STUDIES ON GAMMA-IRRADIATED COCONUT OIL IN FRYING AND BAKING

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FOOD TECHNOLOGY AND BIOCHEMICAL ENGINEERING

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By

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THIS THESIS WORK IS DEDICATED TO MY FAMILY.

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I hereby declare that this thesis contains literature survey and original research work by the undersigned candidate, as a part of her M.Tech (Food Technology and Biochemical Engineering) studies.

All the information in this document has been obtained and presented in accordance with academic rules and ethical contact.

I also declare that, as required by these rules and conduct, I have fully cited and referenced all materials and results that are not original to this work.

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Certificate of Recommendation

The thesis entitled 'Studies on gamma-irradiated coconut oil in frying and baking' prepared under the supervisor of Dr. Paramita Bhattacharjee by Kona Mondal (Class Roll No.001410902004, Registration No. 129082 of 2014-2015), a student of M.Tech final year, has been evaluated by us and found satisfactory. It is therefore being accepted for the partial fulfilment of the requirement for awarding the degree of Master of Technology in Food Technology and Biochemical Engineering.

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This is to certify that **Ms. Kona Mondal** has carried out research work entitled **'Studies on gamma-irradiated coconut oil in frying and baking'**under my direct supervision in the Department of Food Technology and Biochemical Engineering, Jadavpur University, Kolkata. I am satisfied that she has carried out this work independently and with care and confidence. I hereby recommended that this dissertation be accepted in partial fulfilment of the requirements for the degree of Mater of Technology in Food Technology and Biochemical Engineering.

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SYNOPSIS

The thesis entitled **"Studies on gamma-irradiated coconut oil in frying and baking"** investigates the application of 4.2 kGy gamma-irradiated partially deodorized coconut oil in food product development. Banana chips and cookies were selected as model fried and baked food products, respectively.

The thesis deals with odor profile and shelf life analyses of the said food products during storage using the state-of-the-art electronic nose (e-nose) technology, in conjunction with standard sensory evaluation and biochemical assays of rancidity detection. This work envisages developing assay procedures using electronic nose technology to assess odor profile and shelf life of the food products, to ensure the analyses accurate, reliable, user-friendly and reproducible. This would then enable users to forego cumbersome chemical assays and subjective sensory evaluation.

The thesis has been divided into two chapters. Each chapter has been further divided into two parts and each part has been further subdivided into sections for clarity.

Chapter 1 provides a general introduction to the dissertation topic and discusses about the application of gamma-irradiated partially deodorized coconut oil in manufacture of fried banana chips and their storage study using biochemical assays, sensory and e-nose analyses to ascertain their shelf life. **Part I** discusses in detail the shelf life study of the chips using biochemical assays and sensory analysis. **Section 1.1.1** of Part I is the **Introduction and Review of Literature** section which discusses on banana chips, deep frying of banana chips, advantages of deep frying and gamma-irradiation of coconut oil and details of gamma-irradiation facility. The objectives and need of this research work has also been elaborated in this section. **Section 1.1.2** elaborates the **Materials and Methods** employed for this objective of the investigation. This section emphasizes on the irradiation of coconut oil at a particular dose and frying details of banana chips using the same. The biochemical analyses of banana chips with storage over a period of 60 days were carried out. These studies have aided in investigating the changes in 'sample' banana chips fried in irradiated oil, especially their biochemical and sensory characteristics during storage, against a 'control' set of banana chips, manufactured with non-

irradiated coconut oil. **Section 1.1.3** presents **Result and Discussion** on oxidation of banana chips with storage by biochemical analyses. Further, the product evaluation has been conducted by sensory analyses by a semi-trained panel of university staff. The discussion section explains the results obtained from the above studies. **Part II** of Chapter 1 elaborates on e-nose application in odor profile analysis and shelf life estimation of banana chips against a 'control' sample. **Section 1.2.1** is on **Introduction and Review of Literature** in which a general overview of e-nose technology has been provided with examples of usage of this technology in food systems. **Section 1.2.2** is **Materials and Methods** section which provides details of use of e-nose for odor profile analysis of banana chips. **Section 1.2.3** presents **Result and Discussion** which clearly provides all results obtained in the above study with tables and figures and **Section 1.4** gives a **Conclusion** on the shelf life of banana chips by adjudging their biochemical, sensory and e-nose profiles over a storage period of 60 days.

Chapter 2 discusses about the application of gamma-irradiated partially deodorized coconut oil in manufacture of cookies as baking application and their storage study using biochemical assays, sensory and e-nose analyses to ascertain their shelf life. **Part I** discusses in detail the shelf life study of the cookies using biochemical assays and sensory analysis. **Section 2.1.1** of Part I is the **Introduction and Review of Literature** section which discusses on cookies and formulation of cookies with coconut oil as a replacement of hydrogenated oil, adverse effects of hydrogenated oil on health and also discusses about health-beneficial aspects of coconut oil. The objectives and need of this research work has also been explained. **Section 2.1.2** elaborates the **Materials and Methods** employed for this objective of the investigation. This section emphasizes on the irradiation of coconut oil at a particular dose and baking details of cookies using the same. The biochemical analyses of cookies with storage over a period of 150 days were carried out. These studies have aided in investigating the changes in 'sample' cookies, especially their biochemical and sensory characteristics during storage, against a 'control' set of cookies, manufactured with non-irradiated coconut oil. **Section 2.1.3** presents **Result and Discussion** on oxidation of cookies with storage using biochemical analyses. Further, the product evaluation has been conducted by sensory analyses by a semi-trained panel of university staff. The discussion section explains the results obtained from the above studies. **Part II** of Chapter 2 elaborates on e-nose application in odor profile analysis and shelf life estimation of cookies against a 'control' sample. **Section 2.2.1** is on **Introduction and Review of Literature** in which a general

overview of e-nose technology has been provided with examples of usage of this technology in food systems. **Section 2.2.2** is the **Materials and Methods** section which provides details of use of e-nose for odor profile analysis of cookies. **Section 2.2.3** presents **Result and Discussion** which clearly provides all results obtained in the above study with tables and figures and **Section 1.4** gives the **Conclusion** on the shelf life of cookies by adjudging their biochemical, sensory and e-nose profiles over a storage period of 150 days.

Chapter 3 presents the **Summary and Future Prospects** of the entire research. It provides an overall inference of all the objectives of this project. This chapter explains the utility of the results for academic, industries and consumers alike and suggests future prospects of this investigation.

STUDIES ON GAMMA-IRRADIATED COCONUT OIL IN FRYING AND BAKING

INTRODUCTION

India is the one of the leading producers of coconuts (*Cocos nucifera* L.) in the world and the *West coast tall* variety of coconuts is mainly cultivated here (Gopalkrishnan *et al*., 2013). Coconut oil is highly accepted in the southern part of India and it has appreciable health significance. However, coconut oil has a characteristic rancid-acid odor along with fresh copra flavour, which is unacceptable to the population at large, especially in West Bengal. To increase acceptance and consumption of coconut oil across India and globally, there is a need for deodorization of coconut oil. The conventional method of deodorization of oils involves exposure of the oils to high temperature steam which could be detrimental for the phytochemicals present in oil (Femandes, 2007).

Therefore, our previous work had been conducted employing the non-thermal 'green technology' of gamma (γ) irradiation, as an alternate approach for removal of rancid-acid odor of coconut oil. It was found that irradiation at 4.2 kGy dose, followed by 28 days incubation; there was considerable decrease in rancid-acid aroma of the oil without loss in physiochemical properties and the characteristics copra flavour. These findings have been successfully established employing electronic nose technology (Ghosh *et al*., 2015 and Chatterjee, 2015).

It has been reported that γ-irradiation does not leave any radioactive residues in food products and is safe for food applications (Komolpraser, 2007). We therefore envisage that the irradiated coconut oil with reduced rancid-acid odor (post treatment at 4.2 kGy dose) can be safely utilized for food product development in frying and baking. The present work therefore endeavoured to develop food products using the above mentioned irradiated coconut oil in frying and baking applications. Cookies have been selected as a model baked food product wherein hydrogenated fat has been totally replaced by irradiated coconut oil in the cookie dough; while banana chips have been chosen as the model as fried food product wherein irradiated virgin coconut oil has been used as the frying medium.

CHAPTER 1

GAMMA-IRRADIATED COCONUT OIL IN FRYING APPLICATION

Part -I

Biochemical Studies of Banana Chips

1.1.1

Introduction and Review of Literature

1. INTRODUCTION

1.1 Banana Chips

Banana is a very common edible fruit, consumed in both raw and ripened form. It is mostly grown in tropical and subtropical regions of the world, countries such as India, Sri Lanka, Philippines, Cuba and Australia etc. It belongs to the genus *Musa* (Nelson *et al*., 2006). The raw banana is usually consumed after processing or cooking (Khanvilkar *et al*., 2014). Bananas are classified as 'Desert banana' which are yellow and fully ripe when eaten and 'Green banana'. These are valuable source of Vitamin B_6 and Vitamin C (Stover and Simmonds, 1987). In many regions it is also called as 'plantains' (Ploetz *et al*., 2007). The fruits are of leathery berry type having seeds inside the pulp. The *Musa* variety however, is seedless with only small dark specs in the pulp (Simmonds, 1962).

Owing to rapid growth of the snack food industry, the potential of bananas for processing into chips has become important in countries where bananas are produced (Noor, 1984). A few domestic and small scale industrial preparations based on bananas are One Tambon One Product (OTOP) (Thailand), alloko (West Africa), tajadas (Latin America) (pieces of ripe bananas fried in palm oil) and patacones or tostones (Latin America and West Indies) which are slices of unripe banana fried once or twice in a vegetable oil/animal fat mixture (Diaz *et al*., 1996). Among several banana-based products that are industrially produced and commercially available, fried chips from both unripe and ripe banana are very popular in the Indian subcontinent, as well as in Africa, Latin America, West Indies Malaysia and Indonesia (Mallikarjunan *et al*., 1997; Ammawath *et al*., 2001; Mellema, 2003; Akdeniz *et al*., 2006).

Banana chips, made from raw banana, are also popular snacks especially in the Indian subcontinent and in South East Asia and Africa. The chips are either horizontal slice or longitudinally slices of the fruit. These are produced by deep frying in palm-olein and coconut oils and then packaged after plain salting and/or spicing.

1.1.1 Literature review on banana chips

Literature reviews on deep-fried banana chips in coconut oil reveal that acrylamide formation is higher in ripe banana chips than in unripe banana chips at all temperatures of frying (Mulla *et al*., 2016). Reduction of acrylamide formation has been redressed by synergistic combination of blanching and pectin coating as pre-frying treatments (Suyatma *et al*., 2014). Reductions of 91.9 and 90.8% have been achieved for samples pre-treated by edible coating followed by blanching at 90 and 100◦C, respectively (Suyatma *et al*., 2014). It has been reported that *Pisang Abu* (*Musa* variety) at stages of 'green' and 'trace of yellow' yielded better quality chips than *Nangka;* and bananas with higher water content produced chips with lower crispness, while fresh bananas with higher carbohydrate content and fruit firmness resulted in chips with higher crispness when fried in RBD palm olein oil (Ammawath *et al*., 2001). A storage study of deep-fried banana chips in RBD palm olein oil was carried out for eight weeks at 27°C using four types of packaging material. Laminated aluminium foil (LAF) had the highest crispness, lowest rancid odor and had smallest changes in moisture content, water activity and TBARS values during storage vis-à-vis (PP, OPP, LDPE) (Ammawath *et al*., 2002). Khanvilkar *et al*., (2014) reported that deep-fried banana chips packed in LDPE, were crispier for 45 days (total shelf life was 60 days) when fried in sunflower oil rather than in palm oil; while the chips were rancid and with significant loss of texture when packed in PP.

To the best of our knowledge, there is no application of gamma (γ)-irradiated coconut oil as a frying medium for developing food products such as fried banana chips. Therefore, our present work is to investigate the formulation of banana chips using γ-irradiated coconut oil as a frying medium. This would be a novel approach for new product development through application of irradiated coconut oil.

1.2 Frying

Frying is considered to be more an art than science because it is an extraordinarily complex process involving various factors, some of which are dependent on the process itself and others on the food and the types of frying medium used. Frying oil functions primarily as a heat-

conducting medium for the food. This increases the temperature and causes the food to cook. The optimum deep fat frying temperature for fried food ranges from 170°C to 200°C.

1.2.1 Deep frying of banana chips

Deep frying is the cooking of food in pre-heated oil. It is defined as a classical process of cooking and drying food through contact with hot oil which involves simultaneous heat and mass transfer (Moreira *et al*., 1995).

Deep-fried banana chips are a very popular snack food in many countries and they are prepared by frying unripe bananas (Jackson *et al*., 1996). Deep-fat frying seals the banana surface by immersing banana pieces in hot oil which generates crunchy texture along with desirable color and flavor. High temperature causes partial evaporation of water, which renders crispy fries (Ammawath *et al*., 2001; Khalil, 1999; Moyano *et al*., 2002).

1.2.2 Advantages of deep fat frying

1. Deep fat fried foods have desirable golden color which makes deep-fat fried foods very attractive and popular to consumers (Boskou *et al*., 2006).
2. It also produces unique desirable attributes in foods which include crust formation (crispy texture) and unique flavor (especially aroma) generation.
3. It is a fast and a convenient method.

1.3 Gamma (γ) – irradiation of coconut oil

The sun-dried kernel of coconuts, called copra, is used for extraction of Virgin coconut oil (VCO) worldwide, commonly by expeller pressing. However, owing to the native unpleasant rancid- acid odor of this oil, its acceptability as an edible oil in West Bengal and globally is limited. This odor is a consequence of hydrolytic rancidity of the oil. While lactones, chiefly δ-octalactone, is known to impart the characteristic coconut-like aroma; octanoic acid ($C_{8:0}$) is mainly responsible for the rancid acid odor, notably present even in VCO (Santos *et al*., 2011). Deodorization of oils is conventionally conducted by steam stripping industrially. However, this deodorization technique causes removal of the natural characteristic aroma of oils along with their off odors, rendering the final refined oils odorless and bland. Moreover, steam stripping

degrades antioxidants, generates trans-fatty acids and causes neutral oil loss, thereby causing detrimental effects to oil quality (Petrauskaitè *et al*., 2000; Johnson, 2008). Previous work from this research group has successfully established gamma (γ) irradiation as an alternate technology for edible oils for selective removal of off-odors in coconut oil without compromising its characteristic lactonic aroma as well as its native antioxidants (Ghosh, 2016 and Chatterjee, 2015).

1.3.1 Details of γ-irradiation facility employed in this study

In this study, gamma chamber (GC) 5000 of BRIT, Mumbai with ^{60}Co source was employed for irradiation of coconut oil. The schematic diagram of the unit has been shown in Figure 1. The dimensions of this irradiation unit are 1250 mm x 1065 mm x 1500 mm and its weight is 5.6 tonnes. Inside the gamma chamber, the source is placed within a cylindrical cage, deep within a pit of dimensions 350 mm diameter x 700 mm. Maximum source capacity of this unit is 518 TBq (14 kCi). The entire unit has adequate heavy metal shielding with Pb and steel to restrict surface radiation within the maximum permissible limit of 2 mrad. During irradiation, a maximum dose rate of 9 kGy/ h is obtained at the centre of the sample chamber. The sample to be irradiated is placed inside the sample loading chamber (172 mm x205 mm) located in a vertical drawer inside the Pb shielding. There is a motorized drive to place the sample at the center of the radiation field. There is also provision for rotation of the sample up to a speed of 60 rpm for uniform dose distribution within the sample. The minimum duration of the sample must be exposed to the gamma source is for 6 s. The unit could be used in both auto and manual mode (Ghosh, 2016).

