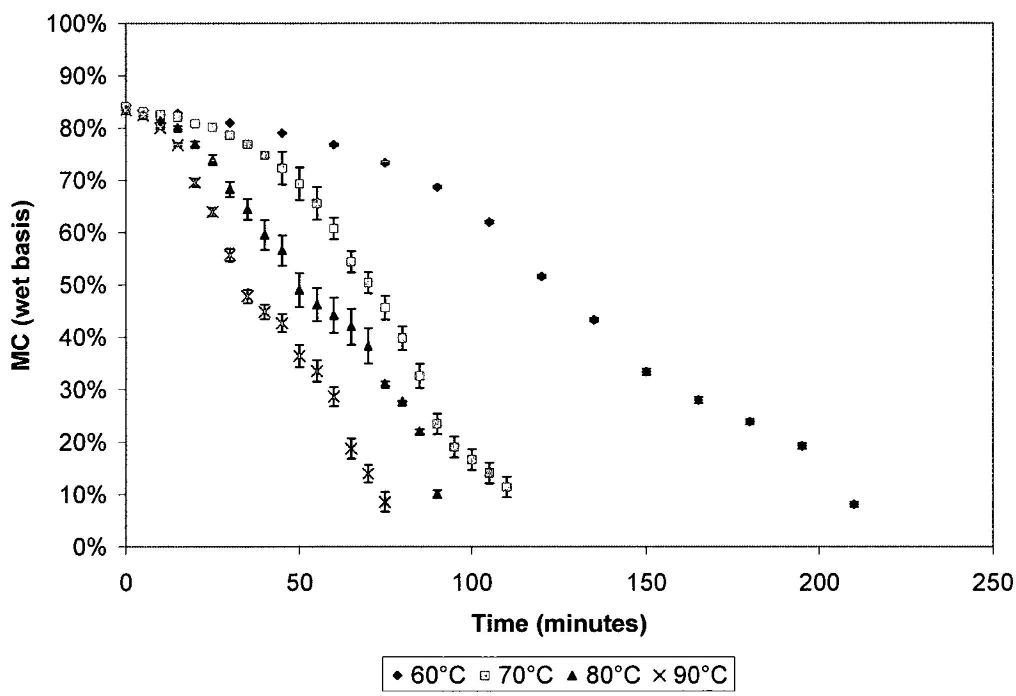

Fig. 8. 4 Moisture contents at fixed temperatures
(Error bars represent standard deviations)

8.4.1.4 Energy consumed

Power rates applied to the magnetron were automatically adjusted and recorded. Under high temperature drying, more power was required. Appropriate power levels were carefully selected to be just enough to maintain the temperature requirements but not enough to overheat the magnetron.

By summing the power rate over time a total amount of consumed energy was calculated. Fig. 8.5 presents power rate and total energy consumed per gram of apple. It is worth emphasizing that although the power rate applied at high temperatures was high, the total consumed energy was low due to the shorter drying time. Hence, high temperature drying was preferred in terms of time and energy efficiency, and this requirement was different than that of aroma retention.

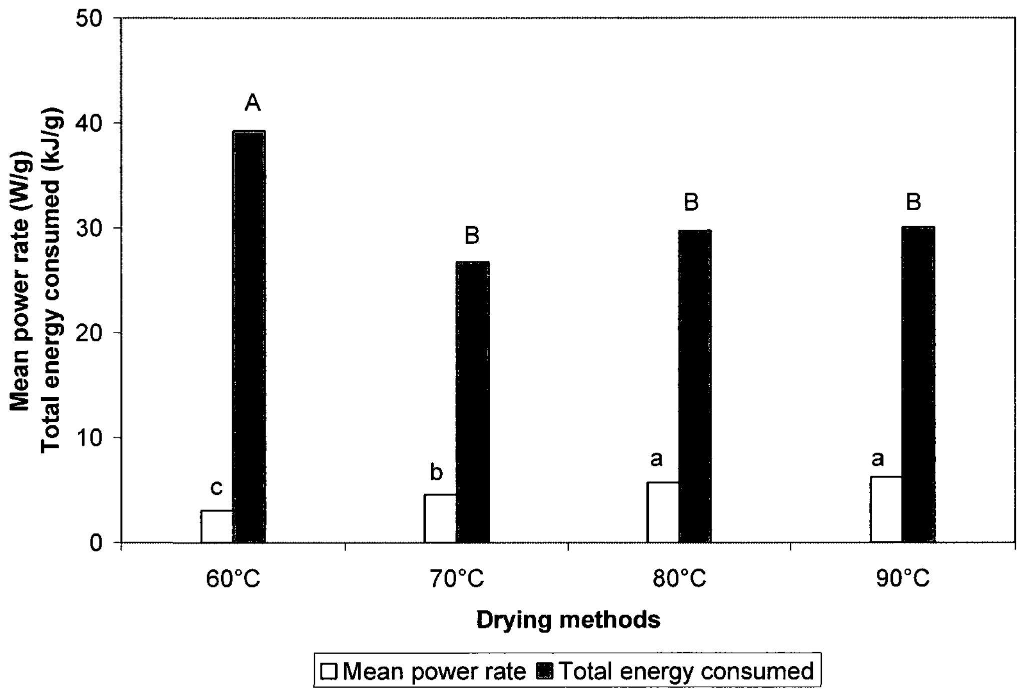

Fig. 8. 5 Energy consumed when drying at fixed temperatures (Mean values, values with different letter are significantly different, $\alpha = 0.05$, letters in lower case: mean power rate; letters in uppercase: total energy consumed)

Overall, with respect to drying time, energy consumed, and aroma variations at four fixed temperatures, it was noted that at higher temperatures drying times were shorter, energy consumed was less, but more aroma was lost and more burning occurred. The phenomenon inspired one to investigate a possibility of developing more effective drying methods to create a compromise between aroma retention, avoiding burns, time and energy consumption in a single drying process.

8.4.2 Fuzzy logic and linear control strategies

Apple was dried using fuzzy and linear controlled methods. Temperatures and drying time were recorded, as well as moisture contents and power rate.

8.4.2.1 Fuzzy control

Analysis of the four peaks recorded in the previous experiments showed that $Peak_2$ had larger value than $Peak_1$. Also $Peak_1$ and $Peak_2$ had similar trend in variation. Hence, $Peak_2$ was chosen as a representative of "apple peaks," while Peak3 was chosen as a representative of "burning peaks" because $Peak_4$, as a large retention time (RT) peak, was difficult to clear from the zNose system which may result in inaccurate control.

As a result, $Peak_2$ and $Peak_3$ were used as the two input variables of the fuzzy logic controller. Output of the fuzzy controller was a temperature between -10°C and +5°C. As this output was preset at a temperature of 70°C, the fuzzy controlled temperature could be varied between 60°C and 75°C during the drying process, depending on aroma variations. The highest temperature was limited to 75°C because at 80°C and above more burning occurred. The lowest temperature was set to 60ºC because at this temperature Peaks1-4 were sufficiently low. The fuzzy-controlled temperature profile is presented in Fig. 8.6. All three replicates revealed similar curves with little fluctuations (the other two are not shown). The actual temperatures followed the fuzzy set temperature rapidly, indicating efficient power control in the whole control process. Total drying time was around 120 minutes.

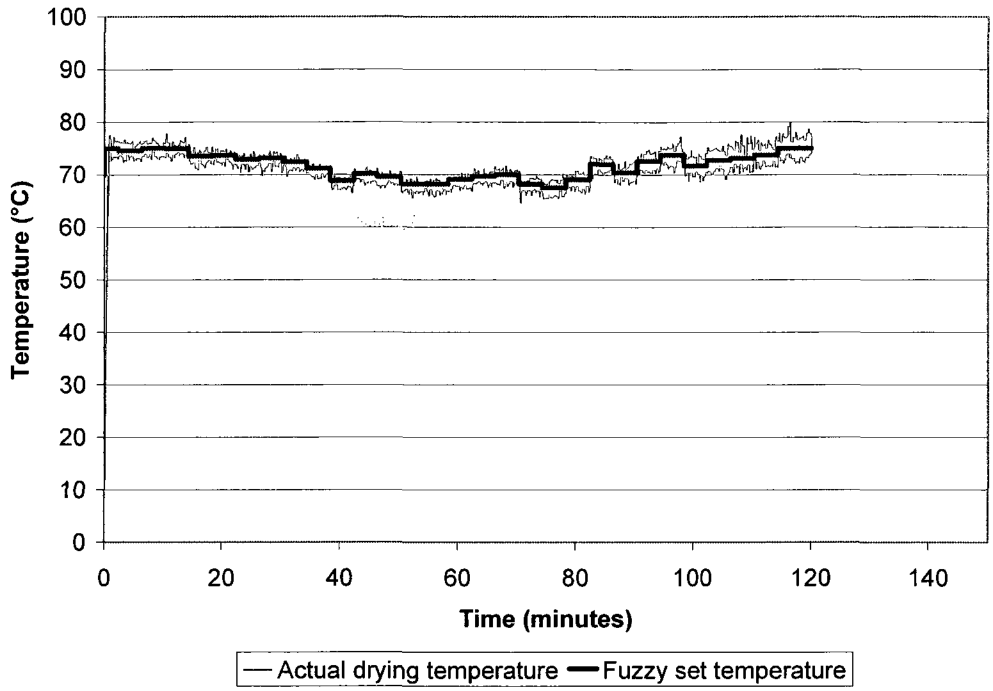

Fig. 8. 6 Apple cube temperature during fuzzy-controlled drying

8.4.2.2 Linear control

Observing the temperature curve under fuzzy logic control, while ignoring minor fluctuations, it is showed that according to aroma control requirements, the temperature should be decreased in the early stages from 75°C to 65°C and then increased to 75°C gradually. Considering that less mass leads to less aroma loss during the last drying stages, the drying temperature was best kept at a constant 65°C during the final stages. Thus, a linear controller was developed. This method simply decreased the temperature from an initial 75°C to 65°C in exactly 60 minutes and then kept the temperature at 65°C until the end of drying.

