Principles of Food Analysis

Food analysis is the discipline dealing with the development, application and study of analytical procedures for characterising the properties of foods and their constituents. These analytical procedures are used to provide information about a wide variety of different characteristics of foods, including their composition, structure, physicochemical properties and sensory attributes. This information is critical to our rational understanding of the factors that determine the properties of foods, as well as to our ability to economically produce foods that are consistently safe, nutritious and desirable and for consumers to make informed choices about their diet.

Foods are analysed by scientists working in all of the major sectors of the food industry including food manufacturers, ingredient suppliers, analytical service laboratories, government laboratories, and University research laboratories.

Government Regulations and Recommendations

Government regulations and recommendations are designed to maintain the general quality of the food supply, to ensure the food industry provides consumers with foods that are wholesome and safe, to inform consumers about the nutritional composition of foods so that they can make knowledgeable choices about their diet, to enable fair
competition amongst food companies, and to eliminate economic fraud.

There are a number of Government Departments Responsible for regulating the composition and quality of foods, including the Food and Drug Administration (FDA), the United States Department of Agriculture (USDA), the National Marine Fisheries Service (NMFS) and the Environmental Protection Agency (EPA). Each of these government agencies is responsible for regulating particular sectors of the food industry and publishes documents that contain detailed information about the regulations and recommendations pertaining to the foods produced within those sectors.

**Voluntary and Mandatory Standards**

Government agencies have specified a number of voluntary and mandatory standards concerning the composition, quality, inspection, and labelling of specific food products.

**Mandatory Standards:**

- **Standards of Identity:** These regulations specify the type and amounts of ingredients that certain foods must contain if they are to be called by a particular name on the food label. For some foods there is a maximum or minimum concentration of a certain component that they must contain, e.g., “peanut butter” must be less than 55% fat, “ice-cream” must be greater than 10% milk fat, “cheddar cheese” must be greater than 50% milk fat and less than 39% moisture.

- **Standards of Quality:** Standards of quality have been defined for certain foods (e.g., canned fruits and vegetables) to set minimum requirements on the colour, tenderness, mass and freedom from defects.

- **Standards of Fill-of-Container:** These standards state how full a container must be to avoid consumer deception, as well as specifying how the degree of fill is measured.

**Voluntary Standards:**
Standards of Grade: A number of foods, including meat, dairy products and eggs, are graded according to their quality, e.g. from standard to excellent. For example meats can be graded as “prime”, “choice”, “select”, “standard” etc according to their origin, tenderness, juiciness, flavour and appearance. There are clear definitions associated with these descriptors that products must conform to before they can be given the appropriate label. Specification of the grade of a food product on the label is voluntary, but many food manufacturers opt to do this because superior grade products can be sold for a higher price. The government has laboratories that food producers send their products too to be tested to receive the appropriate certification. This service is requested and paid for by the food producer.

Nutritional Labelling and Education Act

In 1990, the US government passed the Nutritional Labelling and Education Act (NLEA), which revised the regulations pertaining to the nutritional labelling of foods, and made it mandatory for almost all food products to have standardised nutritional labels. One of the major reasons for introducing these regulations was so that consumers could make informed choices about their diet. Nutritional labels state the total calorific value of the food, as well as total fat, saturated fat, cholesterol, sodium, carbohydrate, dietary fibre, sugars, protein, vitamins, calcium and iron.

The label may also contain information about nutrient content claims (such as “low fat”, “low sodium” “high fibre” “fat free” etc), although government regulations stipulate the minimum or maximum amounts of specific food components that a food must contain if it is to be given one of these nutrient content descriptors. The label may also contain certain FDA approved health claims based on links between specific food components and certain diseases. The information provided on the label can be used by consumers to plan a nutritious and balanced diet, to avoid over consumption of food components.
linked with health problems, and to encourage greater consumption of foods that are beneficial to health.

**Authenticity**

The price of certain foods is dictated by the quality of the ingredients that they contain. For example, a packet of premium coffee may claim that the coffee beans are from Columbia, or the label of an expensive wine may claim that it was produced in a certain region, using a certain type of grapes in a particular year. There are many instances in the past where manufacturers have made false claims about the authenticity of their products in order to get a higher price. It is therefore important to have analytical techniques that can be used to test the authenticity of certain food components, to ensure that consumers are not the victims of economic fraud and that competition among food manufacturers is fair.

**Food Inspection and Grading Services**

The government has a Food Inspection and Grading Service that routinely analyses the properties of food products to ensure that they meet the appropriate laws and regulations. Hence, both government agencies and food manufacturers need analytical techniques to provide the appropriate information about food properties. The most important criteria for this type of test are often the accuracy of the measurements and the use of an official method.

The government has recently carried out a survey of many of the official analytical techniques developed to analyse foods, and has specified which techniques must be used to analyse certain food components for labelling purposes. Techniques have been chosen which provide accurate and reliable results, but which are relatively simple and inexpensive to perform.