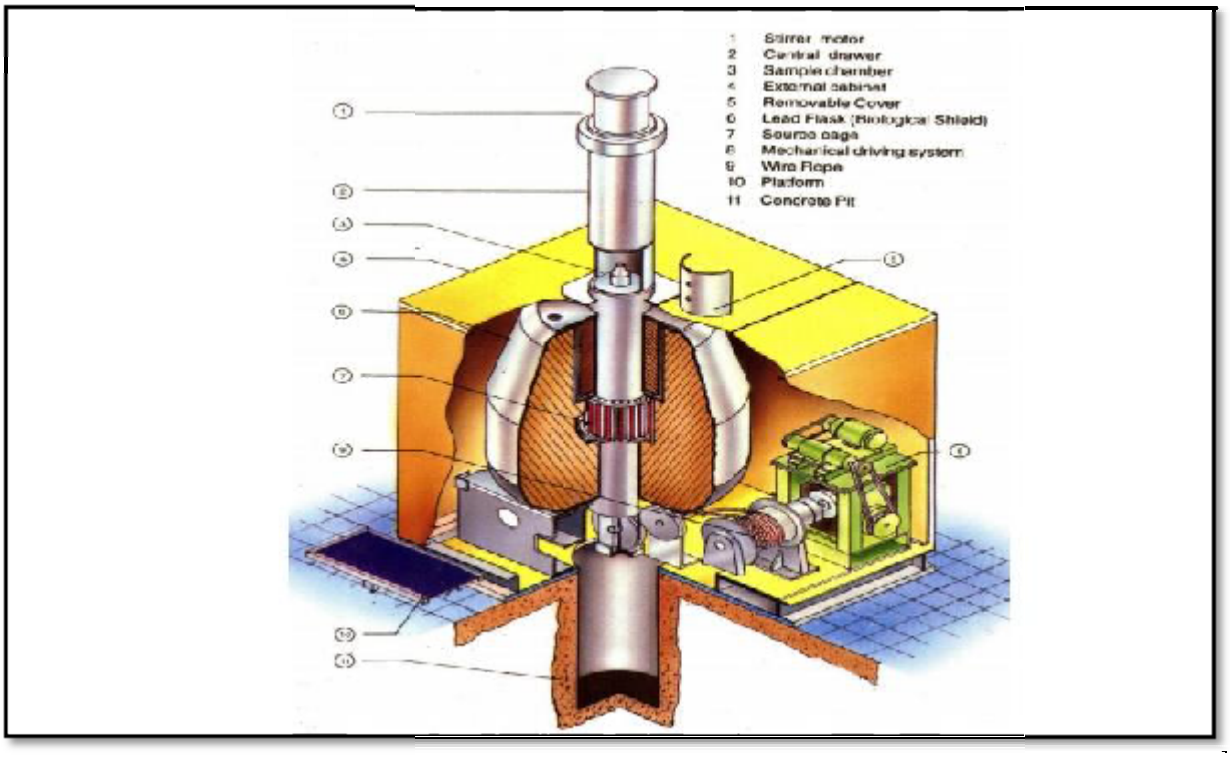

Figure 1: Schematic diagram of a gamma-irradiation chamber

In our present study, a particular dose of 4.2 kGy has been applied for coconut oil deodorization which was established in our laboratory (Chatterjee, 2015). At this dose, the oil underwent least oxidation on processing and during storage. The best deodorized coconut oil had been achieved at 4.2 kGy with the storage period of 28 days as confirmed by sensory evaluation in tandem with electronic nose (mimicking human olfactory system) technology (Sabyasachi, 2015).

This literature survey emphatically established that no work has been conducted on application of γ-irradiated coconut oil in food frying. The current work focused on utilization of γ-irradiated coconut oil without its native unpleasant rancid-acid odor as a frying medium for banana chips. If successful, the product (banana chips) using irradiated coconut oil would certainly have increased acceptability in India nationwide and also globally.

The specific objectives of this work are as follows:

1. To assess suitability of γ-irradiated VCO in frying application.
2. To assess the biochemical changes in the fried chips (‘control’ and ‘sample’) by routine assays.
3. Sensory evaluation (subjective study) of the fried banana chips by semi-trained panelists consisting of university staff and students would be performed to assess the flavor harmony of the chips (contributed by copra flavor of the coconut oil, rancid-acid (octanoic acid) odor of coconut oil and the characteristic banana flavor) in both ‘sample’ and ‘control’ chips in tandem with biochemical assays.
4. Assessment of shelf life of banana chips by correlation of results obtained in points 1, 2 and 3.

1.1.2

Materials and Methods

2. MATERIALS AND METHODS

2.1 Reagents and samples

The food materials comprised of commercial coconut oil (M/s KPL Oil Mills (P) Ltd., Kerala, India) and raw plantains (*kanchkola*) (*Musa* variety), purchased from a local supermarket of Kolkata, India. The commercial oil had been extracted from coconut copra of West coast variety cultivated and harvested under similar conditions and the oil was obtained using expeller method, as confirmed by the manufacturer (Ghosh *et al*., 2014).The coconut copra (ball copra) from West coast tall variety of coconuts was authenticated by the Coconut Development Board, Kolkata, India, cultivated in the state of Kerala, India. The agro climatic requirements of coconut are coastal sandy soil with pH 5.5-7.0, mean temperature of cultivation 30±5°C, and annual rainfall between 1000-3000 mm. The mature coconuts were harvested in the present year (2015), when they were 12 months old. The harvested coconuts were subsequently dried to obtain copra. All this information was collected from Coconut Development Board, Kolkata, India. The copra were sorted and ground by an electric grinder (M/s Philips Electronics Ltd., Kerala, India) for extraction.

Specialty chemicals such as 2-Thiobarbituric acid (TBA) were procured from M/s Sigma-Aldrich Crop. (St. Louis, MO, USA). Sulphuric acid, phenolphthalein, potassium hydroxide, starch, glacial acetic acid and ethanol were purchased from Merck (Darmstadt, Germany). All chemicals used for analyses were of AR grade unless specified. Amber bottles, aluminium foil, LDPE self-sealable pouches bags (20 μ thickness) were purchased from a local market in Kolkata. Deep frying was carried out in a 5 L table-top deep fat fryer (M/s Shiva Kitchen Appliances, Kolkata, India). A T-type thermocouple (2 mm diameter) with a transducer-display was procured from M/s G.B Enterprises, for controlling deep frying temperature. Irradiation was conducted in a Co-60 γ- irradiation chamber (GC 5000; Serial No.GIC 038; Source Cobalt 60 solid; Manufactured by BRIT, Mumbai) at National Instrument Laboratory (NIL) campus of Jadavpur University.

2.2 Irradiation of coconut oil

Irradiation of coconut oil was conducted at 4.2 kGy dose for reasons described in section 1.3. A total of 10 amber bottles of 500 ml each were filled with coconut oil leaving bare minimum headspace, nitrogen purged and sealed with teflon. All bottles were irradiated in the Co-60 γ-irradiation chamber in accordance with the procedure described in section 1.3.

2.3 Preparation of banana chips for frying

2.3.1 Peeling and cutting of plantain

The fresh raw plantains were washed in distilled water and peeled by manual peeler. The plantains were cut as 'chips' of 2 mm thickness with a kitchen knife. The uniformity in the size was desired to obtain homogenous samples.

2.3.2 Blanching of banana chips

Blanching of the banana chips was carried out with the objective obtaining a crisper fried product with lower oil uptake and lower production of acrylamide and with minimum colour change. Therefore, the freshly cut banana chips were blanched to arrest enzymatic activity within the banana chips. The chips were blanched with 0.1% potassium metabisulphite (w/v) at 90°C for 10 minutes. Following blanching, the water was drained, and the chip surfaces were dried using blotting paper.

2.3.3 Deep Frying of banana chips

Frying of banana chips were carried out in an electric deep fat fryer (M/s Shiva Kitchen Appliances, Kolkata, India) using the protocol for deep frying of potato wedges previously standardized in our laboratory (Ghosh *et al*., 2012) with little modifications. In every batch, 300 g of banana chips were fried at 170°C for 8 min in 4 L of 28-day stored 4.2 kGy irradiated coconut oil. This batch of chips was designated as the 'sample' set. The chips were then placed in a strainer to drain the excess oil and then air cooled. The chips were subsequently placed on blotting paper to remove the excess oil from the surface. The 'control' set was obtained in a similar manner using commercial oil instead of irradiated oil.

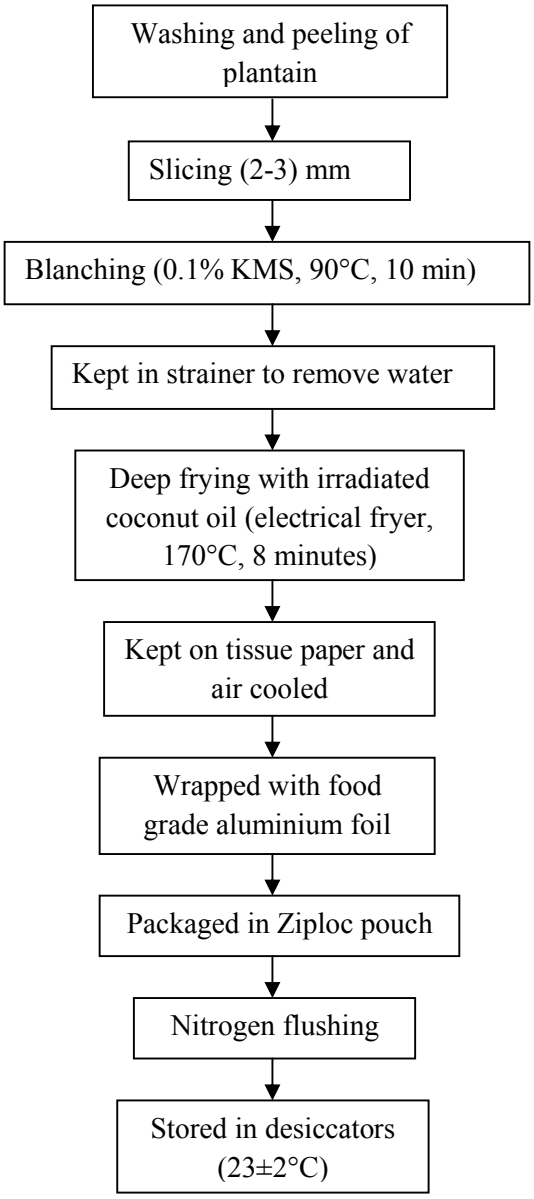

Figure 2: Schematic flow chart of preparation of 'sample' set of fried banana chips

2.4 Product analysis

2.4.1 Chemical assay

2.4.1.1 Determination of free fatty acids (FFA)

FFA content of the oil was analyzed in accordance with AOCS Official Method AOCS, 2002; as described by Chatterjee (2015). The acid values were expressed as % oleic acid.

2.4.1.2 Peroxide value (PV)

PV is a measure of the amount of peroxides formed in fats or oils through auto oxidation and other processes. The PV of oil sample was determined by using standard procedures (AOCS, 2002). The PV values are expressed as milligram of oxygen/ kg of oil or in ppm. The procedure of PV determination was in accordance to that by Ghosh *et al*., (2012).

2.4.1.3 TBARS assay for malondialdehyde in banana chips

In oils, unsaturated aldehydes can undergo oxidation giving rise to secondary products of lipid oxidation, chiefly dialdehydes such as malondialdehyde. To assess the wholesomeness and safety of the fried banana chips prior to consumption, malondialdehyde was determined in deep frying using TBARS assay, in accordance with the method reported by Okhawa *et al*., (1979). The procedure for determination of malondialdehyde was in accordance with the method repeated by Mishra (2013).

2.4.2 Storage studies of banana chips for shelf life assessment

2.4.2.1 Storage

The fried banana chips were wrapped in food grade aluminium foil and packaged in Ziploc pouches. Nitrogen was flushed into them before sealing. Thereafter, the pouches were stored at 23±2°C in desiccators for a total period of 60 days and the samples were withdrawn for analyses at intervals of 0, 10, 20, 30, 45 days and on the 60^{th} of storage. Two separate desiccators were used to store ‘sample’ and ‘control’ sets. To detect the rancidity profile of the banana chips for both ‘control’ as well as ‘sample’ product with the conventional methods, two sets of deliberately rancid sample were prepared. These training sets of banana chips were prepared by

keeping ‘control’ chips and ‘sample’ chips (non-spoiled) in an accelerated rancidity chamber for 1, 4 and 7 days. This rancidity chamber is an incubator to render chips deliberately rancid. The chamber is equipped with UV light to promote oxidation of the chips at constant temperature of 40±2°C. On completion of the ageing treatment, the samples were removed from the same, flushed with nitrogen and stored in an environment control chamber provided with an inert atmosphere of nitrogen at constant temperature of 23±2°C, to maintain the final rancidity stage (Chatterjee *et al*., 2014).

2.4.2.2 Sensory evaluation

Sensory evaluation was assessed by a semi trained panel of university faculty members and research scholars (15 men and 15 women), in the age group of 20-45 years. The sensory evaluation was conducted between 10-12 AM and 3-5 PM as recommended (Rangana, 1987). During testing, the panelists were monitored by the author. The samples (stored ‘control’ and ‘sample’ banana chips) were served in paper saucers (10×10 cm) with two fried banana chips of both type in each. The panelists were served water to cleanse their palate and crackers (as carrier) and were allowed a rest period of 5 min between consecutive samples. The experiment was designed to achieve easy replicability under similar conditions.

This methodology was in accordance to the reported methods wherein acceptability of food products has been adjudged by semi-trained panels using affective method-based acceptance test (such as hedonic score rating) (Rao and Vakil, 1985).The panelists’ evaluated the food products based on the attributes of overall appearance, odor, color, texture, taste and aftertaste using a 9-point hedonic scale (9-like extremely and 1-dislike extremely) in a well-illuminated ventilated room. Each sample was served in triplicate in a session and rounded off mean scores were analyzed (Ghosh, 2016). The score of quality attributes for different doses has been represented graphically by radar plot (Figures 1a and 1b).

1.1.3

Results and Discussion

3. RESULTS AND DISCUSSION

3.1 Chemical analysis of products (banana chips)

3.1.1 Acid value

The changes in acid values (mg KOH/g oil and expressed as% oleic acid) for the storage period of zero day to 60 days are shown in Table 1. The initial acid value of 0 day-stored 'control' chips (using non-irradiated coconut oil) was found to be 0.56, which changed significantly ($p<0.05$) to 0.89 up to 30 days. After 30 days, the acid value was found to constantly change and finally reach a value of 0.90 at the end of 60 days storage. FFA content of 'control' banana chips until 60^{th} day (0.90) of storage was much closer to that of the 1-day stored deliberately rancid chips (0.91). From these observations, it could be stated that rancidity might have been initiated on 60^{th} day in 'control' chips.

The initial acid value of 0-day'sample' chips were 0.45; however, this was much less than that of 0 day-stored 'control' chips. The acid value of 'sample' chips increased linearly to 0.78 at the end of 10 days; after which it attained a constant value of 0.78) for the rest of the storage period. FFA content of 'sample' chips on the 60^{th} day (0.79) was lower than that of the 1-day stored deliberately rancid 'sample' chips (0.90).Therefore, 'sample' banana chips were found to be more stable and less prone to develop rancidity than the 'control' banana chips. The primary oxidation and microbial infestation generate FFAs in food samples and hence this could be used as an indicator of spoilage for fried banana chips. The increase in the acid values of oils or fats indicates hydrolysis of triglycerides (due to moisture, temperature or enzymes) (Arlee *et al*., 2013). According to Lawson (1985) and Che Man *et al*., (1997), hydrolysis is accelerated by high temperature and excessive amounts of water and therefore Volatile organic compounds (VOC) produced by fermentation method has high FFA content. Although, we do not have supporting data in literature on irradiated coconut oils to substantiate our work, the trend observed in our investigation is in agreement with that found for VCO.

3.1.2 PV

The PV values of 'control' chips (using non-irradiated coconut oil) increased significantly ($p<0.05$) until the end of the total storage period of 60 days. At the end of 60days, it was found

that the PV of 'control' chips was 0.006 ppm, lower than that of the 1-day stored deliberately rancid 'control' chips (0.010 ppm). In case of 'sample' chips at the end of 60 days storage, PV was 0.0045 ppm which was lower than that of the 1-day stored deliberately rancid 'sample' chips (0.0079 ppm) as shown in Table 2.

PV is a measure of the oxidation of the triglycerides, and any value less than 5 ppm shows no significant oxidation (Thermo Fisher Scientific, 2004). In the present study, PV is much less than specified. PV measures the primary products of oxidation (hydroperoxides) of the reaction between oxygen and unsaturated fatty acids. Since, fried banana chips were stored in an inert atmosphere of N_2, the spoilage of banana chips cannot be accounted by primary oxidation alone. Therefore, primary oxidation cannot alone be employed as a spoilage indicator for 'control' and 'sample' chips. Hence, TBARS assay was also conducted to check the generation of secondary oxidation products such as malondialdehyde.