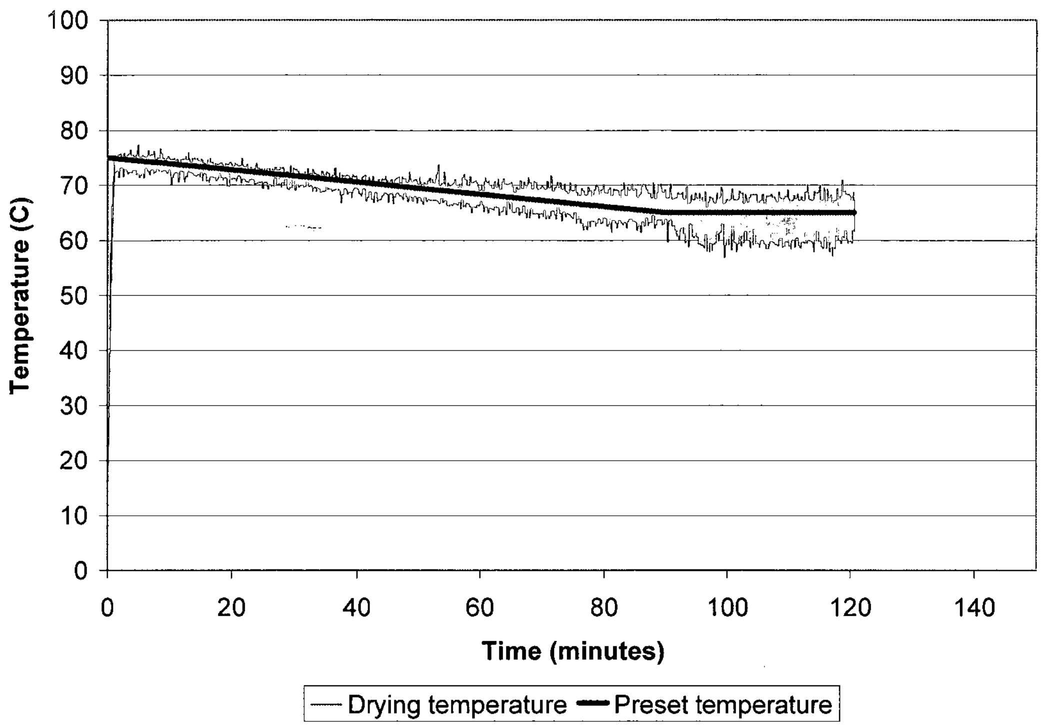

Fig. 8. 7 Temperature of apple cubes under linear-control drying

The linear-control temperature curve (Fig. 8.7) shows a total drying time of 120 minutes, almost the same as for fuzzy control drying. Another benefit of the linear control method is that it does not need the assistance of zNose and can stand alone. As zNose is a costly instrument, the linear control is a more practical and cost-efficient drying method. However, this relation has to be established first for different products.

8.4.2.3 Aroma profiles

Aroma peaks under fuzzy and linear controlled drying are compared in Fig. 8.8a-d. All values were much lower than those under fixed temperature drying (Fig. 8.3a-d), indicating that these two control strategies are effective in terms of aroma retention and to avoid burning.

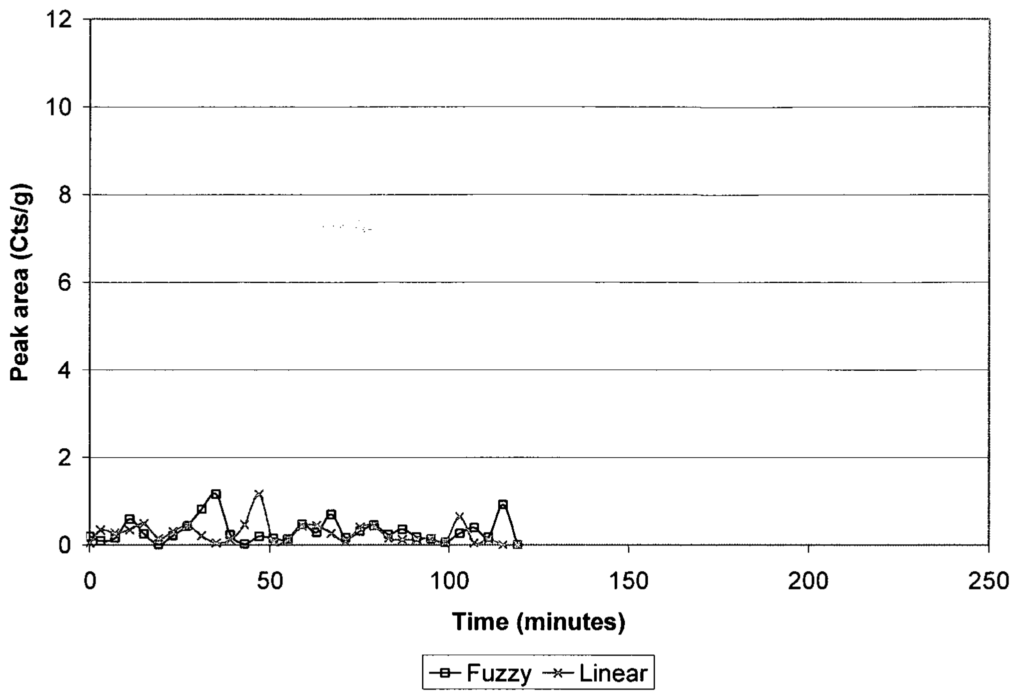

Fig. 8.8a Apple $Peak_1$ under fuzzy and linear controlled drying processes

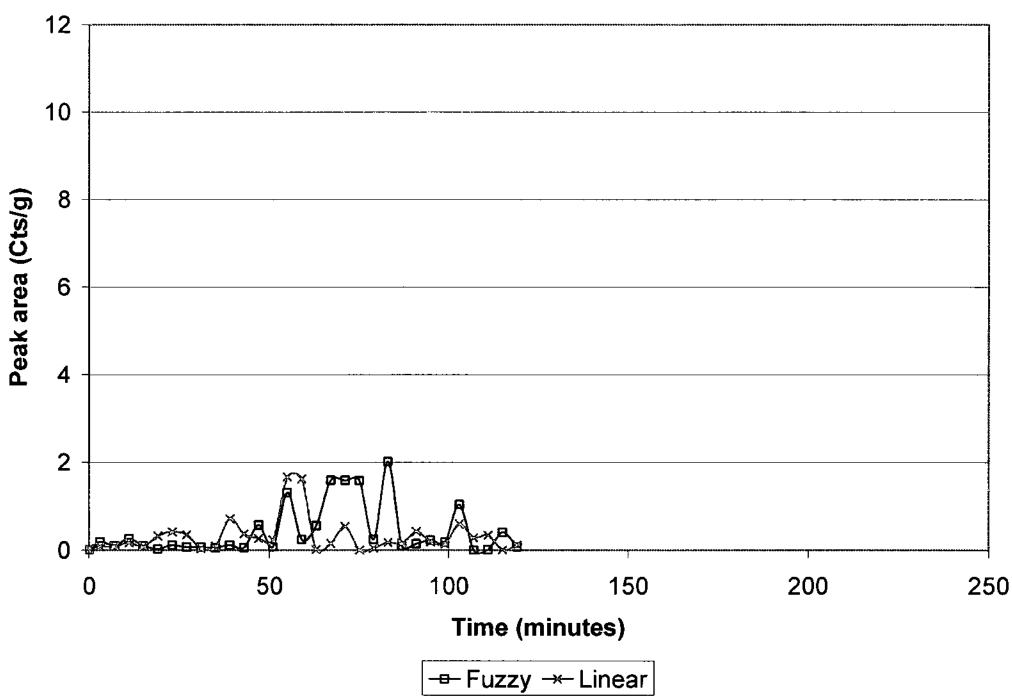

Fig. 8.8b Apple $Peak_2$ under fuzzy and linear controlled drying processes

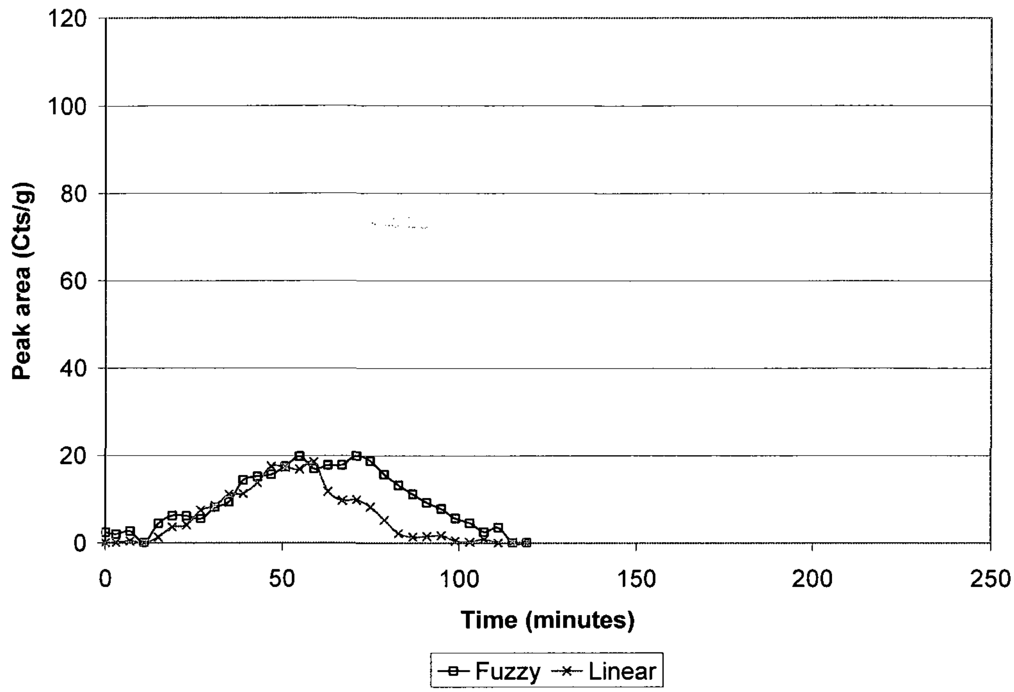

Fig. 8.8c Burning Peak$_3$ under fuzzy and linear controlled drying processes

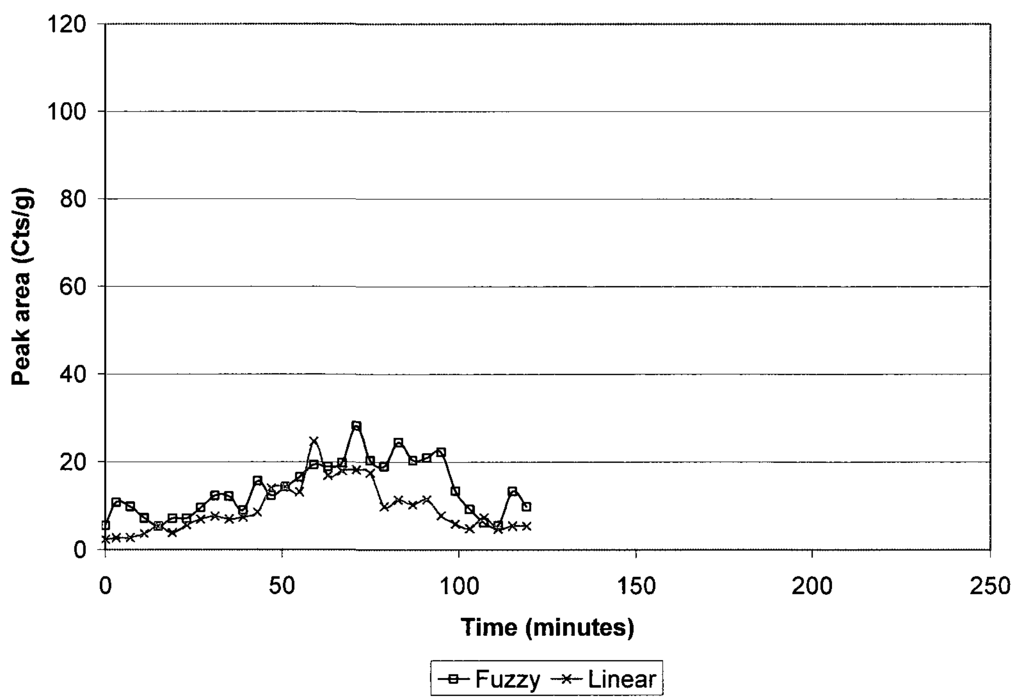

Fig. 8.8d Burning Peak$_4$ under fuzzy and linear controlled drying processes

Fig. 8. 8 a-d Apple Peaks$_{1-4}$ in fuzzy and linear controlled drying process

8.4.2.4 Moisture contents

Plots of moisture contents under fuzzy and linear controlled drying (Fig. 8.9) showed drying rates under these two methods to be roughly the same, as were the drying time costs.