**Analysing Foods**

One of the most important reasons for analysing foods from both the consumers and the manufacturers standpoint is to ensure that they are safe. It would be economically disastrous,
as well as being rather unpleasant to consumers, if a food manufacturer sold a product that was harmful or toxic. A food may be considered to be unsafe because it contains harmful microorganisms, toxic chemicals or extraneous matter. It is therefore important that food manufacturers do everything they can to ensure that these harmful substances are not present, or that they are effectively eliminated before the food is consumed.

This can be achieved by following “good manufacturing practice” regulations specified by the government for specific food products and by having analytical techniques that are capable of detecting harmful substances. In many situations it is important to use analytical techniques that have a high sensitivity, i.e., that can reliably detect low levels of harmful material. Food manufacturers and government laboratories routinely analyse food products to ensure that they do not contain harmful substances and that the food production facility is operating correctly.

**Quality Control**

The food industry is highly competitive and food manufacturers are continually trying to increase their market-share and profits. To do this they must ensure that their products are of higher quality, less expensive, and more desirable than their competitors, whilst ensuring that they are safe and nutritious. To meet these rigorous standards food manufacturers need analytical techniques to analyse food materials before, during and after the manufacturing process to ensure that the final product meets the desired standards. In a food factory one starts with a number of different raw materials, processes them in a certain manner (e.g. heat, cool, mix, dry), packages them for consumption and then stores them. The food is then transported to a warehouse or retailer where it is sold for consumption.

One of the most important concerns of the food manufacturer is to produce a final product that consistently has the same overall properties, i.e. appearance, texture, flavour and shelf life. When we purchase a particular food
product we expect its properties to be the same (or very similar) to previous times, and not to vary from purchase-to-purchase. Ideally, a food manufacture wants to take the raw ingredients, process them in a certain way and produce a product with specific desirable properties. Unfortunately, the properties of the raw ingredients and the processing conditions vary from time to time which causes the properties of the final product to vary, often in an unpredictable way.

How can food manufacturers control these variations? Firstly, they can understand the role that different food ingredients and processing operations play in determining the final properties of foods, so that they can rationally control the manufacturing process to produce a final product with consistent properties. This type of information can be established through research and development work. Secondly, they can monitor the properties of foods during production to ensure that they are meeting the specified requirements, and if a problem is detected during the production process, appropriate actions can be taken to maintain final product quality.

Characterisation of raw materials: Manufacturers measure the properties of incoming raw materials to ensure that they meet certain minimum standards of quality that have previously been defined by the manufacturer. If these standards are not met the manufacturer rejects the material. Even when a batch of raw materials has been accepted, variations in its properties might lead to changes in the properties of the final product. By analysing the raw materials it is often possible to predict their subsequent behaviour during processing so that the processing conditions can be altered to produce a final product with the desired properties. For example, the colour of potato chips depends on the concentration of reducing sugars in the potatoes that they are manufactured from: the higher the concentration, the browner the potato chip. Thus it is necessary to have an analytical technique to measure the concentration of reducing sugars in the potatoes so that the frying conditions can be altered to produce the optimum coloured potato chip.
Monitoring of food properties during processing: It is advantageous for food manufacturers to be able to measure the properties of foods during processing. Thus, if any problem develops, then it can be quickly detected, and the process adjusted to compensate for it. This helps to improve the overall quality of a food and to reduce the amount of material and time wasted. For example, if a manufacturer were producing a salad dressing product, and the oil content became too high or too low they would want to adjust the processing conditions to eliminate this problem. Traditionally, samples are removed from the process and tested in a quality assurance laboratory.

This procedure is often fairly time-consuming and means that some of the product is usually wasted before a particular problem becomes apparent. For this reason, there is an increasing tendency in the food industry to use analytical techniques which are capable of rapidly measuring the properties of foods on-line, without having to remove a sample from the process. These techniques allow problems to be determined much more quickly and therefore lead to improved product quality and less waste. The ideal criteria for an on-line technique is that it be capable of rapid and precise measurements, it is non-intrusive, it is nondestructive and that it can be automated.

Characterisation of final product: Once the product has been made it is important to analyse its properties to ensure that it meets the appropriate legal and labelling requirements, that it is safe, and that it is of high quality. It is also important to ensure that it retains its desirable properties up to the time when it is consumed. A system known as Hazard Analysis and Critical Control Point (HACCP) has been developed, whose aim is to systematically identify the ingredients or processes that may cause problems (hazard analysis), assign locations (critical control points) within the manufacturing process where the properties of the food must be measured to ensure that safety and quality are maintained, and to specify the appropriate action to take if a problem is identified.
The type of analytical technique required to carry out the analysis is often specified.

**Properties Analysed**

Food analysts are interested in obtaining information about a variety of different characteristics of foods, including their composition, structure, physicochemical properties and sensory attributes.