3.1.3 TBARS assay for formation of malondialdehyde

The malondialdehyde content for 'control' chips increased linearly from 0 day to the end of the total storage period of 60 days shown in Table 3. We observed optimum content of malondialdehyde (mmoles malondialdehyde/g dried chips) in chips at the end of the storage period for both 'sample' and 'control' chips. The initial value of 0 day 'control' chips (using non-irradiated coconut oil) was found to be 0.31, which significantly ($p<0.05$) changed to 0.92 at the end of storage. The malondialdehyde content of 60 days stored chips was lower than that of 1-day stored deliberately rancid chips (1.22).

The initial value of 0-day 'sample' chips was 0.45, which significantly changed ($p<0.05$) to 1.25 at the end of storage. The malondialdehyde content of 60 day-stored 'sample' was lower that of the 1-day stored deliberately rancid chips (1.39). From these observations, it could be stated that onset of rancidity started on the 60^{th} day in the 'sample' chips, while the same was lower in 'control' chips'. Therefore it could be stated that 'control' chips were more stable than 'sample' chips since secondary oxidation products have been generated in low amount in the former. This increase of TBARS values (malondialdehyde content) with frying was in agreement with observations of Cheman and Tan (1999). To the best of our knowledge, there are no set standards

and reports in literature that indicate the acceptable level of malondialdehyde in banana chips to enable comparison of our experimental data.

3.1.4 Sensory analysis of banana chips

The response of the panelists for the 'control' (using non-irradiated coconut oil) and 'sample' (using irradiated coconut oil) chips with storage depicts greater sensory acceptability of 'sample' chips in comparison with the 'control'.

There were no differences in overall appearance, colour and texture between 'control' and 'sample' chips with storage. The products were ad judged based on odor, taste and aftertaste. The odor of coconut oil was milder in 'sample' chips from day zero until the end of storage than that of the 'control'. An oddity in the odor was detected in 60 day-stored 'control'. Panelists found that with time, the strong flavour of freshly fried banana chips became milder and there was no off-flavour generation until the 30^{th} day in 'sample' set. Rather, the banana flavor dominated the coconut flavor in both 'control and sample' sets. In the 45 day-stored 'sample' chips, there was slight generation of metallic odor; however, no off-flavour generation was found in 'control' chips. This could be attributed to higher secondary oxidative product generation in 'sample' chips rather than in the 'control' as obtained by TBARS assay; although taste and aftertaste were better in 'sample' compared to 'control' since the former had less oily mouth feel.

TABLES

Table 1: FFA content of 'control' (using non-irradiated oil) and 'sample' (using irradiated oil) banana chips during storage period of 60 days and those of deliberately rancid banana chips

Storage period (days)	FFA (% oleic acid)	
	CONTROL	SAMPLE
Banana Chips		
0	0.56±0.02[a]	0.45±0.01[a]
10	0.68±0.03[b]	0.78±0.03[b]
20	0.79±0.04[c]	0.78±0.04[b]
30	0.89±0.04[d]	0.78±0.03[b]
45	0.89±0.03[d]	0.79±0.04[b]
60	0.90±0.04[d]	0.79±0.03[b]
Deliberately made rancid chips		
1	0.91±0.04[d]	0.90±0.04[c]
4	1.01±0.05[e]	1.01±0.05[d]
7	1.68±0.07[f]	1.57±0.06[e]

Mean ± S.D of three samples of one experimental set

[a-f] Different letters in a row indicates significant difference ($p<0.05$)

Table 2: PV (ppm) content of 'control' (using non-irradiated oil) and 'sample' (using irradiated oil) banana chips during storage period of 60 days and those of deliberately rancid banana chips

Storage period (days)	PV (ppm × 10^{-4})	
	CONTROL	SAMPLE
Banana Chips		
0	5±0.25[a]	3±0.15[a]
10	14±0.70[b]	12±0.60[b]
20	50±2.50[c]	44±2.20[c]
30	52±2.60[c]	43±2.15[c]
45	57±2.85[c,d]	44±2.20[c]
60	65±3.25[d]	45±2.25[c]
Deliberately made rancid chips		
1	101±5.05[e]	79±3.95[d]
4	103±5.15[e]	81±4.05[d]
7	106±5.30[e]	82±4.10[d]

Mean ± S.D of three samples of one experimental set

[a-f] Different letters in a row indicates significant difference ($p<0.05$)

Table 3: Malondialdehyde (mmole/g dried sample) content of 'control' (using non-irradiated oil) and 'sample' (using irradiated oil) banana chips during storage period of 60 days and those of deliberately rancid banana chips

Storage period (days)	MDA (mmole/g dried sample)	
	CONTROL	SAMPLE
Banana Chips		
0	0.31±0.01[a]	0.45±0.01[a]
10	0.49±0.02[b]	0.52±0.02[a,b]
20	0.54±0.02[b,c]	0.57±0.03[b]
30	0.59±0.03[c]	0.69±0.03[c]
45	0.61±0.03[c]	0.88±0.04[d]
60	0.92±0.04[d]	1.25±0.05[e]
Deliberately made rancid chips		
1	1.22±0.05[e]	1.39±0.05[f]
4	1.56±0.06[f]	1.60±0.07[g]
7	2.61±0.12[g]	2.95±0.14[h]

Mean ± S.D of three samples of one experimental set

[a-h] Different letters in a row indicate significant difference ($p<0.05$)

FIGURES

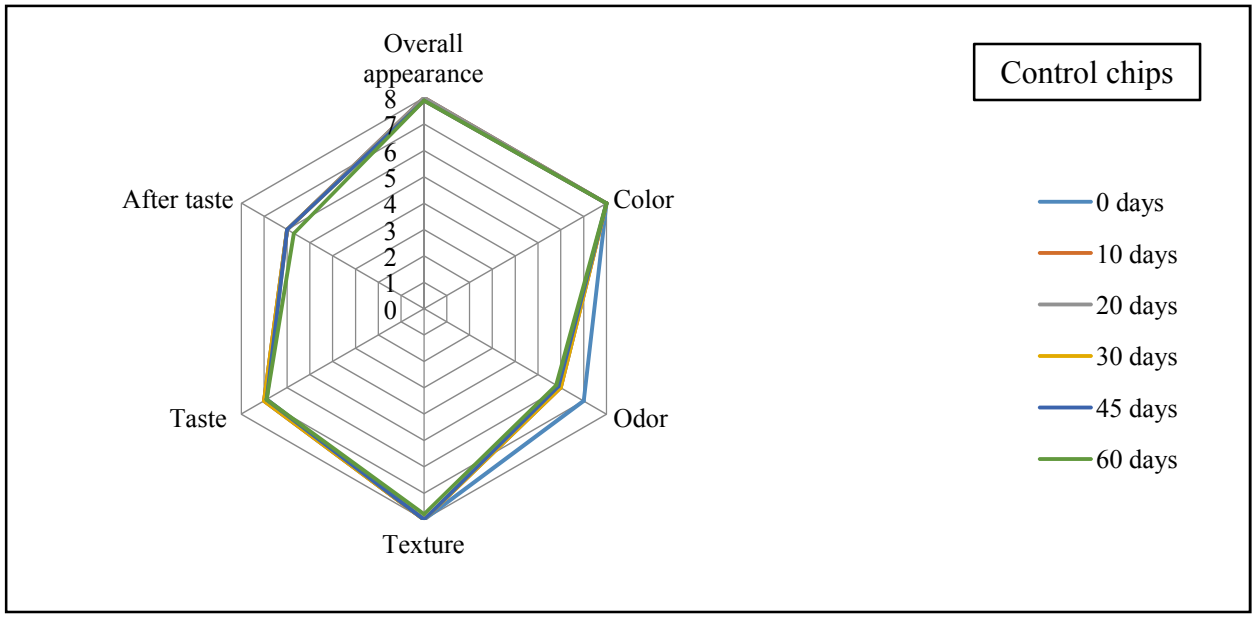

Figure 1(a): Effect of storage on sensory characteristics of 'control' banana chips

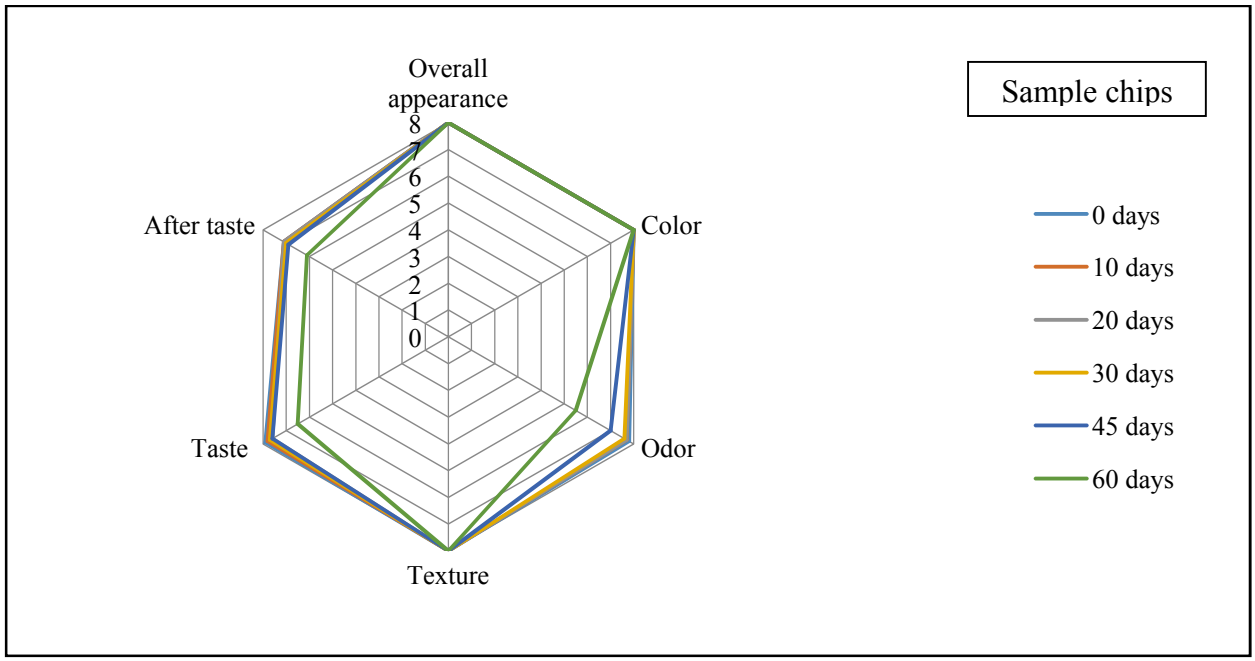

Figure 1(b): Effect of storage on sensory characteristics of 'sample' banana chips

Part–II

Electronic nose Analysis for Odor Detection of Banana Chips

1.2.1

Introduction and Review of Literature

1. INTRODUCTION

Sensory evaluation although mandatory for food product analysis, is subjective and non-reproducible. It is therefore necessary to validate the sensory results by an objective method such as by electronic nose (E-nose). E-nose analysis is widely employed currently for evaluation of flavours of food products. This technology clearly minimises errors arising due to mammalian olfactory analysis of food products and is a preferable technology also owing to its reasonable cost and ease of sample preparation.

1.1 Electronic nose Technology

In 1961, Moncrieff developed an instrument to detect odors. In 1965, studies of redox reactions of odorants at an electrode, modulation of electrical conductivity, and contact potential by odorants were published. The concept of an e-nose as a chemical array sensor system for odor classification was presented for the first time by Persaud and Dodd at the University of Warwick in 1982. The "e-nose" also called an "artificial-nose" is defined as an instrument, which comprises of an array of electronic chemical sensors having partial specificity and an appropriate pattern recognition system to recognize simple or complex odors (Gardner *et al*., 1994). Its main components are: sample handling systems, chemical sensor arrays, signal pre-processing and conditioning, and pattern recognition. The e-nose simulates human olfactory process with fewer sensors, and suitable software analyzes responses from sensors (Sabyasachi, 2015). Each sensor produces a time-dependent electrical signal in response to an odor. The specificity of each sensor may be low, but combinations of several specificity classes results in a wide range of information. Pattern recognition in the e-nose is equivalent to the classification of odors in the brain (Gardner and Barleti, 1999). The details of the electronic nose system have written in hard copy of the thesis.

1.2 Application of e-nose in food analyses

The complexity of most food aromas make them difficult to be characterized with conventional flavour analysis techniques such as gas chromatography or gas chromatography olfactometry. Nevertheless, sensory analysis by a panel of experts is a costly process since it requires trained people who can work for only relatively short period of time; additional problems such as the

subjectivity of human response to odors and the variability between individuals are also to be considered. Hence the need of an instrument such as the electronic nose, whose strengths include high sensitivity and correlation with data from human sensory panels for several specific applications in food control, because they are easy to build, cost-effective and as they provide a short time of analysis, electronic noses are becoming more and more popular as objective automated non-destructive techniques to characterise food flavours. However, there is much research still to be done especially with regard to sensor technology, data processing, interpretation of results and validation studies.

- E- noses use for five major categories in food control in process monitoring, shelf-life investigation, freshness evaluation, authenticity assessment and other quality control studies (Peris et al., 2009).
- Electronic nose instrumentation has advanced rapidly over the last decade with several successful applications in food industries to detect the freshness of food such as meat (Rossi *et al*., 1996), milk (Capone *et al*., 2001), mango (Lebrun *et al*., 2004), mushrooms (Tamaki *et al*., 2007) and fishes such as Atlantic salmon (Haugen *et al*., 2005) and silver pomfret (Yi *et al*., 2010) and also detect shelf-life of cookies (Chatterjee *et al*., 2012).
- This technology has also been widely used in beverage industries to detect the flavour characteristics of tea (Bhattacharyya *et al*., 2004); coffee (Marukami *et al*., 2010) and grape wine (McKellar *et al*., 2005; Chatterjee *et al*., 2012).
- Recently it has found its use in edible oil industry to know about the proportion of blending so as to increase its shelf life (Men *et al*., 2014).
- An electronic nose (e-nose) has applied for identifying the botanical origin of honey as well as determining their main quality components (Huang *et al*., 2014), also used for the Chinese medical herbs discrimination (Li *et al*., 2015) and also used to clarify the dynamic changes of flavour components during hot air drying, comprehensive flavour characterization and volatile compounds of *F. velutipes* (mushroom) has evaluated using this technology (Yang *et al*., 2015).

Considering, all the above flavour compounds, in our present study, individual sensor responses have not been considered since the ENOVISION system does not have sensors specific to sense banana aroma volatiles. We have holistically adjudged the odor profile (flavor harmony) of the

chips, and not on the basis of response of a particular sensor. This approach of holistic harmonised judgement of flavor profile was considered appropriate in the current investigation.

Earlier work conducted in our laboratory has established a new method of detection of coconut odor profile using two models of e-nose. Therefore, in the current work, we believe that e-nose analysis would allow assessment of shelf life of banana chips, in tandem with biochemical assays. The specific objectives of this part of the study are as follows:

1. E-nose analysis for determining shelf life of 'sample' banana chips (irradiated oil used as frying medium) with storage in comparison with 'control' banana chips (frying medium was commercial virgin coconut oil) by Principal component analysis (PCA).
2. Selection of best sensors which detected the odor profile best and ranking them to obtain the short listed sequence of sensors appropriate for analyzing banana chip volatiles fried in irradiated coconut oil.
3. Establishment of co-relation between biochemical assay parameters of rancidity and e-nose analysis.

1.2.2

Materials and Methods

2. MATERIALS AND METHODS

2.1 Reagents and samples

Plantains (*Musa* varity), virgin coconut oil (M/s KPL Oil Mills (P) Ltd., Kerala, India) and LDPE self-sealable pouches bags (20 µ thickness) (M/S UFLEX Ltd.,USA) were purchased from a local supermarket of Jadavpur in Kolkata, India. The commercial oil had been extracted from coconut copra of West coast variety cultivated and harvested under similar conditions and the oil was obtained using expeller method, as confirmed by the manufacturer (Ghosh *et al*., 2014). Specialty chemicals and standards were procured from M/s Sigma-Aldrich Crop. (St. Louis, MO, USA). Copra flavour was purchased from M/s International Flavors and Fragrances, Mumbai, India. All chemicals used for analysis were of AR grade.