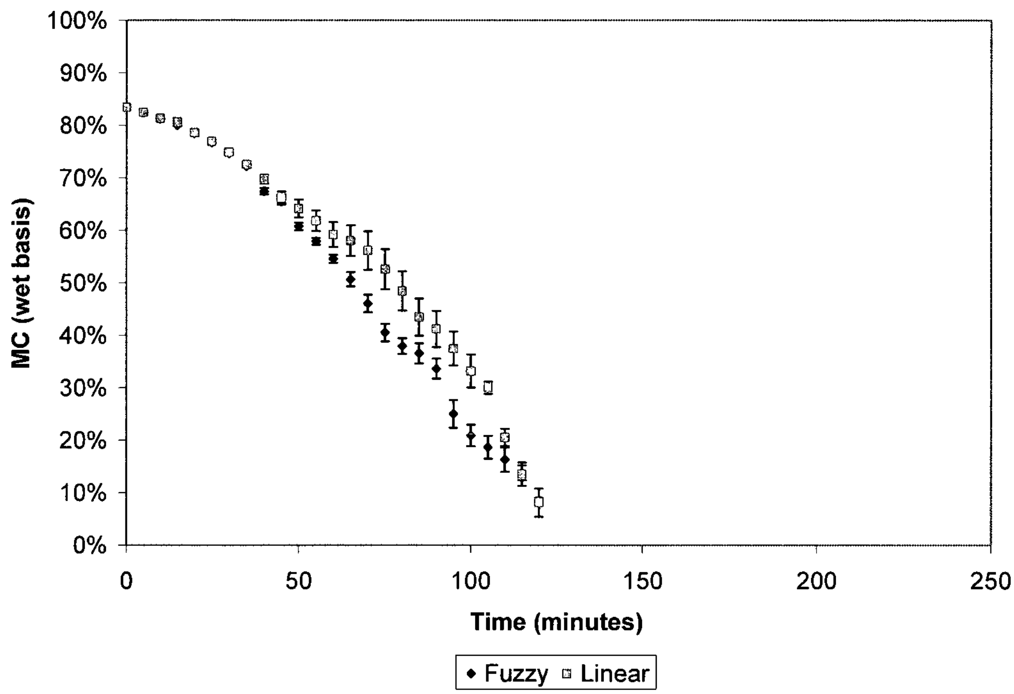

Fig. 8. 9 Moisture contents of apples under fuzzy and linear controlled drying (Error bars represent standard deviations)

8.4.2.5 Energy consumed

The mean power rate and total energy consumed under fuzzy and linear controlled drying are compared in Fig. 8.10, with values for fixed temperature drying included to facilitate comparison. At 60°C, drying requires the greatest energy because of the long drying time. However, the remaining drying methods, either at fixed temperatures or in fuzzy and linear controls, showed little difference despite their different mean power rates.

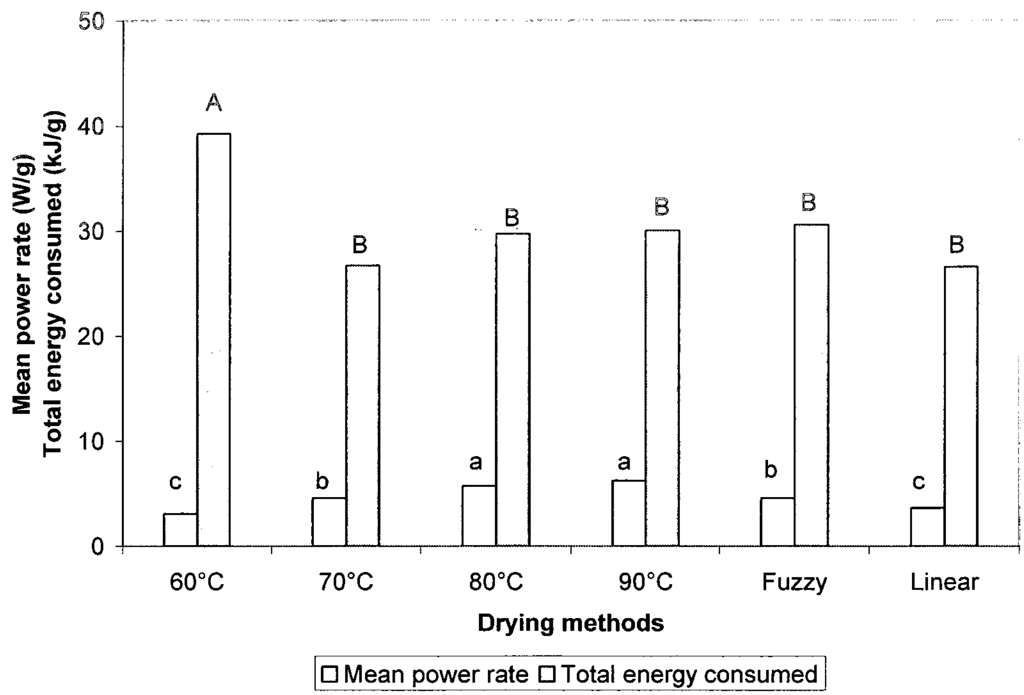

Fig. 8. 10 Energy consumed in drying apple cubes
(Mean values, values with different letters are significantly different, $\alpha = 0.05$, letters in lower case: mean power rate; letters in uppercase: total energy consumed)

8.4.3 Quality evaluation

8.4.3.1 Color characteristics

Color of the final product is an important quality aspect. When burning occurred during the drying process it usually resulted in a dark color and led to an unacceptable flavour. Hence, a lighter product represented a better quality.

The best color was achieved under fuzzy control drying and the worst color occurred under 90°C drying, followed by 80°C drying (Fig. 8.11). Drying at 60°C, 70°C, and linear control drying resulted in similar color quality. However, Duncan's analysis shows no statistical significance among lightness values. This might contribute to similar exterior color appearance despite serious interior burning occurred during high temperature drying.

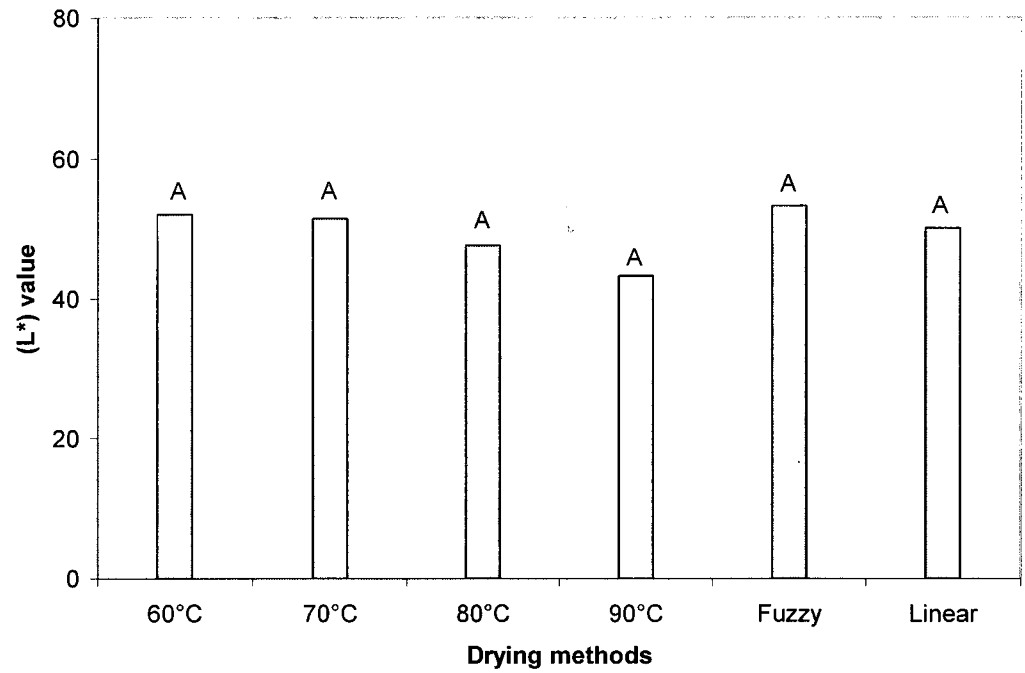

Fig. 8. 11 Lightness of rehydrated apples
(Mean values, values with different letters are significantly different, $\alpha = 0.05$)

8.4.3.2 Sensory evaluation

Sensory evaluation of the rehydrated products was carried out to obtain preliminary information on consumer preference. The results revealed that the best quality was achieved under fuzzy logic control drying and the worst under 90°C drying (Fig. 8.12). The bitter flavour of 90°C dried apples was responsible for their unacceptability.

Considering the quality evaluation for the four fixed temperature drying protocols, one noticed that the best product quality was not achieved at the lowest temperature (60°C), but the highest drying temperature did result in the worst quality. This can be explained as the longest drying time caused the most aroma loss. Hence, long duration drying should be avoided in terms of aroma retention. Acceptable product quality was also achieved in fuzzy and linear control drying.

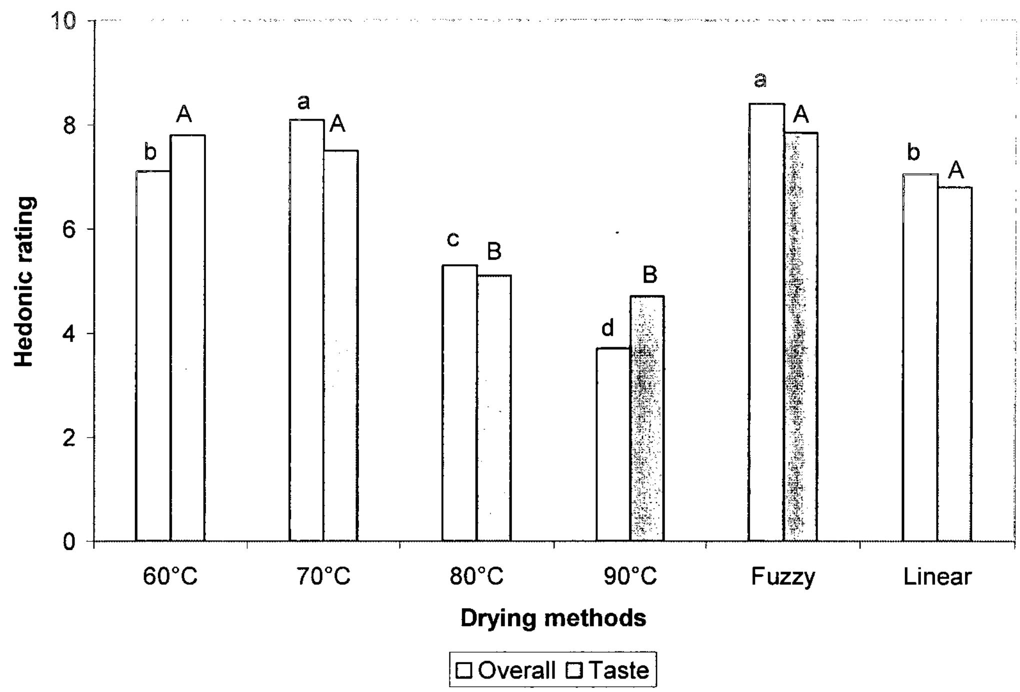

Fig. 8. 12 Overall appearances and taste of rehydrated microwave-dried apples (Mean values, values with different letter are significantly different, $\alpha = 0.05$, letters in lower case: overall appearance; letters in uppercase: taste)

Krokida and Philippopoulos (2006) analyzed some representative aroma compounds during the freezing and convective drying of red delicious apples. They identified 36 substances by GC/MS and examined three representative components (*ethyl acetate*, *ethyl butyrate* and *methyl anthranilate*) in detail. A rapid loss of flavour compounds was observed under convective drying, whereas a good retention of volatile compounds occurred under freeze drying. Under convective drying, temperature increases were found to have an adverse effect on flavour compounds retention. This last observation is very similar to the trends in aroma variations observed in the current study. However, in this study, the lowest temperature treatment was not found to be the best in terms of aroma retention. Furthermore, the flavour detection of Krokida and Philippopoulos (2006) was offline and time intervals were very long (in hours), which might lead to a significant loss of flavour information between two adjacent measurements.

8.5 Conclusions

The highest drying temperature resulted in the worst product quality because of aroma loss and occurrence of burning. However, the best quality was not achieved at the lowest temperature due to aroma loss over the longer drying time. Instead, 70°C was the best drying temperature within the four fixed temperature drying methods. Drying at the lowest temperature cost the most time and energy, although the mean power rate was low. Hence, high and low temperatures should be avoided in microwave drying when aroma retention, burning avoidance, time and energy efficiency are all considered.

Based on these considerations, a fuzzy controller was developed. This new control strategy successfully retains aroma and avoids burning with acceptable time and energy consumption. A linear control method was further attempted to imitate the fuzzy control without the assistance of zNose. The control effect of linear control was comparable to that of fuzzy control.

8.6 Acknowledgements

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CHAPTER IX

GENERAL SUMMARY AND CONCLUSIONS

Passive and dynamic methods on food aroma measurements were studied in this thesis. Classification of Chinese spirits, ripeness and rot evaluation of mango fruits were conducted in passive aroma studies. Dynamic aroma study was implemented in microwave drying processes. A microwave drying system with combined control strategies was developed and used to improve the food quality in terms of aroma retention. Carrot and apple were dried as representatives of vegetables and fruits.

In the study of Chinese spirits, six Fenjiu samples were well classified by their headspace volatiles. The retention time and peak area were useful parameters for classification and prediction methods. Principal components analysis and canonical discriminant analysis were proven to be excellent tools for statistical analysis in spirit identification and classification. Measurement of dielectric properties was also proven to be a potential method for the prediction of alcohol concentration, serving as a supplement to the zNose in electronic sensory characteristics evaluation of Chinese spirits.

Aroma variations of mango fruits during their shelf life were investigated. Seven marked peaks in aroma profiles were identified and three were selected for rot prediction and ripeness evaluation. The rot prediction method developed has potential applications for the mango industry in terms of detecting side and stem end rots in an ultra fast way. The ripeness evaluation method developed can be used to judge the best consumable period for mango fruits.

A real time aroma monitoring and control system was developed and integrated into a microwave drying process. Aroma was detected with the zNose and the signals were used for online aroma control. Aroma control was achieved by means of varying drying temperatures, which was further implemented by adjusting microwave power with a phase controller. Two control strategies, fuzzy logic control and linear control, were developed to improve drying strategies.

Drying tests with carrot and apple samples showed that high temperatures were not suitable for food drying because of the possible occurrence of burning. Further, the best quality could not be achieved at the lowest drying temperature due to the greater aroma loss resulting from the longer drying time. Instead, medium temperatures were found to be suitable in terms of quality conservation. Drying at the lowest temperature also leads to longer time and higher energy consumption. Hence, high and low temperature should be avoided in microwave drying when aroma retention, burning avoidance, time and energy efficiency are to be considered.

The fuzzy logic controllers developed were able to achieve a compromise among all operating and response parameters. Although the main objectives of the fuzzy logic control were aroma retention and burning avoidance, other quality aspects such as color, taste, and overall appearance were all improved at the same time. Drying time and energy efficiency were also improved with the fuzzy logic controller. Based on the fuzzy logic controlled result, simple linear control methods were further developed where the assistance of zNose was avoided. All the quality aspects investigated could be improved with these simpler control methods, although different linear functions are required to be determined for different food types.

By analysing temperature profiles resulting from the above experiments, it was shown that a fixed drying temperature throughout the whole drying process could not achieve the best drying effects. New drying strategies are required to improve the microwave drying process. In this study, fuzzy logic and linear control methods were employed to improve aroma retention and reduce drying time as well as energy consumption. Fuzzy logic control needs online detection of aroma as variables, while the linear method does not. Linear control is a simplification of fuzzy control. Improved final product quality was achieved with these new control strategies.

CHAPTER X

CONTRIBUTIONS TO KNOWLEDGE AND RECOMMENDATIONS FOR FUTURE STUDIES

10.1 Contributions to knowledge

The major contributions to knowledge of this study are:

1) Through passive (static) aroma studies of both liquid and solid foodstuffs, aroma signals detected with electronic nose technology were demonstrated to have the potential of qualitatively and quantitatively distinguishing, classifying, and grading different food varieties. A new approach to evaluating food quality by aroma was hence established and proven to be faster than traditional chemical analytical instruments.
2) For the first time, a microwave drying system with automatic aroma, temperature and power control was developed. A phase controller was built for power control, a feedback controller was used for temperature control, and a fuzzy logic controller was employed for aroma control. Different control strategies demonstrated their key advantages in the design of overall microwave operating systems and their applications.
3) Through the dynamic aroma investigations of both vegetable and fruit with the system developed, aroma profiles were established and used for online control of the drying temperatures through automatic and continuous power adjustment. Better quality could be achieved and less energy and time were consumed with the combined control strategies.
4) For the first time, a fixed drying temperature throughout the whole microwave drying process was demonstrated insufficient in terms of aroma retention and quality conservation. The study leads to simple approaches without the assistance of aroma detection instruments and the final product quality could also be improved while keeping drying time and energy used at their optimum levels.

10.2 Recommendations for further studies

The methods developed for evaluating Chinese spirits can provide a very useful approach in the spirits industry. Large peaks with long retention times may correspond to key chemicals related to the aging process, which is a critical problem facing all spirit-making industries. However, further in-factory studies are recommended to compare aroma peak methods with the results from a regular GC and oenophiles.

The rot prediction method developed in this study can be immediately applied in the mango industry to prevent the spread of disease. Similar studies can be conducted on other food items to investigate possible disease prediction methods.

An electronic scale with a PC readable signal is necessary for automatic mass measurement in the designed aroma monitoring and control system. With online mass measurements implemented, not only could more detailed information of moisture contents be established, but also new control strategies based on moisture contents in different drying stages could be developed, in an attempt to obtain an optimum drying temperature, energy intensity, and aroma proportion, along with varying moisture contents.

Further studies can be also applied to different food items to establish different optimum drying profiles. Different fuzzy logic and linear control methods can be attempted to compare their drying effects, and to develop optimal methods.

Power rate is another promising direction for further study. By carefully analyzing the recorded data, a simple energy profile can be established in order to avoid the usage of the phase controller, the Labview program, and the data acquisition board, which would make industry application possible.

Food processes beyond microwave drying can also be attempted with a simple adjustment of aroma collection methods. For example, a bell shape vapour collector can be built for aroma collection, and hence the online aroma monitoring and control methods can be expanded to many food processing processes.

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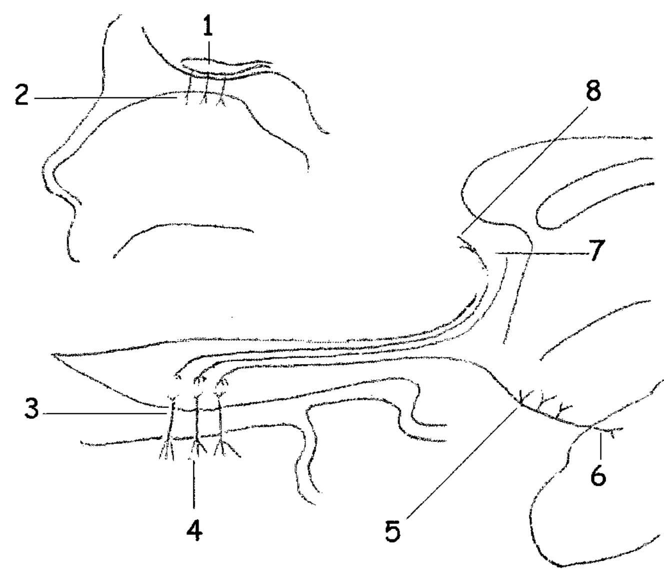
APPENDIX A

Cross-section of the skull, showing the location of the olfactory epithelium, olfactory sensory neurons, cribriform plate, olfactory bulb, and some central connections

(Schiffman and Pearce, 2004)

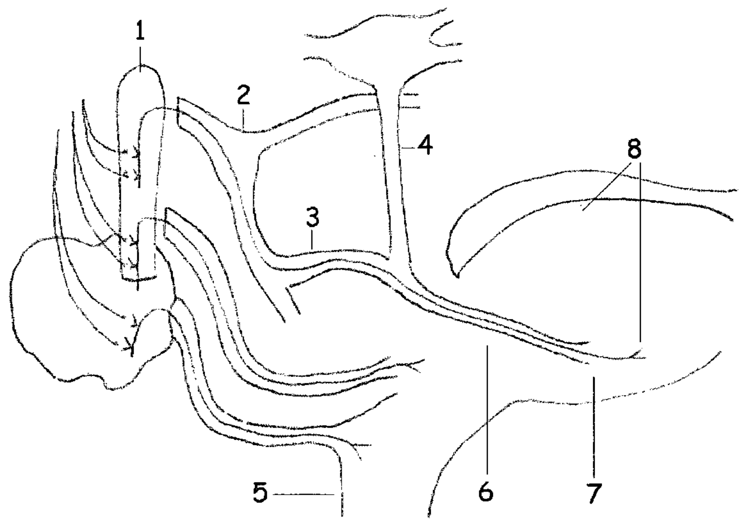


1. Olfactory Bulb
2. Olfactory Mucosa
3. Cribriform Plate of Ethmoid Bone
4. Olfactory Epithelium
5. Lateral Olfactory Stria
6. Uncus
7. Medical Olfactory Stria
8. Olfactory Portion of Anterior Commissure

APPENDIX B

Anatomy of taste showing the cranial nerves and nucleus of solitary tract

(Schiffman and Pearce, 2004)



1. Solitary Tract and Nucleus
2. Greater Superficial Petrosal Nerve
3. Chorda Tympani
4. Lingual Nerve
5. Epiglottis
6. Circumvallae Papillae
7. Foliate Papillae
8. Fungiform Papillae