2.2 Irradiation of coconut oil

Procedure of irradiation of coconut oil has been described in 2.2 of Part I of Chapter 1.

2.3 Preparation of banana chips

Banana chips were prepared as described in section 2.3 of Part I of Chapter 1.

2.4 Optimization of electronic nose system

Optimization of e-nose system parameters such as amount of banana chips required, heating time, headspace generation time, sampling time and purging time were fixed through preliminary trials. The e-nose system was trained with a deliberately made rancid 'sample' and 'control' chips. This procedure was in accordance to our previous work on e-nose analysis of rancidity in fried potato wedges (Chatterjee *et al*., 2014).

2.5 E-nose analysis of banana chips

E-nose analysis of banana chips ('sample' as well as 'control') was carried out at an interval of 0, 10 and 15 days for a storage period of 60 days. The responses of the eight sensors of the e-nose for sample of banana chips was determined from $\Delta R/R$ value, which is the change in the resistance of metal oxide sensor due to the VOCs of banana chips with respect to the base value

(Bhattacharyya *et al*., 2008). The base value of banana chips is the resistance shown by the sensors due to VOCs of freshly (non-spoiled) batch of prepared banana chips.

2.6 Storage of banana chips

The chips were stored as described in section 2.4.2.1 of Chapter 1.To detect the odor profile and shelf life of the fried banana chips, the e-nose system was trained using deliberately rancid chips.

2.7 Selection and ranking of sensors

In order to screen the metal oxide gas sensors that are most sensitive towards VOCs generated from the banana chips 'sample' as well as 'control', ranking of the sensors was carried out by plotting the graph between individual response with time (storage days). The signal responses (ΔR/R) of 8 sensors were collected from banana chips 'sample' and 'control' on 0, 10, 10, 20, 30, 45 and 60 days of storage. Among, the 8 sensor, best sensors were selected with respect to the variation in signal response and the sensors giving maximum value over a specific time period with maximum slope and trend line. Deliberately made rancid chips for both 'control' and 'sample' have also been analysed for 1,4,7 days interval for supporting the best sensor data.

The (ΔR/R) is a dimensionless value. To obtain (ΔR/R), the resistance of freshly prepared sample has been subtracted from the resistance obtained for the sample analysed on a particular storage day. The value obtained is negative. This is because of characteristic aroma compounds such as isopentyl acetate of banana aroma and the copra flavour of coconut oil. Therefore the absolute values are taken as the sensor responses (|ΔR|/R) (Chatterjee, 2015).

Statistical analysis

Statistical analysis of customized e-nose data was conducted by Principal Component Analysis (PCA) using XLSTAT, 2015 software. Regression equations were generated to predict the PV values, FFA and MDA content as a function of ''spoilage index'' by using STATISTICA 8.0 software. Significant differences between means were determined by Duncan's multiple-range test. A p value of 0.05 was used to verify the significance of all tests.

1.2.3

Results and Discussion

3. RESULTS AND DISCUSSION

3.1 Optimization of electronic nose system

Ground banana chips of 25 gm (dp = 1.5 mm) were placed in a 100 ml glass vial and heated for 400 s at 50°C. The headspace generation was carried out for 30 s to ensure adequate concentration of VOCs released by the chips in the sample holder by blowing regulated flow of TOC grade air on the sample. Sampling time was kept constant for 50 s, where the sensor array was exposed to a constant flow of VOCs through pipelines inside the electronic nose. Purging operation was carried out for 400 s, where sensor heads were cleared with the blow of fresh air so that the sensors go back to their base line values. The e-nose system was previously calibrated with a set of deliberately-rancid banana chips as discussed earlier. The responses shown by the sensors due to the VOCs of these batches were selected as standard odor profiles of banana chips having different degrees of rancidity (Vinaixa *et al*., 2005).

3.2 Selection and ranking of sensors

The signal response of the individual eight sensors for 'sample' chips and 'control' chips over a time period of 60 days on 0, 10, 20, 30, 45 and 60 days is represented in Figures 1a, 1b, 2a and 2b. A graph was plotted taking time period in X-axis and resistance in Y-axis for both types of chips with trend line and slope equation.

We clearly found that TGS 816, TGS 832, TGS 2600 and TGS 2611 gave higher responses for the VOCs generated from for both 'sample' and 'control' banana chips (Figures 2a and 1a). These four sensors are generally used for detecting combustible gases, methane, butane, propane, CFC, air contaminants and ethanol and have been found to be working good for detecting VOCs from banana chips. E-nose results were analyzed with data obtained from these screened sensors.

It has been previously established that TGS 823 is the best sensor for coconut oil flavour (copra flavour) and octanoic acid (rancid-acid aroma), reportedly known to detect alcoholic compounds and solvent vapors. In our present study, a particular sensor response for the odor analysis has not been considered owing to the fact that freshly fried banana chips have complex volatile compounds from different sources, namely, native coconut oil, fried coconut oil and the banana

fruit itself. As mentioned earlier, these compounds could be 'isopentyl acetate' the characteristic aroma compound of banana, few esters of pentanol, such as those of acetic, propionic and butyric acids, esters of butanol and hexanol with acetic acid and butyric acids and eugenol, *o*-methyleugenol and elemicin (Belitz *et al*., 2009) and characteristic volatile compounds generated during deep frying of coconut oil such as *n*-alkanes, 1-alkenes, alkanals, 2-alkenals, 2-alkanones, γ- and δ-lactones, FFA, esters and very intense flavored carbonyl compounds from coconut oil heated at 180°C for 48 h (David and Thomas, 1989). Since the flavor harmony of the fried banana chips would be owing to contribution of all the above-mentioned flavors, we have holistically considered the response of the entire sensor array and four sensors from them have been selected based on their high values of (|ΔR|/R).

Using the above-discussed methodology and the graphs obtained as discussed in section 2.7, higher slope was obtained for 'sample' than for 'control' chips. This could be owing to the flavors discussed above and also more due to additional flavors generated in fried irradiated coconut oil. γ-Irradiation reportedly causes radiolysis of fatty acids (Cm:n, m= number of carbon atoms, n=number of double bonds) forming two groups of long chain volatile hydrocarbons (Cm-1:n and Cm-2:n+1) by rupture of side chains in the α and β positions with respect to carbonyl group, according to the theory of Nawar (1986). Although γ-irradiation at 4.2 kGy dose was previously established for deodorization of VCO, where radiolysis of octanoic acid occurred, it also generated hydrocarbons consequent to oxidation during frying and generated more secondary oxidation products in food samples. However, the native oil (not subjected to frying) irradiated at 4.2 kGy and stored for six months was non-rancid as obtained by analyses using Heracles E-nose (Ghosh, 2016).

3.3 Shelf life assessment of fried banana chips by PCA analysis

An odor map was generated on the basis of PCA plot (Figures3 and 4) with discrimination indices of 98.73% and 98.66% for 'control' and 'sample 'chips indicating the clusters to be distinctly separated. It was observed that all the 'sample' chips with different storage periods have formed a well-defined cluster along with the 'control' chips. The 'control' chips at 45 (C45) and 60 days (C60), formed a distinct cluster and was present in the same quadrant along with the cluster of 'deliberately-made rancid chips (TS)'. It has also been shown that |⊿R|/R values of the

C60 storage chips had crossed those of the cluster of TS1, whereas the same for C45 storage chips were still quite far apart from those of TS1. Therefore, it could be concluded that 60-day stored 'control' chips had similar odor profile as that of the deliberately rancid chips, indicating onset of rancidity on the 60^{th} day of storage in the 'control' chips.

The 45-day and 60-day stored 'sample' chips formed a distinct cluster and was present in the same quadrant along with the cluster of 'deliberately-made rancid chips' and the45 day-stored chips had crossed the cluster of TS1. Therefore, it could be concluded that 'sample' chips with storage period of 45 days had similar odor profile as that of the rancid chips, indicating that 'sample chips tend to become rancid on 45^{th} day of storage. However, visible degradation of chips was not noticeable at the end of the storage period.

3.4 Correlation between the rancidity parameters of banana chips determined by e-nose analysis and biochemical assays

From the experimental data, a linear correlation was found between the responses of the different sensors of e-nose system and rancidity parameters obtained by biochemical assays. Therefore, multiple regression analyses were carried out to develop three linear regression equations to predict the FFA, PV and TBA value (individually) of the fried banana chips as a function of responses of four metal oxide gas sensors (TGS 816, TGS 832, TGS 2600 and TGS 2611), which are provided below.

$$Y_1 = 0.4512 - 0.2909 \times X_1 + 0.4213 \times X_2 - 0.2486 \times X_3 + 1.2758 \times X_4 \quad (1)$$

$$Y_2 = 0.0002 - 0.0052 \times X_1 + 0.0469 \times X_2 - 0.0023 \times X_3 + 0.0064 \times X_4 \quad (2)$$

$$Y_3 = 0.4480 - 0.7587 \times X_1 + 0.7086 \times X_2 - 0.4686 \times X_3 + 1.7003 \times X_4 \quad (3)$$

where, Y_1 is the FFA value of the 'sample' chips (% oleic acid), Y_2 is the PV of the 'sample' chips (ppm) and Y_3 is the TBA value of the 'sample' chips (mmol malondialdehyde/g dried chips). X_1, X_2, X_3 and X_4 are the e-nose responses obtained from sensor TGS 816, TGS 832, TGS 2600 and TGS 2611 respectively. The correlation coefficients obtained from eqs. 1–3 are 0.99, 0.95 and 0.99 respectively. The validity of the fitted models were indicated by the

insignificant lack of fit ($p<0.1$) for these equations and by F-values of 59.64, 2.09 and 112.97 corresponding to eqs. 1, 2 and 3 respectively. Therefore, these equations obtained from sensor data analysis of ENOVISION, could successfully be used to predict the conventional rancidity parameters of the fried banana chips since the e-nose sensor responses well correlated to the results of the biochemical assays for the entire storage period of 60 days.

1.4

Overall findings and conclusion

4. Overall findings and conclusion

The quality attributes of the 'sample' chips with respect to 'control' were evaluated through this study using different parameters. Biochemical studies indicated that FFA and PV (primary oxidation) are less in 'sample' rather than in 'control' chips whereas secondary oxidised product, malondialdehyde content (MDA) is more in 'sample' banana chips. Sensory panel acceptability was more for 'sample' chips rather than for 'control', but at the end of 45 days, a metallic off-odor in 'sample' chips was detected. Also on the basis of malondialdehyde content obtained in our study, we cannot recommend the 'sample' banana chips for market consumption. Further investigation is required to ascertain the safety of the chips for consumption. It could be stated that irradiated coconut oil at this particular (4.2 kGy) dose when used for deep frying was more prone to oxidation and more secondary oxidised compounds were generated.

The shelf life of the chips has been ascertained through PCA analysis. By comparison of 'control' and 'sample' PCA plots, it could be clearly stated that 'sample' chips had lower shelf life compared to the 'control'. Moreover, from the biochemical and sensory analyses of either set of chips in tandem with e-nose analysis, we have holistically recommended that 'sample' chips are not good for health owing to their higher malondialdehyde content and low shelf life. Three regression equations have been established which could be used to predict the conventional rancidity parameters of the fried banana chips from e-nose analyses. This can forego routine biochemical assays of chips.

TABLES

Table 1: E-nose sensor responses (|ΔR|/R) of 'control' fried banana chips during the storage period of 60 days

Storage period (Days)	Sensor Response(|ΔR|/R)							
	TGS 816	TGS 832	TGS 2600	TGS 2611	TGS 823	TGS 2620	TGS 830	TGS 2610
0	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
10	0.0947	0.0445	0.0762	0.0150	0.0819	0.0135	0.0390	0.0972
20	0.2018	0.0575	0.0839	0.0945	0.0738	0.0304	0.0387	0.0764
30	0.2398	0.0635	0.0943	0.1052	0.0167	0.0208	0.0360	0.0881
45	0.2500	0.0740	0.1190	0.1386	0.0824	0.0271	0.0393	0.0941
60	0.2774	0.0731	0.1380	0.2129	0.0302	0.0299	0.0723	0.0297

Table 2: E-nose sensor responses (|ΔR|/R) of 'sample' fried banana chips during the storage period of 60 days

Storage period (Days)	Sensor Response (|ΔR|/R)							
	TGS 816	TGS 832	TGS 2600	TGS 2611	TGS 823	TGS 2620	TGS 830	TGS 2610
0	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
10	1.1319	0.1131	0.7959	0.6258	0.0604	0.0952	0.0302	0.8381
20	1.2656	0.1540	0.7975	0.6623	0.0371	0.0785	0.0182	0.8626
30	1.6219	0.2207	0.8227	0.7083	0.0247	0.1278	0.0732	0.9015
45	2.1616	0.2518	0.8616	0.8437	0.0993	0.1715	0.1355	1.1021
60	2.9390	0.3322	1.5404	1.1273	0.0252	0.0873	0.0696	0.8923

Table 3: E-nose sensor responses (|ΔR|/R) of deliberately made rancid 'control' fried banana chips for best four sensors during the storage period of 7 days

Storage period (Days)	Sensor Response (|ΔR|/R)			
	TGS 816	TGS 832	TGS 2600	TGS 2611
0	0.0000	0.0000	0.0000	0.0000
1	0.2703	0.0772	0.1369	0.1925
4	0.2793	0.0812	0.1425	0.1997
7	0.2814	0.0856	0.1475	0.2022

Table 4: E-nose sensor responses (|ΔR|/R) of deliberately made rancid 'sample' fried banana chips for best four sensors during the storage period of 7 days

Storage period (Days)	Sensor Response (|ΔR|/R)			
	TGS 816	TGS 832	TGS 2600	TGS 2611
0	0.0000	0.0000	0.0000	0.0000
1	2.4964	0.2567	0.9254	0.6735
4	2.5784	0.2710	0.9486	0.7187
7	2.8526	0.3065	1.1988	0.7614

FIGURES

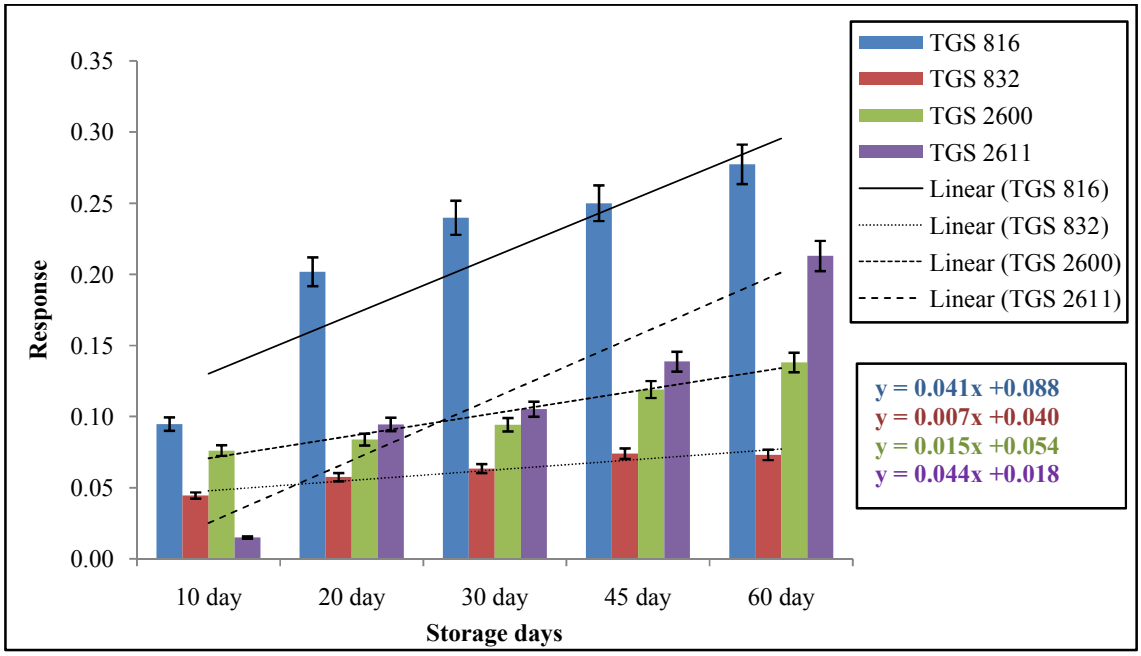

Figure 1(a): Plot variation of signal response with storage period of 'control' fried banana chips over a time period of 60 days