APPENDIX C

1. *Nyquist theorem*: a theoretical basis for digital sampling of analogue signals. It states that for a limited bandwidth signal with maximum frequency f_{max}, the sampling frequency f_s must be equal or greater than twice the maximum frequency f_{max} in order to have the signal reconstructed without "aliasing", i.e., sufficient samples must be recorded to capture the peaks and troughs of the original signal. If a signal is sampled at less than twice of its maximum frequency, false lower frequency components may appear in acquired data which were actually higher frequency components. This phenomenon is called "aliasing". The *Nyquist theorem* was developed by Harry Nyquist in 1928 and proved by Claude Shannon in 1949. It is also called *Nyquist-Shannon sampling theorem* or simply *sampling theorem.*

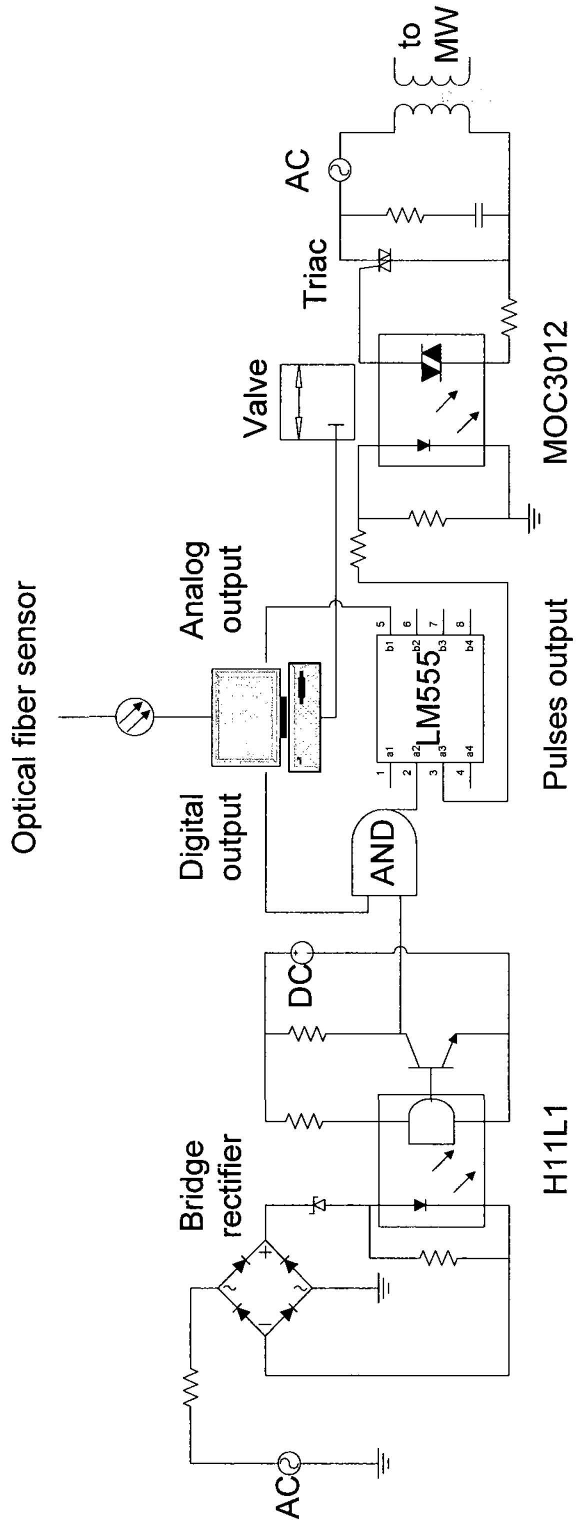
2. *Kovats index*: in "classical" chromatographic methods, each chromatographic peak can be characterized by two parameters: area, which is proportional to the quantity of substance being eluted from column; and its position on the chromatogram (retention time, RT), which reflects the interaction between the sorbate (analyte) and sorbent (stationary phase). However, the "raw" retention times by themselves are not useful for any chemical interpretation owing to their dependence on numerous conditions of analysis and adjusted retention indices (such as *Kovata index*) are frequently used. The *Kovats index,* or *Kovats retention index* of a component is a number obtained by interpolation, relating the RT of the sample component to the RTs of two *n*-alkanes standards eluted before and after the peak of the sample component by the following formula:

$$I = 100[\frac{\log X_i - \log X_z}{\log X_{z+1} - \log X_z} + z]$$

Where X refers to the retention times, z is the number of carbon atoms of the n-alkanes eluting before and $(z + 1)$ is the number of carbon atoms of the n-alkanes eluting after the interested peak i.

APPENDIX D

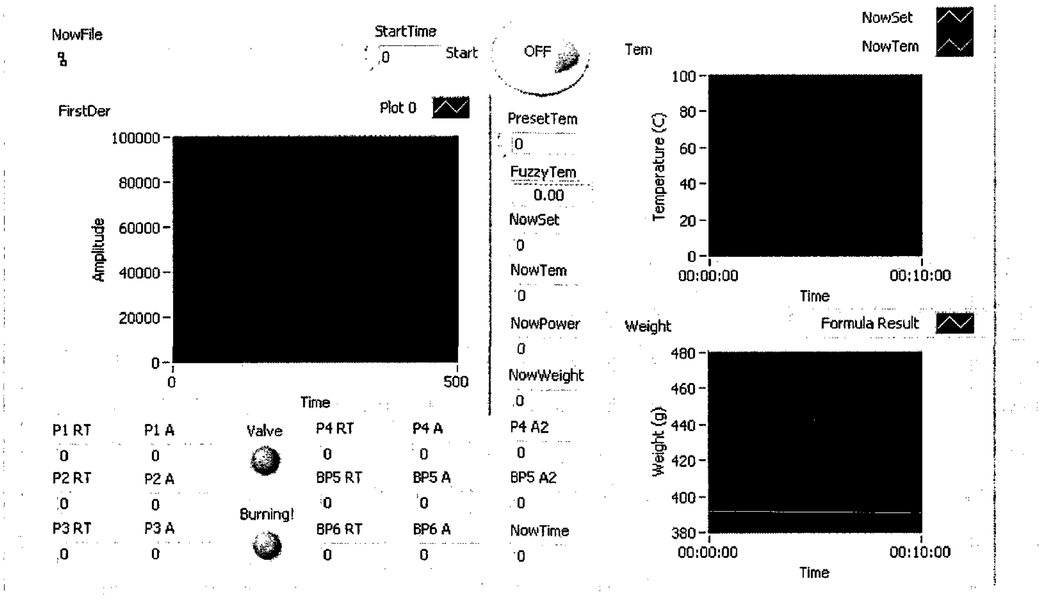
Power control circuits



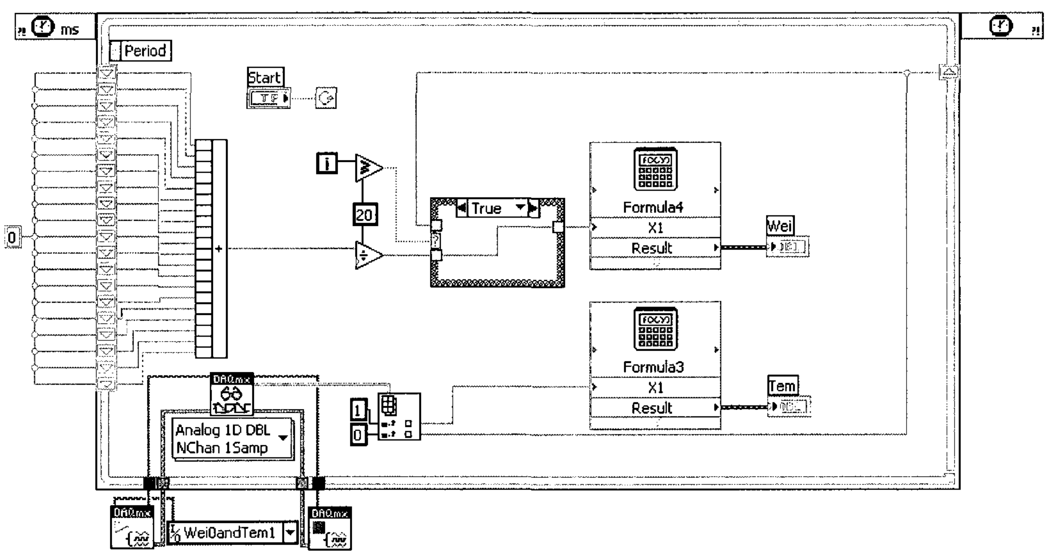
APPENDIX E

Labview programs

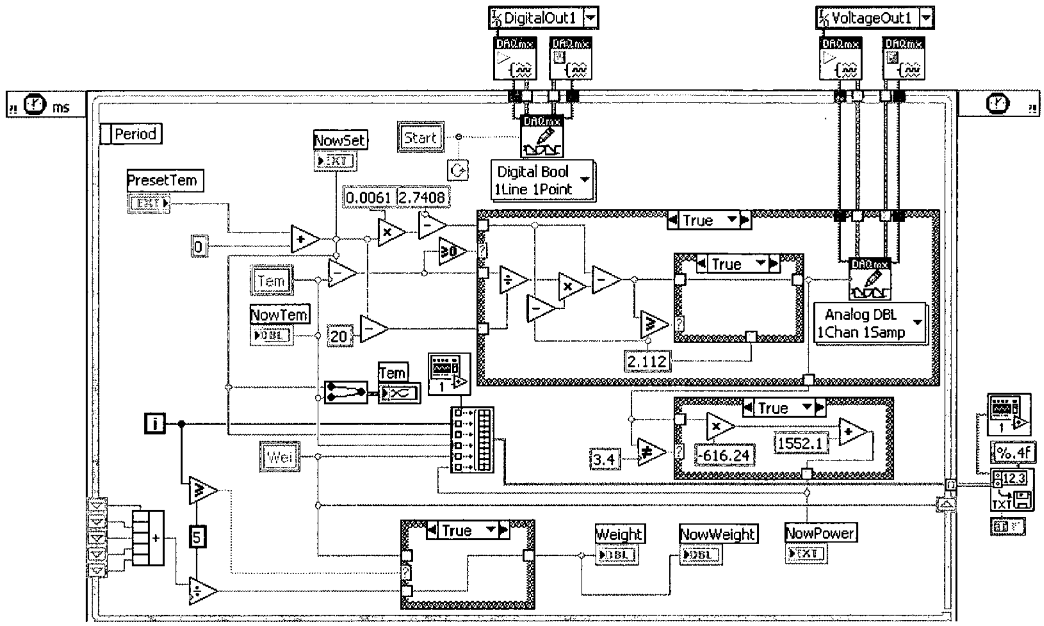
General control - Front panel



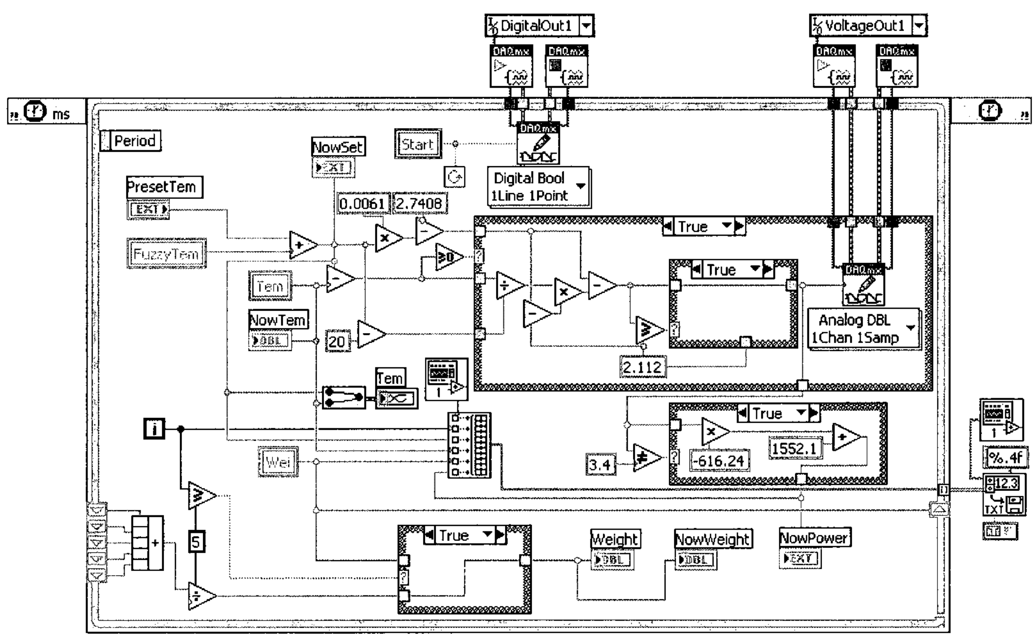
Temperature and mass measurement - Block diagram