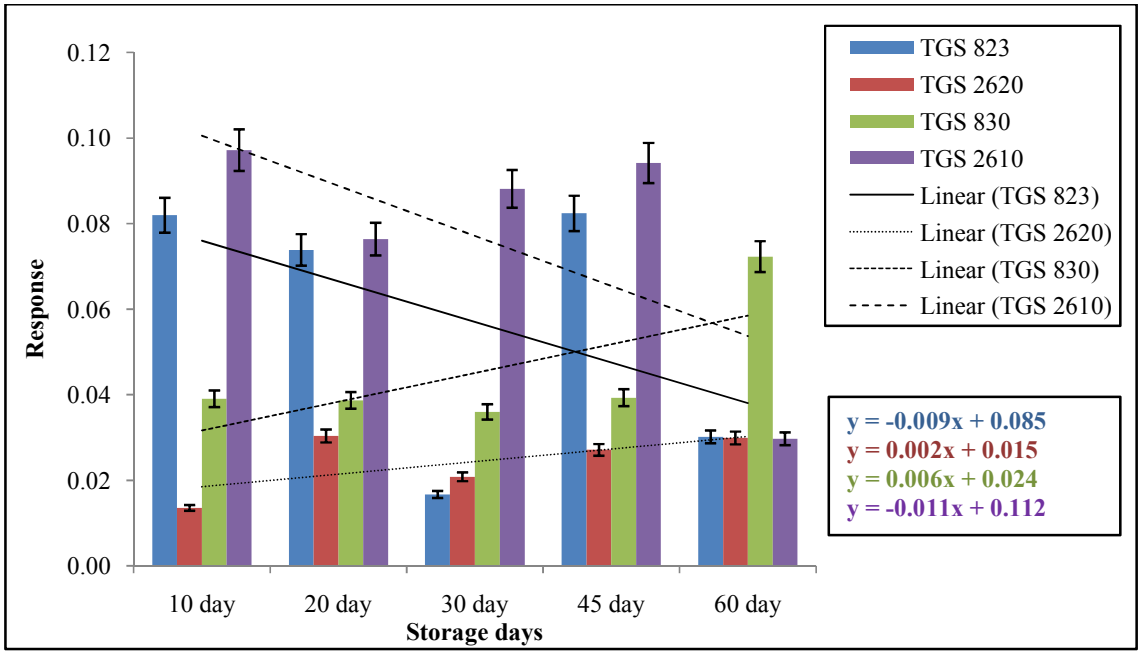

Figure 1(b): Plot variation of signal response with storage period of control fried banana chips over a time period of 60 days

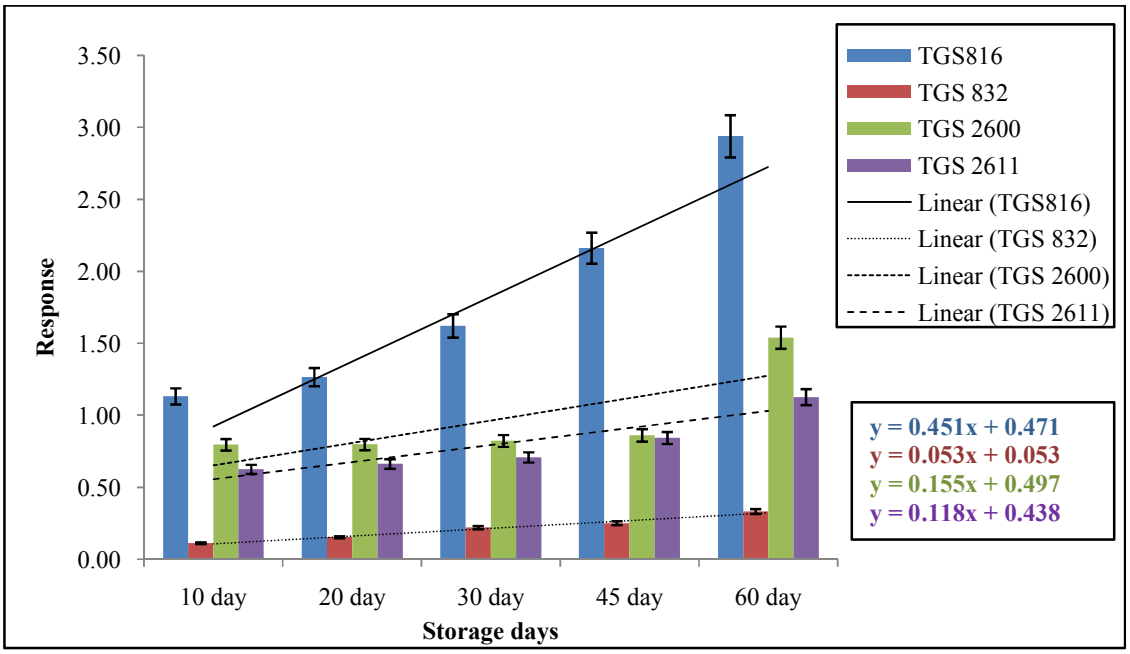

Figure 2(a): Plot variation of signal response with storage period of 'sample' fried banana chips over a time period of 60 days

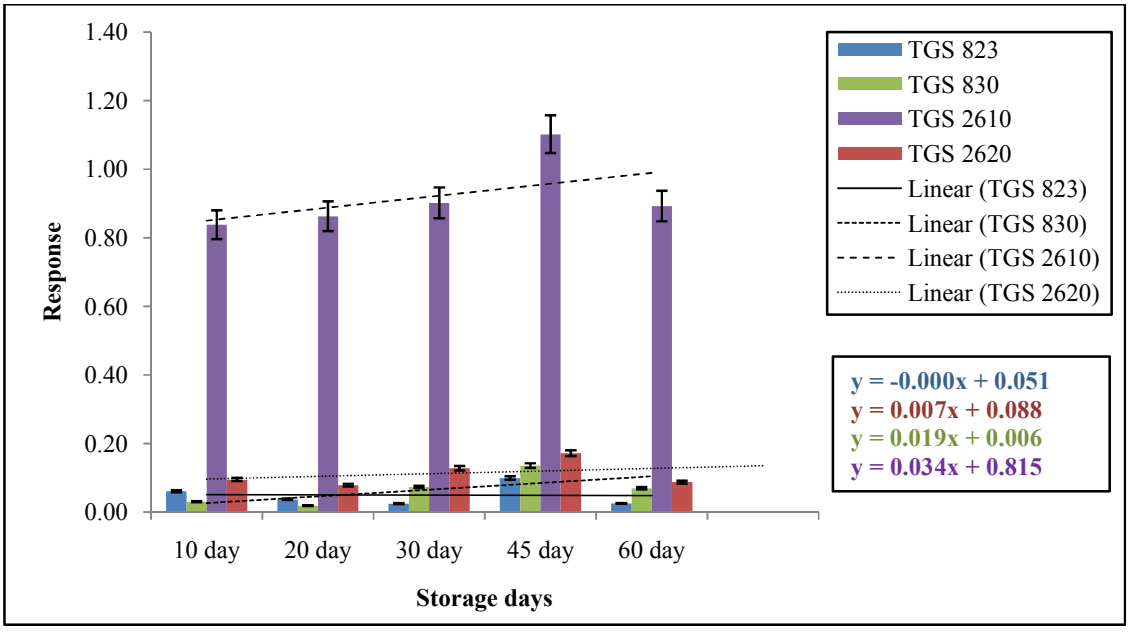

Figure 2(b): Plot variation of signal response with storage period of 'sample' fried banana chips over a time period of 60 days

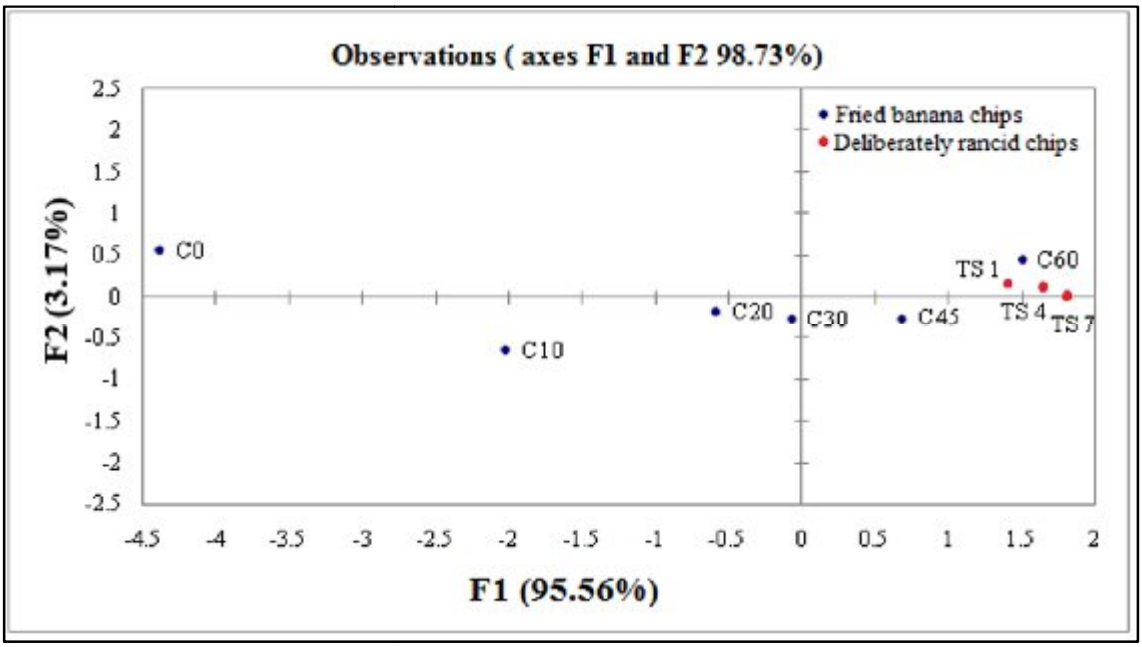

Figure 3: Odor map of different 'control' banana chips set on the basis of PCA using four metal oxide sensors of ENOVISION Ver.1.Q e-nose system

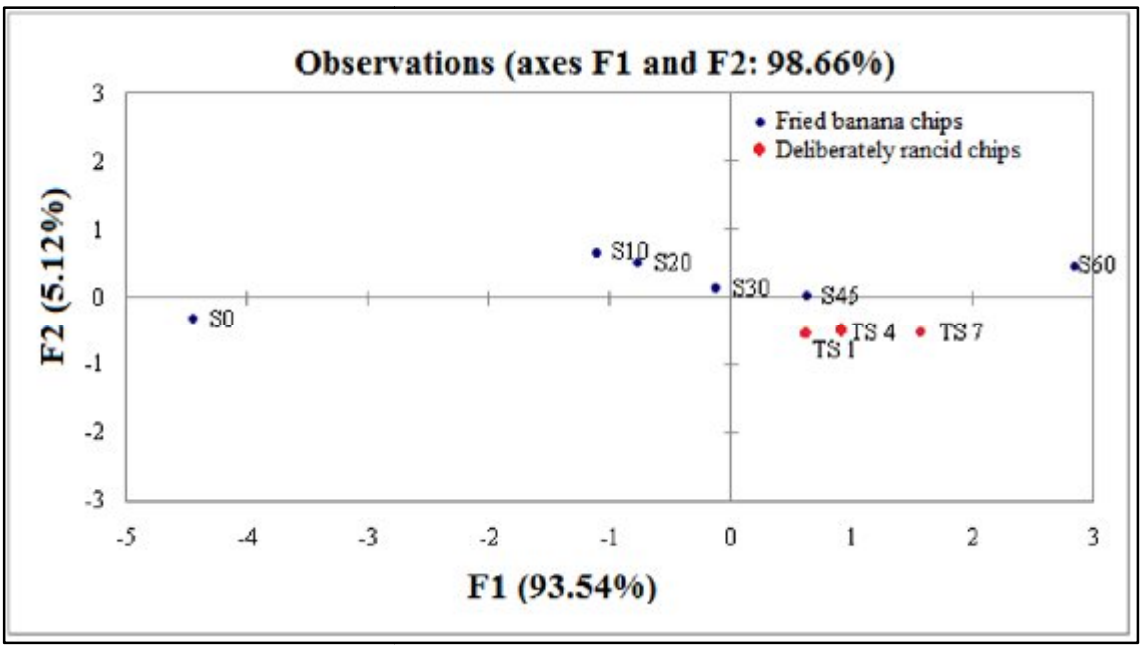

Figure 4: Odor map of different 'sample' banana chips set on the basis of PCA using four metal oxide sensors of ENOVISION Ver.1.Q e-nose system

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CHAPTER 2

GAMMA-IRRADIATED COCONUT OIL IN BAKING APPLICATION

Part-I

Biochemical Studies of Cookies

2.1.1

Introduction and Review of Literature

1. INTRODUCTION

1.1 Cookies

Cookies are one of the most popular bakery products consumed globally due to its ready-to-eat nature and high nutritional value, derived from the Dutch word koekie (little cake). It contains high amount of fat which imparts desirable organoleptic quality and also contributes to texture and flavour of the product. The fat is one of the main ingredients and influences lubrication, aeration, spread and eating quality besides sensory appeal (Blanshard *et al*., 1986). Commercially fat present in the cookies are mainly hydrogenated fat. Hydrogenated fat has adverse effect to human health (Ascherio and Willet, 1997). VCO on the other hand has impressive health benefits.

1.2 Advantage of coconut oil as a fat replacer

- The MCTs ($C_8 - C_{12}$) in coconut oil are similar in structure to the fats in mother's milk that gives babies immunity to diseases. There are also similar beneficial effects in adults, (Kabara *et al*., 2000).
- VCO possesses anti-inflammatory, anti-microbial and antioxidant properties which work together to protect arteries from atherosclerosis and the human heart from cardiovascular diseases (CDVs) (Fife *et al*., 2004).
- VCO protects against CDVs by increasing high density lipoprotein (HDL) that collects the excess or unused cholesterol in the body for excretion by the liver.
- VCO provides protection from infectious diseases not easily cured by known antibiotics.
- VCO is digested easily without the need for bile and goes directly to the liver for conversion into energy (Dayrit *et al*., 2003).
- VCO stimulates metabolism, boosts energy and prevents deposition of fats thereby preventing obesity (Dayrit *et al*., 2003).
- VCO improves the nutritional value of food by increasing absorption of vitamins, minerals and amino acids (Fife *et al*., 2004).
- VCO is the world's only natural, low-calorie fat (Fife *et al*., 2004).
- VCO inhibits the action of cancer-forming substances (Lim-Syliangco *et al*., 1987).

This part of the study endeavours to investigate formulation of cookies with irradiated VCO (with no obnoxious odor) as a replacer of hydrogenated fat for imparting health beneficial properties of the former and for acceptable flavour to cookies. Two sets of cookie samples have been formulated namely 'control' and 'sample' cookies. 'Sample' cookie was prepared with irradiated VCO in replacement of hydrogenated fat; whereas, the 'control' set was prepared with commercial VCO (non-irradiated). The ingredients for formulation of cookies using VCO as a replacer of hydrogenated fat have shown in Table 1.

Table 1: Formulation of cookies in replacement of hydrogenated oil with coconut oil

Ingredients	% (Wet basis, w/w of dough weight)
Wheat flour	50
Table salt	0.6
Ammonium bicarbonate	0.8
Sodium bicarbonate	0.4
Milk powder	2
Powder sugar	20
Coconut oil (as replacement of hydrogenated fat)	20
Butter	2
Water	8

The specific objectives of this work are as follows:

1. To assess suitability of γ-irradiated VCO in baking application.
2. To assess the biochemical changes in the cookies ('control' and 'sample') by routine assays.
3. Sensory evaluation (subjective study) of the cookies by semi-trained panelists consisting of university staff and students would be performed to assess the flavor harmony of the chips (contributed by copra flavour and rancid-acid octanoic acid odor of coconut oil) in both 'sample' and 'control' cookies in tandem with biochemical assays.
4. Assessment of shelf life of cookies by correlation of results obtained in points 1, 2 and 3.