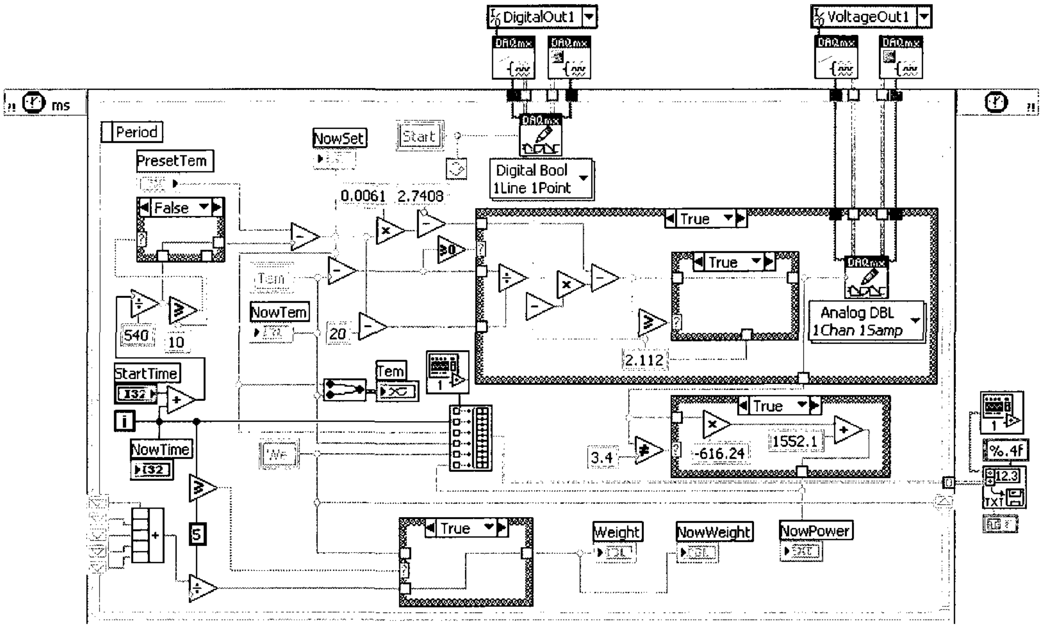
Temperature and power control for fixed temperature - Block diagram



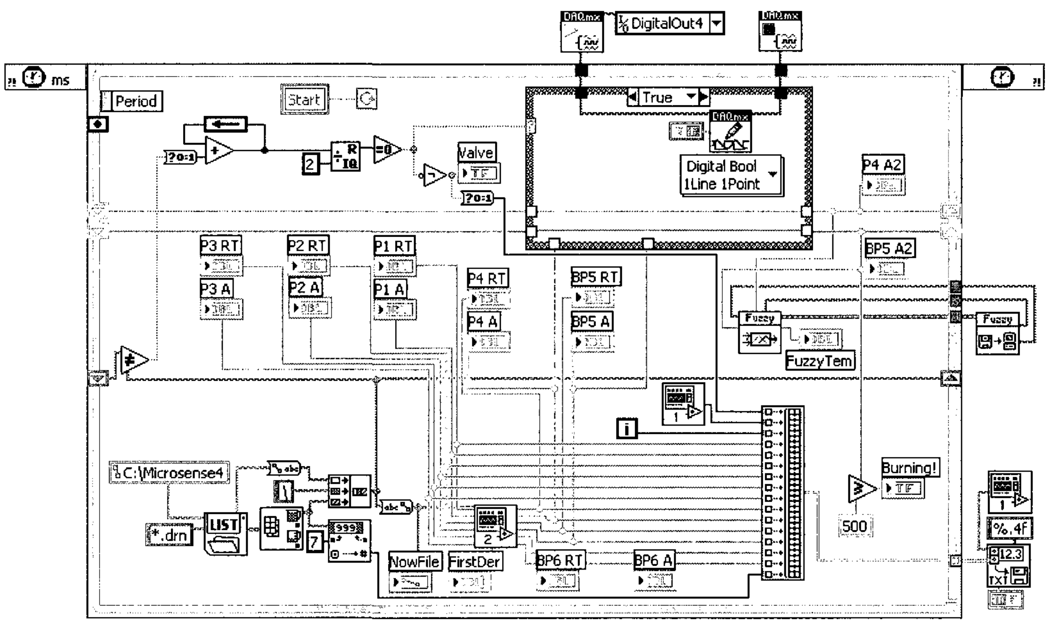
Temperature and power control for fuzzy control - Block diagram



Temperature and power control for linear control - Block diagram

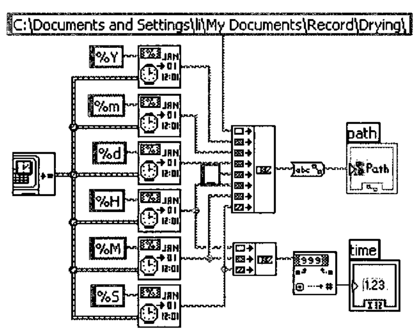


Aroma detection - Block diagram

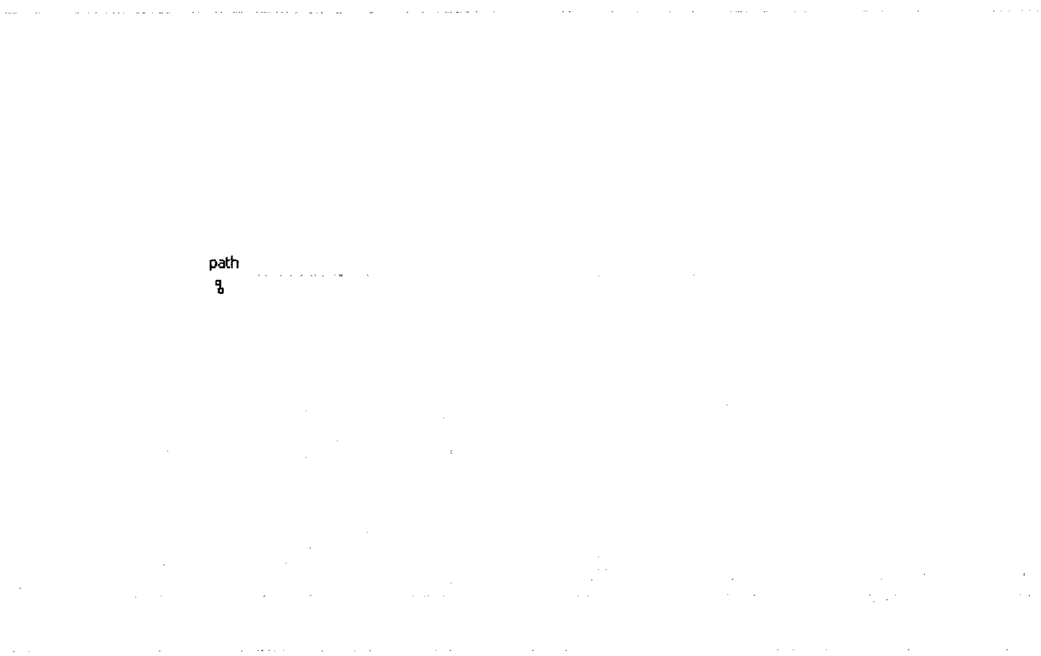


File path SubVI for automatic data record - Front panel

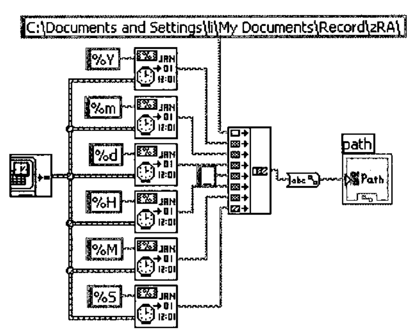
File path SubVI for automatic data record - Block diagram