2.1.2

Materials and Methods

2. MATERIALS AND METHODS

2.1 Reagents and Samples

Reagents and samples are as described in section 2.1 of Part I of Chapter 1. Baking was carried out in a baking air oven (Chanmag Bakery Machine Co., Ltd., Model CM-8108).

2.2 Irradiation of coconut oil

Irradiation of coconut oil was made as described in section 2.2 of Part I Chapter 1.

2.3 Manufacture of cookies

2.3.1 Dough making

Cookies were prepared as explained by Chatterjee *et al*., (2014), with few modifications. Wheat flour (50%), sugar (20%), VCO (20%), butter (2%), milk powder (2%), ammonium bicarbonate (0.8%), sodium bicarbonate (0.4%) and water (8%), all on w/w of dough weight (wet weight), constituted the dough for the 'control' cookie set. 'Sample' cookies were prepared with 4.2 kGy γ-irradiated VCO (20%) and the rest of the ingredients were similar to those of the 'control'. The flow chart for manufacture of the 'sample' cookie set is shown in Figure 1.

At first, the ingredients (wheat flour, sugar, milk powder, ammonium bicarbonate, table salt and sodium bicarbonate) were mixed in a container for 3-4 min and remixed with coconut oil, butter and water for another 5 min to make the dough. The dough was then manually shaped into circular pieces of diameter 50 mm and thickness of 10 mm.

2.3.2 Baking, cooling and storage

The dough was subsequently baked in a baking air oven at 190°C for 13 min and cooled to room temperature (23±2°C) to obtain ready-to-eat cookies. The cookies were packed in aluminium foil, placed in Ziploc pouches, flushed with nitrogen and stored at room temperature (23±2°C) for five months.

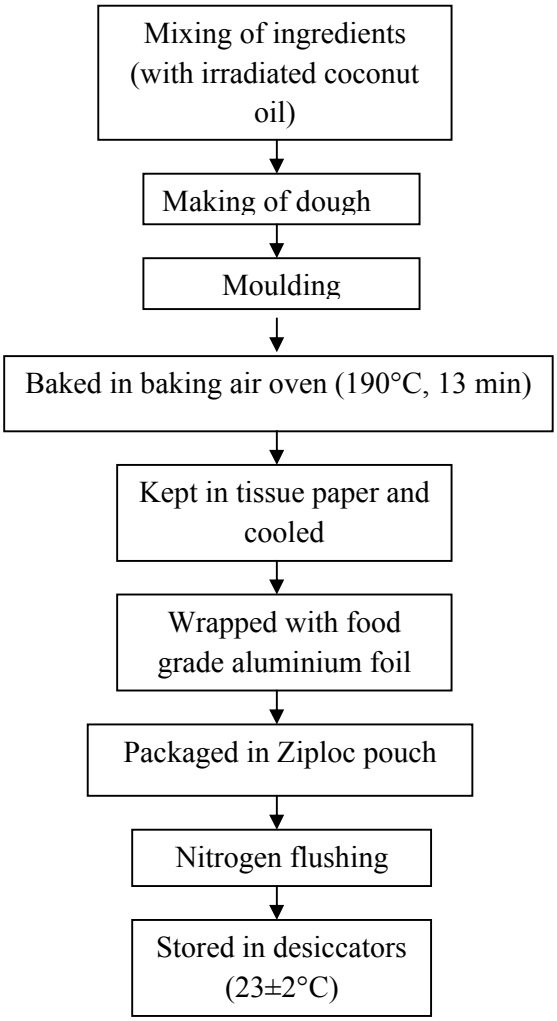

Figure 1: Schematic flow chart of preparation of 'sample' set of cookies

2.4 Product analysis

2.4.1 Chemical assay

2.4.1.1 Determination of free fatty acids

Determination of FFA was evaluated as described in section 2.4.1.1 of Chapter 1.

2.4.1.2 Peroxide value (PV)

Determination of PV was evaluated as described in section 2.4.1.2 of Chapter 1.

2.4.1.3 TBARS assay for malondialdehyde in cookies

Determination of malondialdehyde content was evaluated as described in section 2.4.1.3 of Chapter 1.

2.4.2 Storage studies of cookies for shelf life assessment

2.4.2.1 Storage

The methodology for storage of cookies was similar to that described in section 2.4.2.1 of Chapter 1. Cookies were stored for a total period of 150 days and they were withdrawn at an interval of 0, 10, 20, 30, 45, 60, 75, 90,120 and 150 days for assays. A set of cookies were made deliberately rancid and withdrawn for analyses at an interval of 1, 3, 10, 20 and 30 days as described by Chatterjee *et.al* (2014).

2.4.2.2 Sensory evaluation

Sensory evaluation was carried as described in section 2.4.2.2 of Chapter 1.

2.1.3

Results and Discussion

3. RESULTS AND DISCUSSION

3.1 Chemical analysis of products (cookies)

3.1.1 Acid value

The acid values (mg KOH/g oil and expressed as % oleic acid) of the cookies for the storage period of zero days to 150 days are shown in Table 1. The initial acid value of 0-day 'control' cookies was 0.34, which increased significantly ($p<0.05$) to 0.79 at the end of 150 days. FFA content of 'control' cookies until 150 days (0.79) of storage was lower than those of the 1-day stored deliberately rancid cookies (0.88).

However, the initial acid value of 0.33 for 0-day 'sample' cookie was much closer to that of 0-day 'control cookies'. The acid value of 'sample' cookies increased slowly and steadily to 0.58 at the end of 150 days storage period. FFA content of 'sample' cookie until 150 days (0.58) of storage was lower than the 1-day stored deliberately rancid 'sample' cookie (0.67). The difference in the FFA values was observed between 'control' and 'sample' cookies only at the end of the storage period, when 'control' cookie showed higher value (0.79) than that of the 'sample' (0.58). Therefore, 'sample' cookie was found to be more stable and less prone to develop rancidity than the 'control'. As discussed earlier, the increase in the acid values of oils or fat indicates hydrolysis of triglycerides (due to moisture, temperature or enzymes) (Arlee *et al*., 2013). According to Lawson (1985) and Che Man *et al*., (1997), hydrolysis is accelerated by high temperature and excessive amounts of water and therefore VCO produced by fermentation method has high FFA content. Although, we do not have supporting data in literature on usage of irradiated coconut oil in food products to substantiate our work, the trend observed in our investigation is in agreement with that obtained in our previous study on γ-irradiated coconut oil (Chatterjee, 2015).

3.1.2 PV

The PV values of 'control' cookies increased slowly and steadily until the end of the total storage period of 150 days. At the end of total storage period, it was found that the PV value of 'control' cookies was 0.0075 ppm which was lower than the 1-day stored deliberately rancid cookies (0.0085 ppm). In case of 'sample' cookies, at the end of 150 days, PV value was 0.0071 ppm which was lower than that of the 1-day stored deliberately rancid cookies

(0.0082 ppm) (Table 2). However, no significant difference was found in the PV values of 'control' and 'sample' cookies.

In the present study, PV is much less than the acceptable limit of 5 ppm. Since, cookies were stored in an inert atmosphere of $N_{2;}$ the spoilage of cookies cannot be accounted by primary oxidation alone. Therefore, primary oxidation cannot alone be considered for assessing spoilage in cookies. Hence, TBARS assay was also carried out to evaluate secondary oxidation products such as malondialdehyde in cookies.

3.1.3 TBARS assay for formation of malondialdehyde

The malondialdehyde content for 'control' cookies increased linearly from 0-day until the end of the total storage period of 150 days (Table 3). We observed maximum content of malondialdehyde (mmole malondialdehyde/g dried cookie) in cookies at the end of the storage period for both 'sample' and 'control' sets. The initial value of 0-day 'control' cookies was 1.09, which significantly ($p<0.05$) changed to 2.14 at the end of the storage period. The malondialdehyde content of 150 days-stored cookies was lower than that of the 1-day stored deliberately rancid cookies (2.21).

The initial value of 0-day 'sample' cookies (using irradiated coconut oil) was 0.85, which increased significantly ($p<0.05$) to 1.82 at the end of the storage period. The malondialdehyde content of 150 days-stored cookies was lower than that of the 1-day stored deliberately rancid cookies (2.19). From these observations, it could be stated that malondialdehyde content was lower in 'sample 'cookies compared to the 'control'. Therefore it could be stated that 'sample' cookies were more stable than 'control' since the secondary oxidation products have been generated in low amount in the former. At the same time, it could be stated that, 150 days-stored 'control' cookies were more prone to oxidative rancidity. The malondialdehyde values of the cookies were determined in accordance to the method reported by Chatterjee *et al*., (2014). To the best of our knowledge, there are no set standards and reports in literature that indicate the acceptable level of malondialdehyde in coconut oil cookies to enable comparison of our data.

3.1.4 Sensory analysis

The response of the panelists for the 'control' and 'sample' cookie sets with storage depicted that the acceptability of the latter was higher than the former. This is represented in the radar plots (Figures 1a and 1b).

There were no differences in overall appearance, colour and texture between 'control' and 'sample' cookies with storage. The products were adjudged based on odor, taste and aftertaste. The strong odor of coconut oil was milder in 'sample' cookies from the day zero to end of the storage than 'control', at the same time an obnoxious (rancid) type odour was found in 150 days-stored 'control' cookies, which was unaccepted by panelists. Panelists also found that the freshness flavour i.e., the copra flavour was milder and more acceptable for 'sample' cookies until the end of the storage period of 150 days and no obnoxious odor was detected in the sample set throughout the storage period. When cookies were analyzed on the 150^{th} day, there was generation of metallic odor and obnoxious coconut odor in 'control' cookies. Taste and aftertaste were better in 'sample' cookies having no metallic taste and had less oily mouth feel until the end of the storage study, vis-à-vis the control set. Thus from day zero until 150^{th} day of evaluation, 'sample' cookies were more acceptable than 'control' cookies. These results of sensory evaluation were in perfect agreement to those obtained by biochemical assay indicators of rancidity.

TABLES

Table 1: FFA content of 'control' (using non-irradiated oil) and 'sample' (using irradiated oil) cookies during storage period of 150 days and those of deliberately rancid cookies

Storage period (days)	FFA (% oleic acid)	
	CONTROL	SAMPLE
Cookies		
0	0.34±0.01[a]	0.33±0.01[a]
10	0.45±0.02[b]	0.34±0.01[a]
20	0.45±0.01[b]	0.34±0.02[a]
30	0.55±0.02[c]	0.44±0.02[b]
45	0.56±0.02[c]	0.45±0.02[b]
60	0.63±0.03[d]	0.45±0.02[b]
75	0.66±0.01[d]	0.50±0.02[c]
90	0.67±0.02[d]	0.56±0.02[d]
120	0.76±0.03[e]	0.56±0.02[d]
150	0.79±0.03[e]	0.58±0.02[d]
Deliberately made rancid cookies		
1	0.88±0.04[f]	0.67±0.03[e]
3	1.01±0.05[g]	0.75±0.03[f]
10	1.13±0.06[h]	0.79±0.04[f]
20	0.90±0.04[f]	0.90±0.04[g]
30	1.26±0.06[i]	1.01±0.05[h]

Mean ± S.D of three samples of one experimental set

[a-i] Different letters in a row indicate significant difference ($p<0.05$)

Table 2: PV (ppm) content of 'control' (using non-irradiated oil) and 'sample' (using irradiated oil) cookies during storage period of 150 days and those of deliberately rancid cookies

Storage period (days)	PV (ppm× 10^{-4})	
	CONTROL	SAMPLE
Cookies		
0	2±0.10[a]	2±0.10[a]
10	5±0.25[a]	3±0.15[a]
20	11±0.55[b]	19±0.95[b]
30	18±0.90[b]	20±1.00[b]
45	43±2.15[c]	39±1.95[c]
60	52±2.60[d]	53±2.65[d]
75	56±2.80[d]	54±2.70[d]
90	66±3.30[e]	67±3.35[e]
120	79±3.95[e,f]	77±3.85[f]
150	75±3.75[e,f]	71±3.55[e,f]
Deliberately made rancid cookies		
1	85±4.25[g]	82±4.10[g]
3	95±4.75[h]	85±4.25[g]
10	97±4.85[h]	86±4.30[g]
20	101±5.05[i]	91±4.55[g,h]
30	106±5.30[i,J]	94±4.70[h]

Mean ± S.D of three samples of one experimental set

[a-j] Different letters in a row indicate significant difference ($p<0.05$)

Table 3: Malondialdehyde (mmole/g dried sample) content of 'control' (using non-irradiated oil) and 'sample' (using irradiated oil) cookies during storage period of 150 days and those of deliberately rancid cookies

Storage period (days)	MDA (mmole/g dried sample)	
	CONTROL	SAMPLE
Cookies		
0	1.09±0.04[a]	0.85±0.03[a]
10	1.48±0.06[b]	0.96±0.04[a,b]
20	1.64±0.07[b,c]	1.03±0.04[a,b]
30	1.69±0.08[b,c]	1.12±0.05[b]
45	1.67±0.07[b,c]	1.41±0.06[c]
60	1.70±0.08[b,c]	1.43±0.06[c,d]
75	1.76±0.08[c,d]	1.59±0.07[c,d,e]
90	1.84±0.09[c,d]	1.62±0.08[d,e]
120	1.94±0.1[d,e]	1.66±0.08[e]
150	2.14±0.11[e,f]	1.82±0.09[f]
Deliberately made rancid cookies		
1	2.21±0.10[f]	2.19±0.09[g]
3	2.28±0.11[f]	2.21±0.10[g]
10	3.39±0.15[g]	3.11±0.14[h]
20	5.18±0.23[h]	4.62±0.23[i]
30	5.52±0.26[i]	5.18±0.24[j]

Mean ± S.D of three samples of one experimental set

[a-j] Different letters in a row indicate significant difference ($p<0.05$)

FIGURES

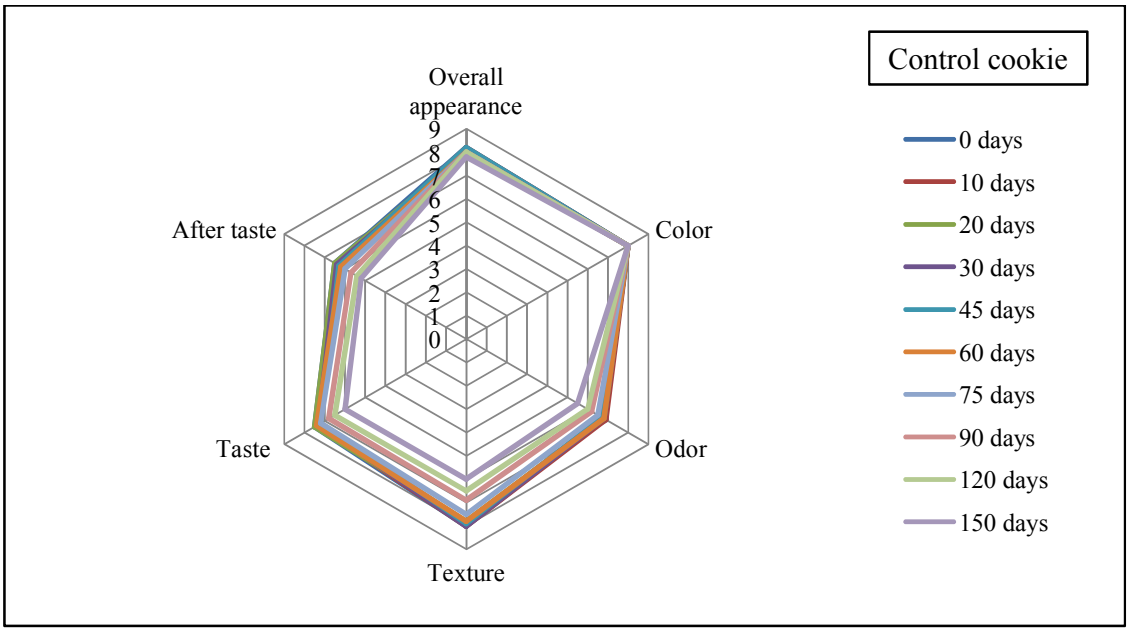

Figure 1(a): Effect of storage on sensory characteristics of 'control' cookies

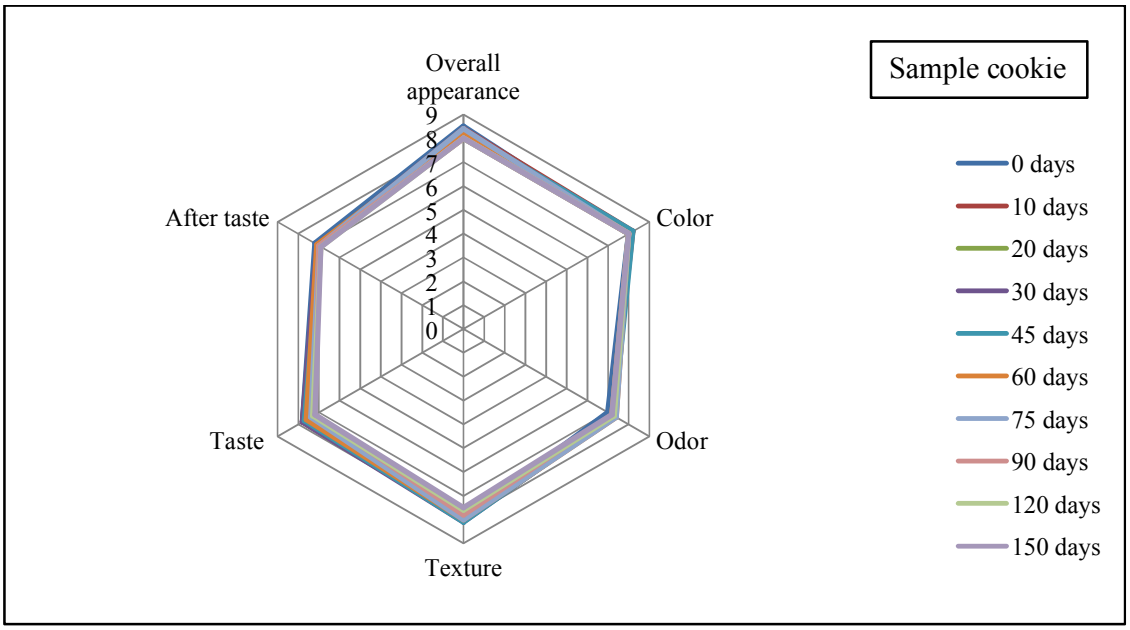

Figure 1(b): Effect of storage on sensory characteristics of 'sample' cookies

Part II

E-nose Analysis for Odor Detection of Cookies

2.2.1

Introduction and Review of Literature

1. INTRODUCTION

In our present study, we have employed irradiated coconut oil for another application that is 'baking', where cookies have been formulated using this oil as a replacement of hydrogenated fat (namely *vanaspati*). E-nose technology has been used for the same purpose as described in Chapter 1. Our research group had erstwhile reported on usage of e-nose for detection of rancidity and assessment of shelf life of 'antioxidant-rich cookies' prepared with hydrogenated oil (Chatterjee *et al*., 2014). To the best of our knowledge, γ-irradiated coconut oil has been used for the first time in baking application for formulation of cookies.

We have once again implemented the e-nose technology for assessment of the shelf life of cookies (formulated with 4.2 kGy irradiated coconut oil) as described in part II of Chapter 1. The specific objectives of this part of work are as follows:

1. E-nose analysis for determining shelf life of 'sample' cookies (irradiated coconut oil used as replacer of *vanaspati*) with storage in comparison with 'control' cookies (non-irradiated coconut oil used as replacer of *vanaspati*) by Principal component analysis (PCA).
2. Selection of best sensors which detected the odor profile best and ranking them to obtain the short listed sequence of sensors appropriate for analyzing cookie volatiles.
3. Establishment of co-relation between biochemical assay parameters of rancidity and e-nose analysis.

2.2.2

Materials and Methods

2. MATERIALS AND METHODS

2.1 Reagents and samples

The food materials comprised of commercial coconut oil (M/s KPL Oil Mills (P) Ltd., Kerala, India) and Wheat flour, Sugar, Butter, and Table Salt were purchased from a local supermarket of Kolkata, India. Reagents and samples are same as described in section 2.1 of Part I of Chapter 1.

2.2 Irradiation of coconut oil

Procedure of irradiation of coconut oil was made as described in section 2.2 of Part I of Chapter 1.

2.3 Preparation of cookies

Cookies ('sample' and 'control') were made as described in part I of chapter 2.

2.4 Optimization of ENOVISION (C-DAC) system

Optimization of e-nose system parameters was done as described in 2.4 of Part II of Chapter 1.

2.5 E-nose analysis of cookies

E-nose analysis (ENOVISION Ver.1.Q) of cookies ('sample' as well as 'control') was carried out at an interval of 0, 10, 15 and 30 days for a total storage period of 150 days.The procedure of analysis was similar to that described in section 2.5 of Part II of Chapter 1.

2.6 Storage of cookies

The cookies were stored as described in section 2.4.2.1 of Part I of chapter 2.

2.7 Selection and ranking of sensors

In order to screen the metal oxide gas sensors that are most sensitive towards VOCs generated from cookies, ranking of the sensors was carried out by plotting the graph between individual sensor responses with time (storage days). The signal responses ($\Delta R/R$) of 8 sensors were collected from cookies on 0, 10, 10, 20, 30, 45, 60, 75, 90, 120 and 150days of

storage. Among the 8 sensors, four sensors are selected based on high slope values and best fitted trend lines of the storage period vs. (|ΔR|/R) values of the sensors. Deliberately made rancid cookies for both 'control' and 'sample' have also been analysed on 1,3,10, 20, 30 days for supporting the best sensor data. Selection and ranking of sensors were conducted as described in section 2.7 of Part II of chapter 1.

2.8 Statistical analysis

Statistical analysis was done as described in section 2.8 of Part II of chapter 1.

2.2.3

Results and Discussion

3. RESULTS AND DISCUSSION

3.1 Optimization of electronic nose system

Ground cookies of 50gm (dp = 1.5 mm) were placed in a 100 ml glass vial and heated for 450 s at 50°C. Rest of the procedure was done as described in section 3.1 of Part II of chapter 1.

3.2 Selection and ranking of sensors

The signal responses of the individual eight sensors for 'sample' and 'control' cookies over a time period of 150 days on 0, 10, 10, 20, 30, 45, 60, 75, 90, 120 and 150days are represented in Figures 1a, 1b, 2a and 2b. A graph was plotted taking time period in X-axis and resistance in Y-axis for both types of cookies and their trend lines and slope equations were deduced.

We clearly found that TGS 816, TGS 830, TGS 832 and TGS 2600 gave higher responses for the VOCs generated from for both 'sample' and 'control' cookies (Figures 2a and 1a). These four sensors are generally used for detecting combustible gases, methane, butane, propane CFC, air contaminants and ethanol, are found to be more specific for detecting VOCs from cookies. E-nose results were analyzed with data obtained from these screened sensors.

It has been previously established that TGS 823 is the best sensor for coconut oil flavour (copra flavour) and octanoic acid (rancid-acid aroma), reportedly known to detect alcoholic compounds and solvent vapors. In our present study, a particular sensor response for the odor analysis has not been considered owing to the fact that cookies also contains different volatile compounds from different sources, namely, native coconut oil and other cookie ingredients. Since the flavor harmony of the cookies would be owing to contribution of all flavors, we have holistically considered the response of the entire sensor array and four sensors from them have been selected based on their high values of ($|\Delta R|/R$).

3.3 Shelf life estimation by PCA analysis

An odor map was generated on the basis of PCA plot (Figures 3 and 4) with discrimination indices of 99.28% and 99.15%for 'control' and 'sample' cookies, respectively, indicating the clusters to be distinctly separated. It was observed that all the 'sample' cookies with different storage periods have formed a well-defined cluster along with the 'control'. The 'control' cookies with storage period of 150 days (C150) and less formed a distinct cluster and was

present in the same quadrant along with the cluster of 'deliberately-made rancid cookies (TS1)'. Also C150 cookies were very close to TS1 set. Therefore, it could be concluded that 150 days-stored 'control' cookies had similar odor profile as that of the rancid cookies, indicating that 'control' cookies were more prone to become rancid.

120 and 150-days stored (S120, S150) 'sample' cookies formed a distinct cluster and was present in the same quadrant along with the cluster of 'deliberately-made rancid cookies' and it was also observed that S150 cookies had maintained a distance from the cluster of TS1. Therefore, it could be concluded that 'sample' cookies even on 150^{th} day of storage remained unspoilt.

3.4 Correlation between the rancidity parameters of cookies determined by e-nose analysis and biochemical assays

From the experimental data, a linear correlation was found among the responses ofthe different sensors of e-nose system and rancidity parameters obtained by biochemical assays. Therefore, multiple regression analyses were carried out to develop three linear regression equations to predict the FFA, PV and TBA values (individually) of the cookies as a function of responses of four metal oxide gas sensors (TGS 816, TGS 832, TGS 832 and TGS 2600) which are provided below.

$$Y_1 = 0.2953 - 0.0858 \times X_1 - 0.3538 \times X_2 + 1.7882 \times X_3 + 0.2041 \times X_4 \quad (1)$$

$$Y_2 = -0.0004 - 0.0165 \times X_1 + 0.0472 \times X_2 - 0.0014 \times X_3 + 0.0386 \times X_4 \quad (2)$$

$$Y_3 = 0.8062 - 1.4512 \times X_1 + 3.2785 \times X_2 - 2.3149 \times X_3 + 3.9042 \times X_4 \quad (3)$$

where, Y_1 is the FFA value of the 'sample' cookies (% oleic acid), Y_2 is the PV value of the 'sample' cookies (ppm) and Y_3 is the TBA value of the 'sample' cookies (mmol malondialdehyde/g dried chips).X_1, X_2, X_3 and X_4 are the e-nose responses obtained from sensor TGS 816, TGS 830, TGS 832 and TGS 2600 respectively. The correlation coefficients obtained from eqs. 1-3 are 0.96, 0.95 and 0.96 respectively. The validity of the fitted models were indicated by the insignificant lack of fit ($p<0.1$) for these equations and by F-values of 15.37, 13.69 and 15.47 corresponding to eqs. 1, 2 and 3 respectively. Therefore, these equations obtained from sensor data analysis of ENOVISION, could successfully be used to

predict the conventional rancidity parameters of the cookies since the e-nose sensor responses well correlated with the results of the biochemical assays for the entire storage period of 150 days.

2.4

Overall findings and conclusion

4. Overall findings and conclusion

The quality attributes of the 'control' and 'sample' cookies throughout the total storage period of 150 days were evaluated in this study using different parameters. Biochemical studies indicated that 'sample' cookies are more acceptable in comparison with the 'control' set. The assays also indicated that FFA, PV and malondialdehyde contents are less in 'sample' rather than in 'control' cookies and also sensory panel acceptability was more for 'sample' cookies. Significant ($p<0.05$) changes were observed in the above biochemical parameters for both 'control' and 'sample' cookies during storage. From this study, it has been found that shelf life of 'sample' cookies was higher than the 'control'; whereas, a metallic odor was found in 'control' cookies at end of storage.

The shelf lives of the cookies have been ascertained by PCA analysis. By comparison of 'control' and 'sample' PCA plots, it could be clearly stated that 'sample' cookies have better shelf life compared to 'control'. Moreover, from the biochemical and sensory analyses of either set of cookies in tandem with e-nose analysis, we have holistically recommended that 'sample' cookies are good for health owing to their lower malondialdehyde content and higher shelf life. Three regression equations have been established which could be used to predict the conventional rancidity parameters of the cookies from e-nose analyses. This can forego routine biochemical assays of cookies.

Therefore, this study will help the baking industry to use irradiated coconut oil in entire India breaking the closed boundary of southern India. Therefore, adjudged holistically, 4.2 kGy irradiated coconut oil can be safely recommended for baking application.

TABLES

Table 1: E-nose sensor responses (|ΔR|/R) of 'control' cookies during the storage period of 150 days

Storage period (Days)	Sensor Response(|ΔR|/R)							
	TGS 816	TGS 832	TGS 2600	TGS 2611	TGS 823	TGS 2620	TGS 830	TGS 2610
0	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
10	0.3978	0.0179	0.0233	0.1068	0.0364	0.0806	0.0140	0.0310
20	0.4884	0.0272	0.0254	0.1900	0.0532	0.1568	0.0290	0.1073
30	0.5554	0.0328	0.0313	0.1680	0.0164	0.1234	0.0419	0.1559
45	0.5565	0.0354	0.0450	0.1080	0.0284	0.1925	0.0579	0.1008
60	0.5679	0.0423	0.0791	0.1048	0.0166	0.1008	0.0836	0.1888
75	0.5958	0.0590	0.0928	0.1290	0.0013	0.1085	0.0840	0.1792
90	0.5961	0.0687	0.1002	0.0870	0.0511	0.0930	0.1052	0.0241
120	0.6192	0.0753	0.1011	0.1160	0.0209	0.1349	0.1110	0.0436
150	0.6557	0.0860	0.1410	0.2090	0.0932	0.2431	0.1305	0.2132

Table 2: E-nose sensor responses (|ΔR|/R) of 'sample' cookies during the storage period of 150 days

Storage period (Days)	Sensor Response (|ΔR|/R)							
	TGS 816	TGS 832	TGS 2600	TGS 2611	TGS 823	TGS 2620	TGS 830	TGS 2610
0	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
10	0.0449	0.0421	0.0260	0.0591	0.0170	0.0194	0.0186	0.0577
20	0.1046	0.0582	0.0294	0.0531	0.0550	0.0870	0.0821	0.1808
30	0.1725	0.0847	0.0395	0.0081	0.0628	0.0557	0.0905	0.1731
45	0.1969	0.0840	0.0679	0.0764	0.0188	0.0872	0.1120	0.3540
60	0.2131	0.0913	0.0699	0.1133	0.1117	0.1368	0.1128	0.0087
75	0.2284	0.1066	0.0831	0.0023	0.0132	0.0770	0.1129	0.0354
90	0.2522	0.1326	0.1134	0.0662	0.0890	0.0944	0.1319	0.02176
120	0.3887	0.1421	0.1548	0.0879	0.0583	0.0618	0.1644	0.1947
150	0.4402	0.1625	0.1911	0.0211	0.0136	0.0197	0.1730	0.0860

Table 3: E-nose sensor responses (|ΔR|/R) of deliberately made rancid 'control' cookies for best four sensors during the storage period of 30 days

Storage period (Days)	Sensor Response (|ΔR|/R)			
	TGS 816	TGS 830	TGS 832	TGS 2600
0	0.0000	0.0000	0.0000	0.0000
1	0.6325	0.1746	0.1105	0.1567
3	0.6536	0.2430	0.1714	0.3111
10	0.7047	0.4595	0.2381	0.4991
20	0.7769	0.7114	0.5124	0.6296
30	0.8491	0.9870	0.8571	0.9667

Table 4: E-nose sensor responses (|ΔR|/R) of deliberately made rancid 'sample' cookies for best four sensors during the storage period of 30 days

Storage period (Days)	Sensor Response (|ΔR|/R)			
	TGS 816	TGS 830	TGS 832	TGS 2600
0	0.0000	0.0000	0.0000	0.0000
1	0.4908	0.2098	0.1942	0.2534
3	0.5158	0.2491	0.2022	0.2687
10	0.5802	0.2986	0.2177	0.3188
20	0.6757	0.3339	0.2322	0.3910
30	0.7523	0.4782	0.2961	0.4456