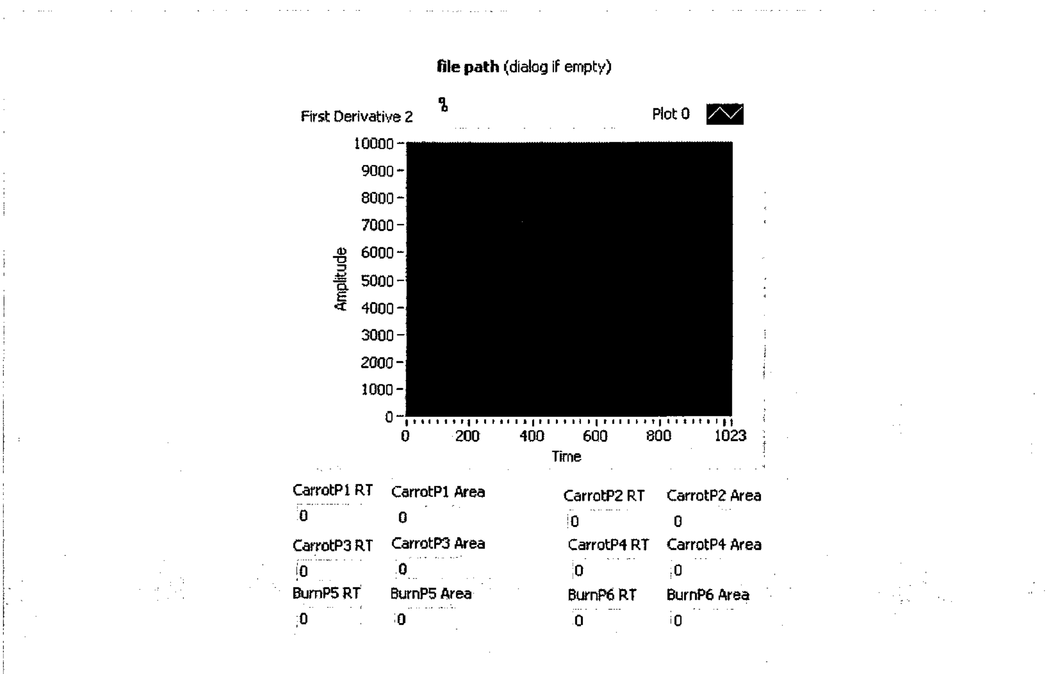
Timing SubVI - Front panel



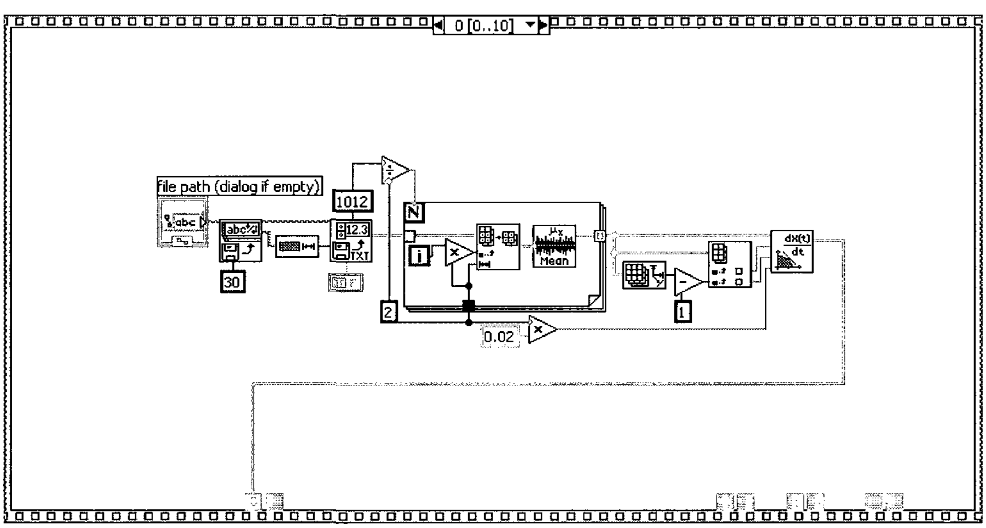
Timing SubVI - Block diagram

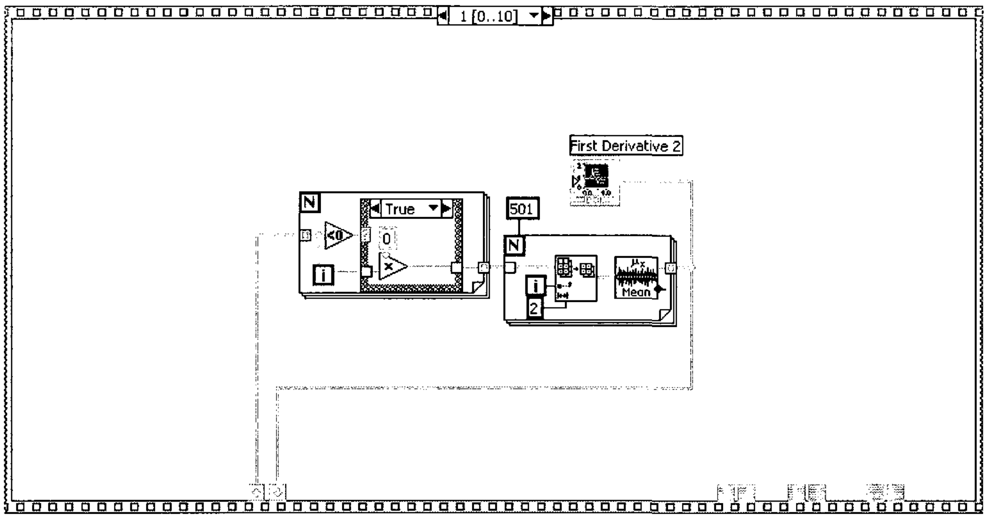


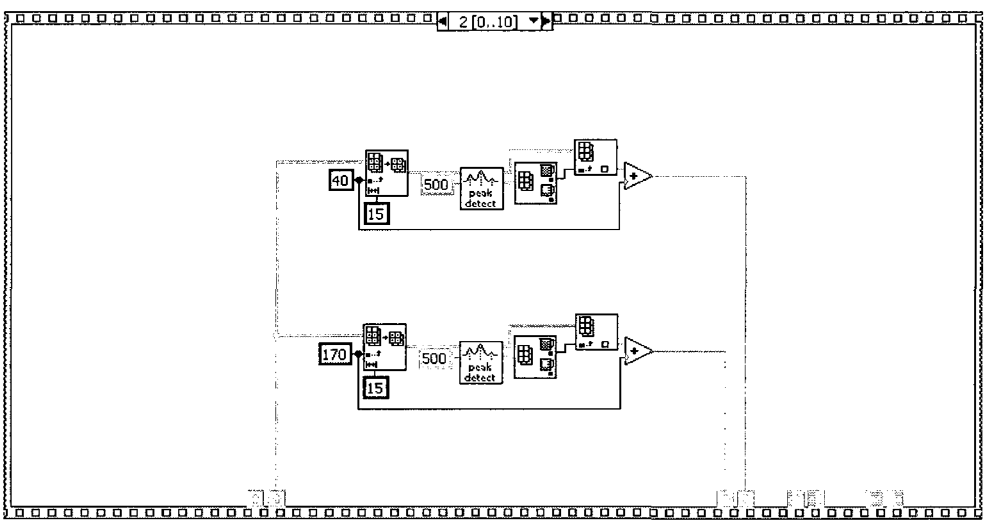
Peak area calculation SubVI - Front panel

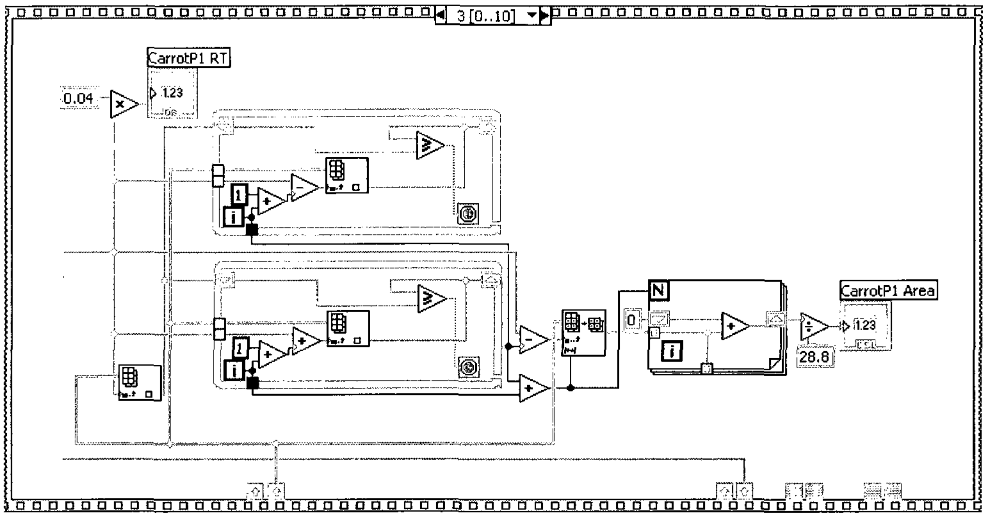


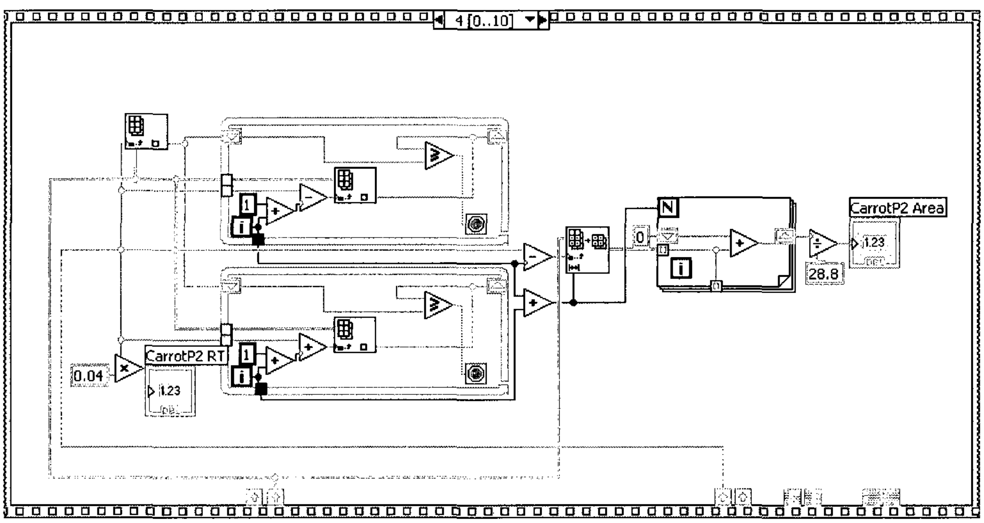
Peak area calculation SubVI - Block diagram