FIGURES

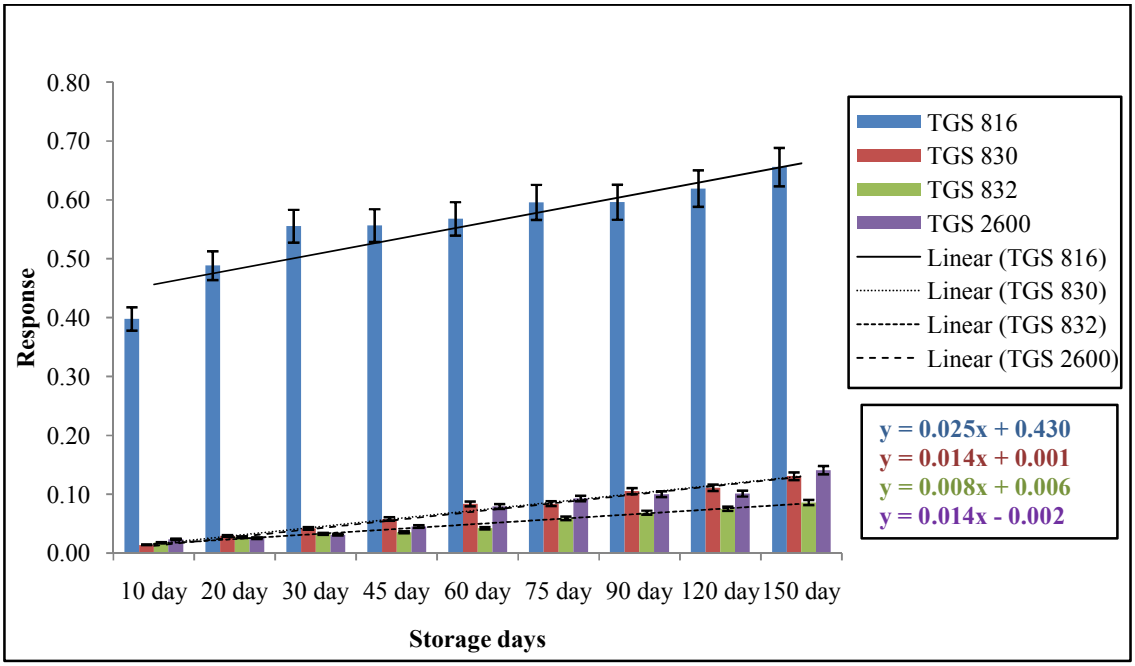

Figure 1(a): Plot variation of signal response with storage period of 'control' cookies over a time period of 150 days

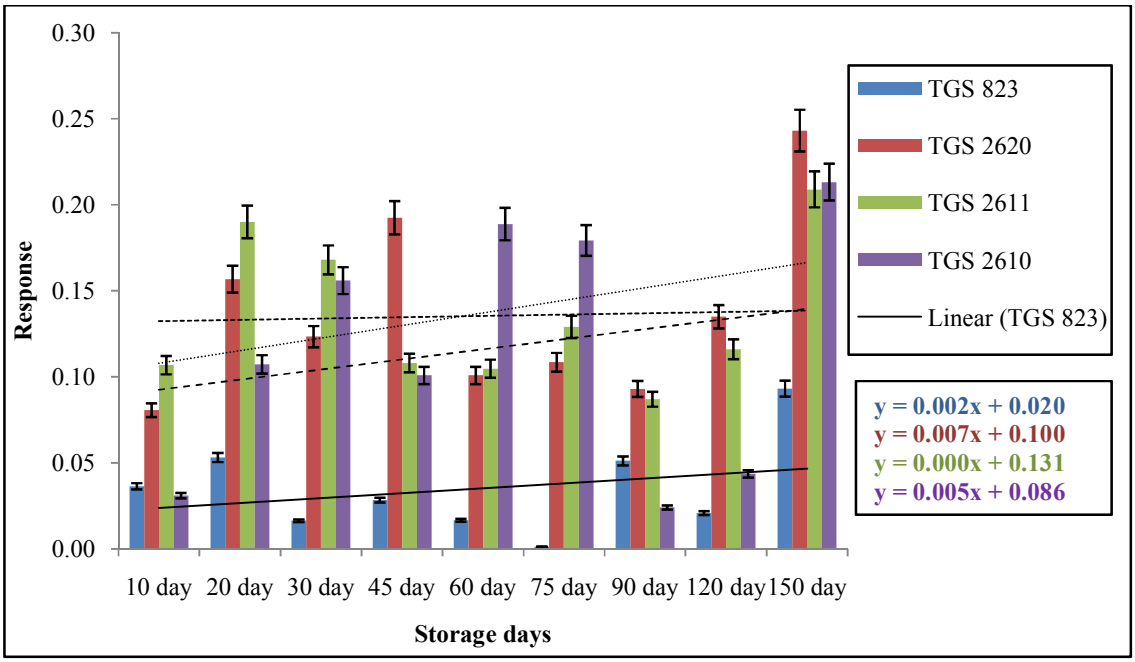

Figure 1(b): Plot variation of signal response with storage period of control cookies over a time period of 150 days

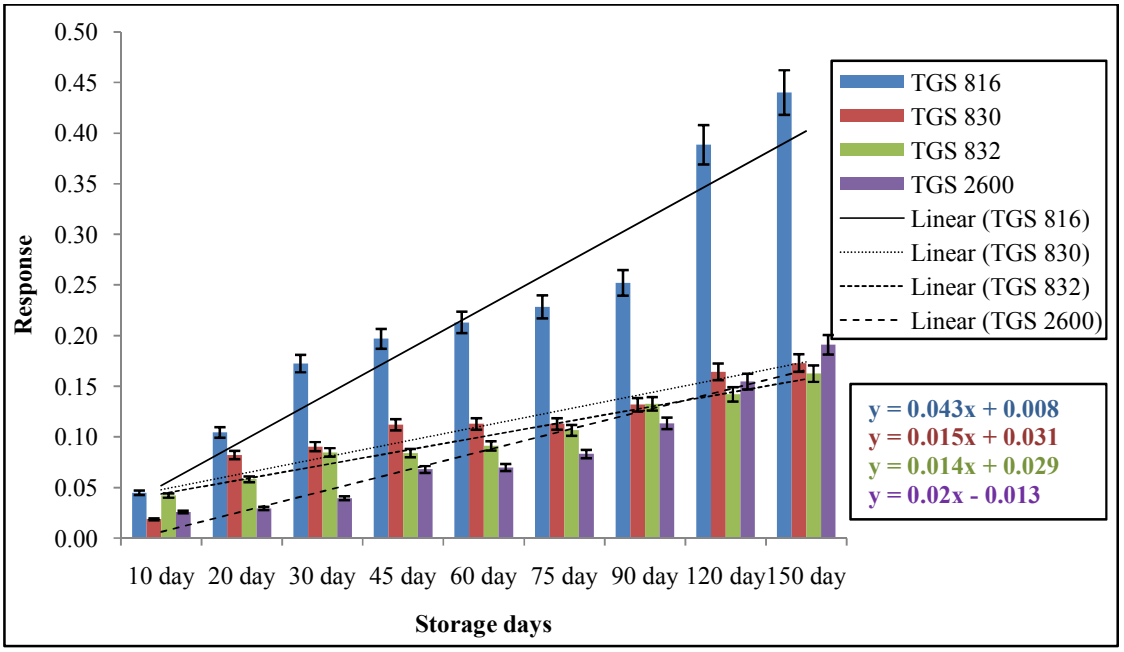

Figure 2(a): Plot variation of signal response with storage period of 'sample' cookies over a time period of 150 days

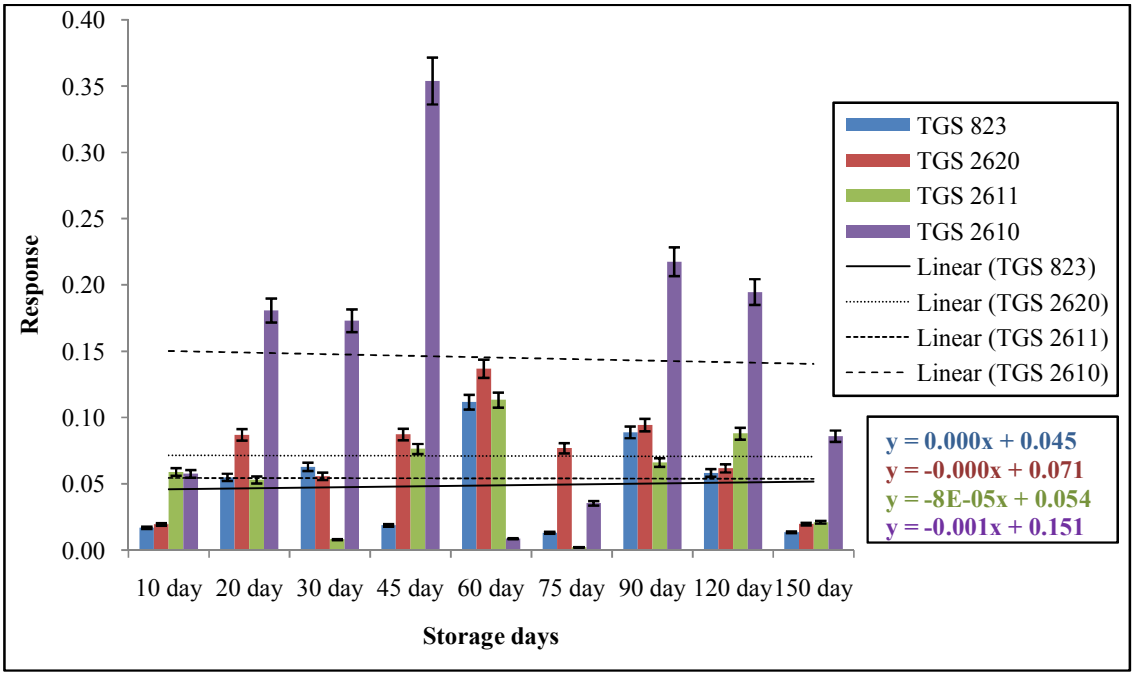

Figure 2(b): Plot variation of signal response with storage period of 'sample' cookies over a time period of 150 days

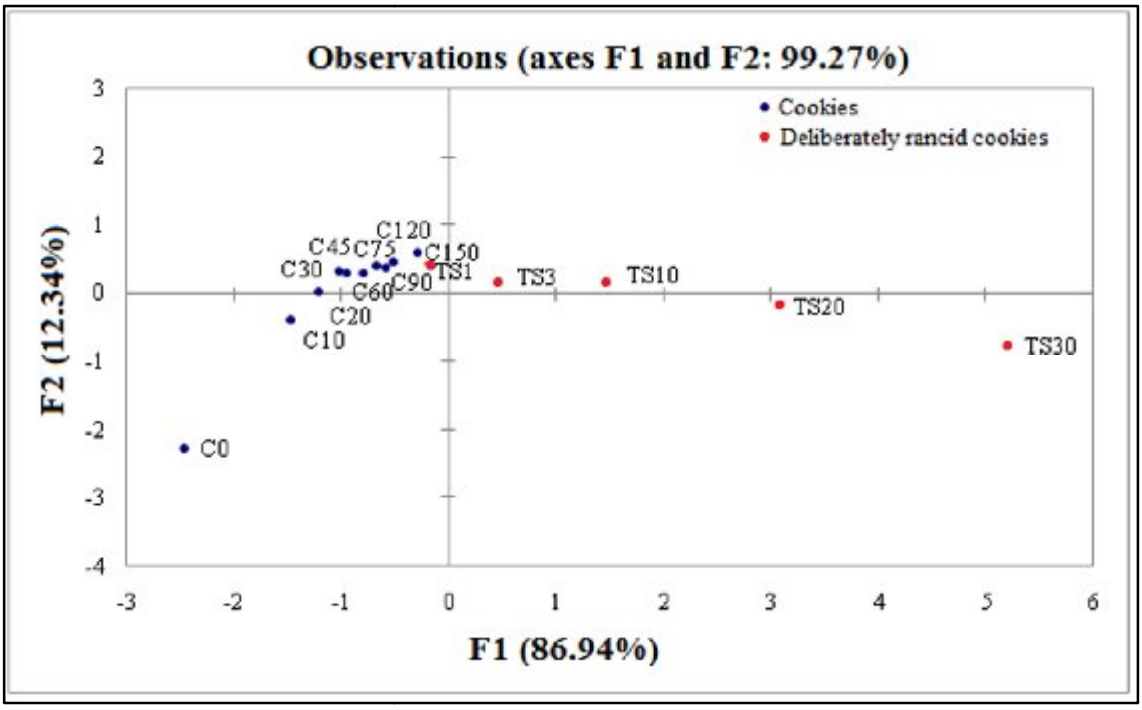

Figure 3: Odor map of different 'control' cookies set on the basis of PCA using four metal oxide sensors of ENOVISION Ver.1.Q e-nose system

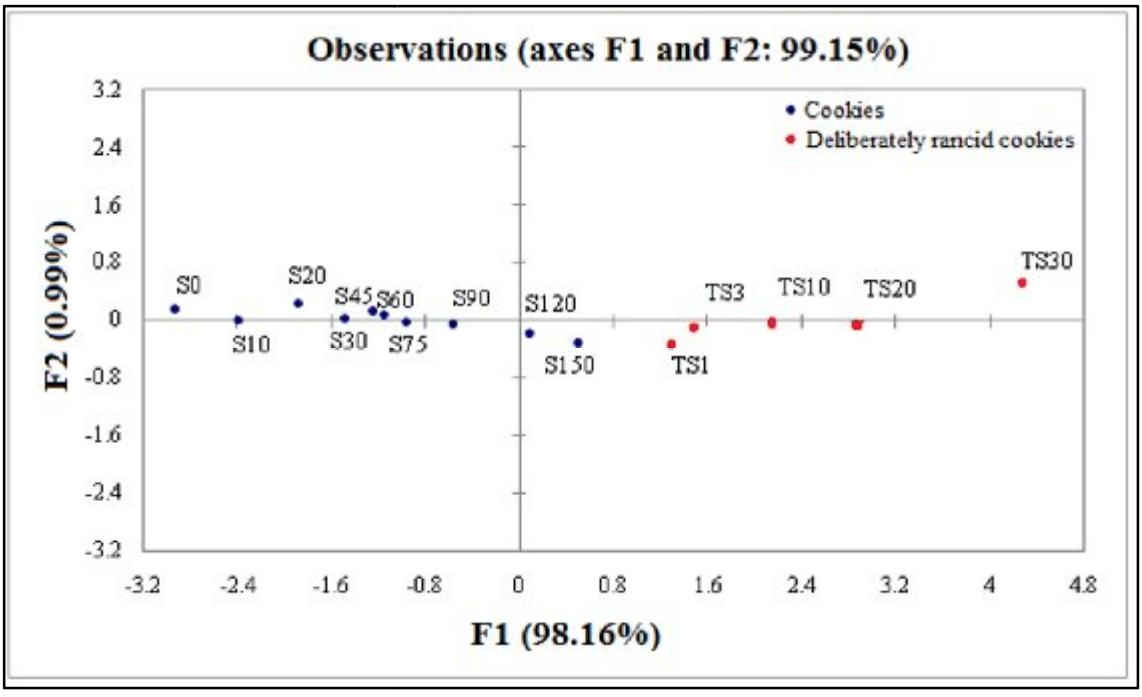

Figure 4: Odor map of different 'sample' cookies set on the basis of PCA using four metal oxide sensors of ENOVISION Ver.1.Q e-nose system

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SUMMARY AND FUTURE PROSPECTS

This study focused on application of 4.2 kGy γ-irradiated partially deodorized coconut oil after storage for 28 days in formulation of fried and baked food products such as banana chips and cookies, respectively. Assays of biochemical and sensory properties of fried banana chips samples over a storage period of 60 days, established that the chips fried in irradiated coconut oil was unhealthy and inferior over its non-irradiated counterpart. Whereas the biochemical and sensory properties of cookies over a storage period of 150 days, established that cookies formulated with γ-irradiated coconut oil was healthy and superior over its non-irradiated counterpart. The odor profile and shelf life analysis using e-nose technology also concluded that baking was most suitable for application of γ-irradiated coconut oil at 4.2 kGy dose than for frying application.

E-nose technology could become an aid for the extrusion and bakery industries for on-line quality assessment. The method developed in this investigation can replace cumbersome analytical processes for detection of shelf life and odor profile in fried banana chips and cookies. This work will boost coconut oil producing industries since they can cater to a larger consumer base, nationally and globally.

However, to detect particular odor of a sample, there is a need to customise the sensor of e-nose. In our current study, we have holistically considered the entire sensor array of the e-nose device since we did not possess any particular standardised sensor, specific for coconut oil, banana and cookie volatiles. This leaves scope for improvement in the design/selection of specific sensors for e-nose.

This state of the technology can also be applied to other food products, where sensory evaluation by human olfactory system plays a crucial role in quality assessment. It can also be used in process monitoring, freshness evaluation, authenticity assessment and other quality control studies. This study will enable better evaluation of odor profile of different food products more accurately and will also help understand the odor-impact compounds in food samples, foregoing routine chemical assays.