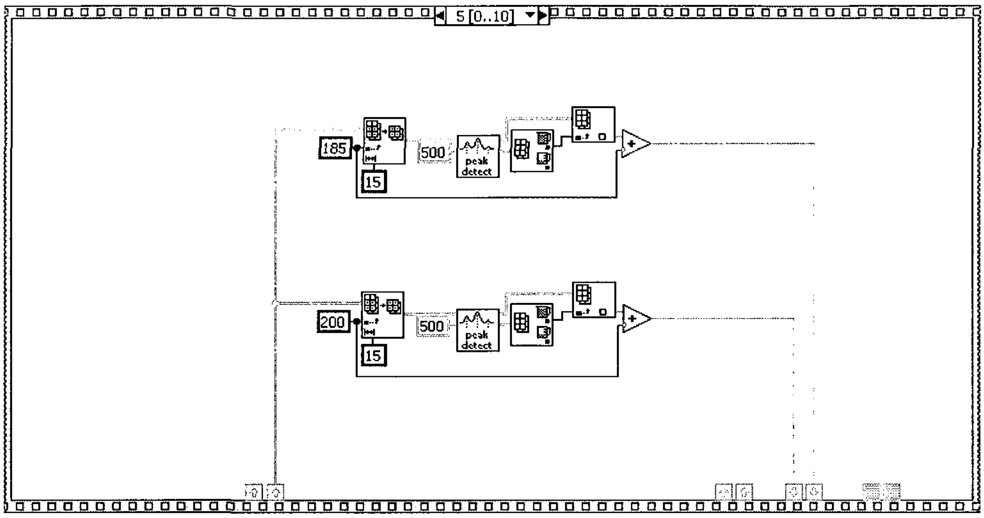


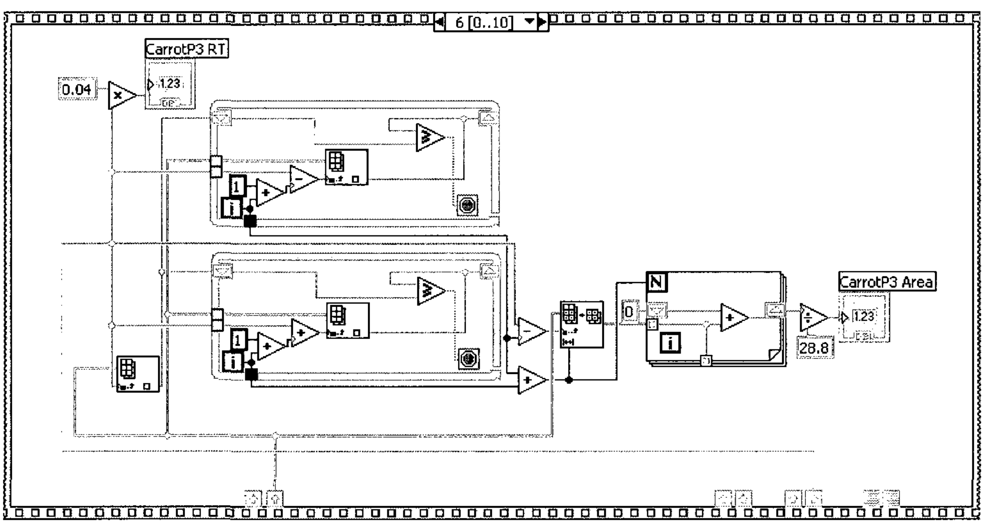

1 [0..10]
First Derivative 2
501
N
True
0
i
2
Mean

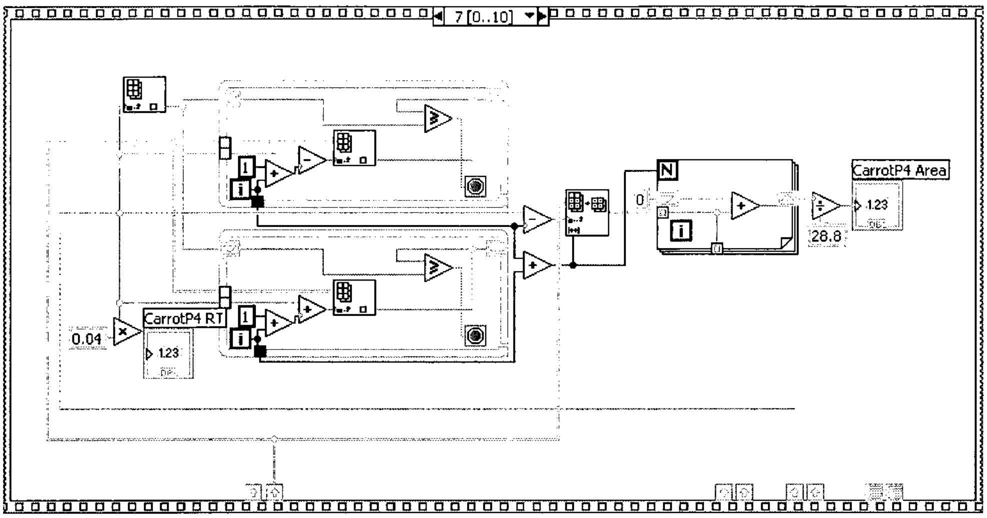

2 [0..10]
40
15
500
peak detect
170
15
500
peak detect

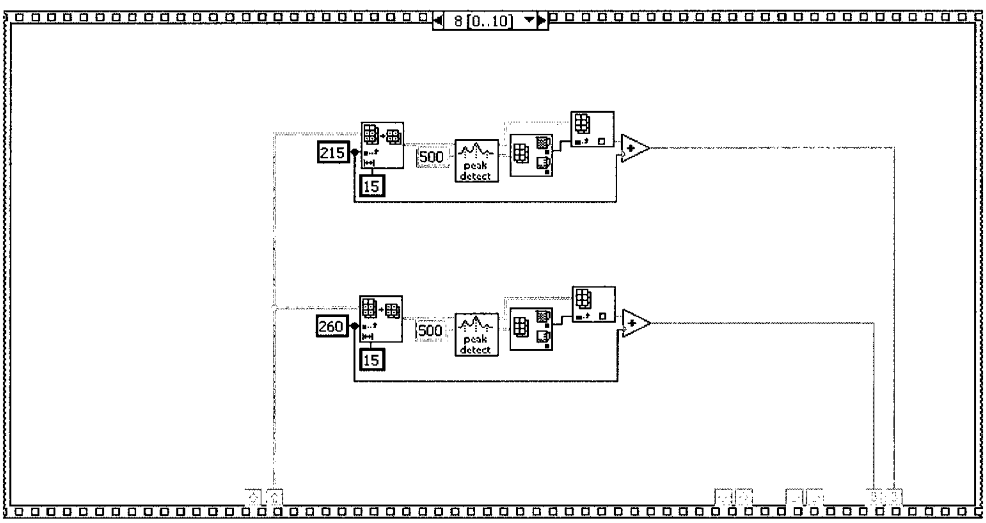

3 [0..10]
CarrotP1 RT
0.04
1.23
N
0
CarrotP1 Area
28.8

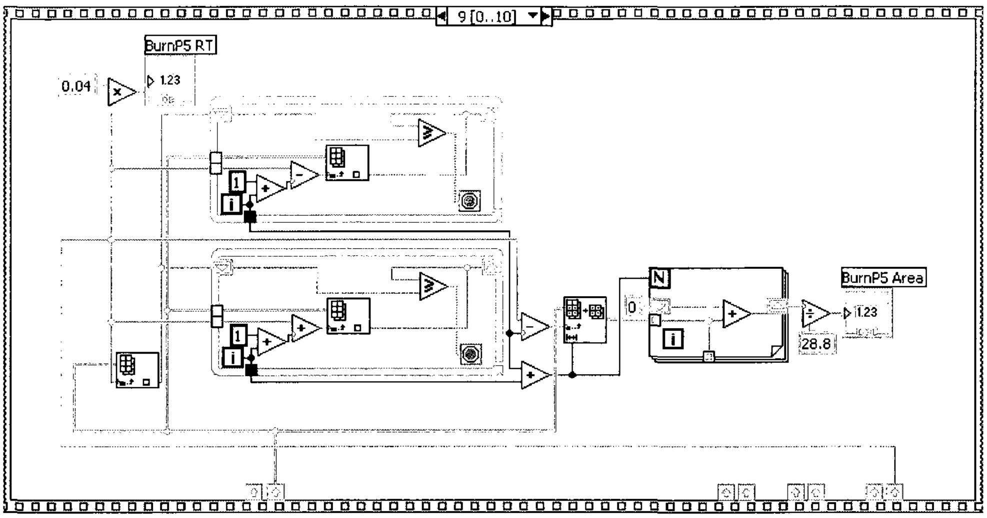

4 [0..10]
CarrotP2 RT
0.04
1.23
N
0
CarrotP2 Area
28.8

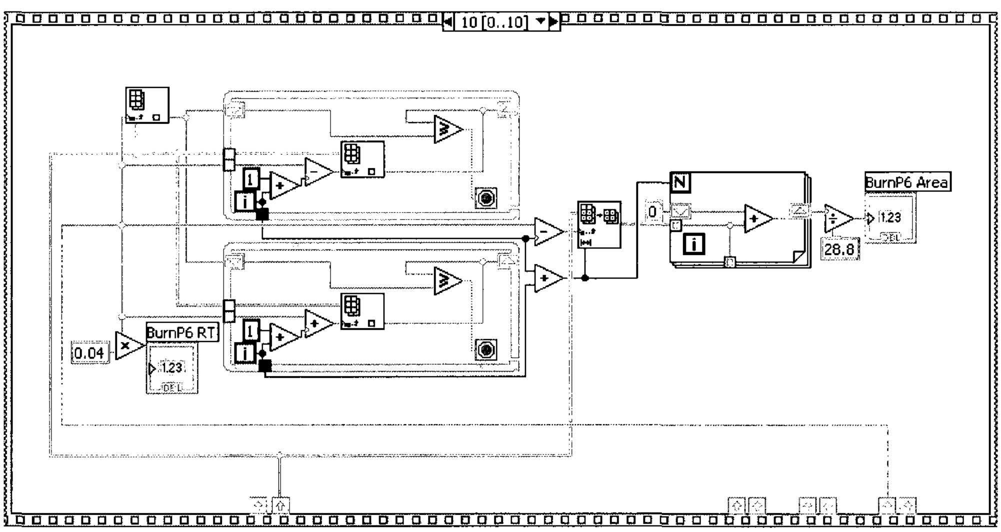

5 [0..10]
185
500
peak detect
15
200
500
peak detect
15


6 [0..10]
CarrotP3 RT
0.04
1.23
1
i
1
i
N
0
i
CarrotP3 Area
1.23
28.8


7 [0..10]
CarrotP4 Area
CarrotP4 RT
0.04
28.8
0
N
1.23


8 [0..10]
215
15
500
peak detect
260
15
500
peak detect


9 [0..10]
BurnP5 RT
0.04
1.23
BurnP5 Area
0
28.8


10 [0..10]
BurnP6 RT
0.04
1.23
BurnP6 Area
0
28.8

APPENDIX F

Standard deviations of carrot aroma peaks

Peaks	Methods	50°C	60°C	70°C	80°C	Fuzzy	Linear
1	Max	11.61	39.39	53.65	95.15	4.56	26.76
	Mean	0.44	1.46	1.56	4.01	0.65	1.01
	Min	0.02	0.04	0.08	0.12	0.04	0.00
2	Max	0.79	47.07	65.15	11.80	1.19	19.62
	Mean	0.22	2.22	3.15	1.08	0.27	0.96
	Min	0.00	0.06	0.03	0.20	0.04	0.05
3	Max	118.46	276.08	102.22	1781.20	128.67	935.45
	Mean	4.06	14.34	10.42	122.25	6.38	36.58
	Min	0.13	1.25	0.08	1.21	0.12	1.48
4	Max	193.74	775.18	918.25	1818.95	287.67	522.04
	Mean	10.82	43.73	76.81	192.03	41.47	35.24
	Min	0.39	2.04	3.82	0.38	3.16	6.02
5	Max	2.05	3.95	13.79	100.59	3.80	4.23
	Mean	0.81	1.99	5.68	23.20	1.39	1.89
	Min	0.00	0.00	0.00	0.00	0.00	0.00
6	Max	2.24	4.49	11.11	45.94	4.15	4.42
	Mean	0.72	2.21	3.91	15.41	1.34	2.03
	Min	0.00	0.03	0.15	1.30	0.10	0.38

Standard deviations of Apple aroma peaks

Peaks	Methods	60°C	70°C	80°C	90°C	Fuzzy	Linear
1	Max	0.97	2.57	2.46	3.08	1.83	2.00
	Mean	0.35	0.51	0.70	1.22	0.32	0.33
	Min	0.00	0.03	0.00	0.03	0.00	0.00
2	Max	2.37	3.53	3.99	4.59	2.75	2.88
	Mean	0.40	1.65	1.24	1.75	0.55	0.41
	Min	0.00	0.12	0.00	0.00	0.00	0.00
3	Max	6.67	6.91	23.90	33.15	7.61	13.64
	Mean	1.91	3.49	7.38	14.06	3.08	4.99
	Min	0.04	0.27	0.01	0.13	0.00	0.01
4	Max	6.31	13.62	20.59	38.84	16.17	19.14
	Mean	2.15	5.05	7.32	22.71	5.45	6.31
	Min	0.09	0.51	0.47	3.68	0.80	0.60