**Composition of Food**

The composition of a food largely determines its safety, nutrition, physicochemical properties, quality attributes and sensory characteristics. Most foods are compositionally complex materials made up of a wide variety of different chemical constituents. Their composition can be specified in a number of different ways depending on the property that is of interest to the analyst and the type of analytical procedure used: specific atoms; specific molecules, types of molecules, or specific substances. Government regulations state that the concentration of certain food components must be stipulated on the nutritional label of most food products, and are usually reported as specific molecules or types of molecules.

The structural organisation of the components within a food also plays a large role in determining the physicochemical properties, quality attributes and sensory characteristics of many foods. Hence, two foods that have the same composition can have very different quality attributes if their constituents are organised differently. For example, a carton of ice cream taken from a refrigerator has a pleasant appearance and good taste, but if it is allowed to melt and then is placed back in the refrigerator its appearance and texture change dramatically and it would not be acceptable to a consumer.

Thus, there has been an adverse influence on its quality, even though its chemical composition is unchanged, because of an alteration in the structural organisation of the constituents caused by the melting of ice and fat crystals.
Another familiar example is the change in egg white from a transparent viscous liquid to an optically opaque gel when it is heated in boiling water for a few minutes. Again there is no change in the chemical composition of the food, but its physiochemical properties have changed dramatically because of an alteration in the structural organisation of the constituents caused by protein unfolding and gelation.

The structure of a food can be examined at a number of different levels:

— Molecular structure (1 - 100 nm). Ultimately, the overall physicochemical properties of a food depend on the type of molecules present, their three-dimensional structure and their interactions with each other. It is therefore important for food scientists to have analytical techniques to examine the structure and interactions of individual food molecules.

— Microscopic structure (10 nm - 100 m). The microscopic structure of a food can be observed by microscopy (but not by the unaided eye) and consists of regions in a material where the molecules associate to form discrete phases, e.g., emulsion droplets, fat crystals, protein aggregates and small air cells.

— Macroscopic structure (> 100 m). This is the structure that can be observed by the unaided human eye, e.g., sugar granules, large air cells, raisons, chocolate chips

All of these different levels of structure contribute to the overall properties of foods, such as texture, appearance, stability and taste. In order to design new foods, or to improve the properties of existing foods, it is extremely useful to understand the relationship between the structural properties of foods and their bulk properties. Analytical techniques are therefore needed to characterise these different levels of structure. A number of the most important of these techniques are considered in this course.

Physicochemical Properties of Foods

The physiochemical properties of foods (rheological, optical,
stability, "flavour") ultimately determine their perceived quality, sensory attributes and behaviour during production, storage and consumption.

The optical properties of foods are determined by the way that they interact with electromagnetic radiation in the visible region of the spectrum, e.g., absorption, scattering, transmission and reflection of light. For example, full fat milk has a "whiter" appearance than skim milk because a greater fraction of the light incident upon the surface of full fat milk is scattered due to the presence of the fat droplets.

The rheological properties of foods are determined by the way that the shape of the food changes, or the way that the food flows, in response to some applied force. For example, margarine should be spreadable when it comes out of a refrigerator, but it must not be so soft that it collapses under its own weight when it is left on a table.

The stability of a food is a measure of its ability to resist changes in its properties over time. These changes may be chemical, physical or biological in origin. Chemical stability refers to the change in the type of molecules present in a food with time due to chemical or biochemical reactions, e.g., fat rancidity or non-enzymatic browning. Physical stability refers to the change in the spatial distribution of the molecules present in a food with time due to movement of molecules from one location to another, e.g., droplet creaming in milk. Biological stability refers to the change in the number of microorganisms present in a food with time, e.g., bacterial or fungal growth.

The flavour of a food is determined by the way that certain molecules in the food interact with receptors in the mouth (taste) and nose (smell) of human beings. The perceived flavour of a food product depends on the type and concentration of flavour constituents within it, the nature of the food matrix, as well as how quickly the flavour molecules can move from the food to the
sensors in the mouth and nose. Analytically, the flavour of a food is often characterised by measuring the concentration, type and release of flavour molecules within a food or in the headspace above the food. Foods must therefore be carefully designed so that they have the required physicochemical properties over the range of environmental conditions that they will experience during processing, storage and consumption, e.g., variations in temperature or mechanical stress. Consequently, analytical techniques are needed to test foods to ensure that they have the appropriate physicochemical properties.

**Sensory Attributes**

Ultimately, the quality and desirability of a food product is determined by its interaction with the sensory organs of human beings, e.g., vision, taste, smell, feel and hearing. For this reason the sensory properties of new or improved foods are usually tested by human beings to ensure that they have acceptable and desirable properties before they are launched onto the market. Even so, individuals' perceptions of sensory attributes are often fairly subjective, being influenced by such factors as current trends, nutritional education, climate, age, health, and social, cultural and religious patterns.

To minimise the effects of such factors a number of procedures have been developed to obtain statistically relevant information. For example, foods are often tested on statistically large groups of untrained consumers to determine their reaction to a new or improved product before full-scale marketing or further development. Alternatively, selected individuals may be trained so that they can reliably detect small differences in specific qualities of particular food products, e.g., the mint flavour of a chewing gum.

Although sensory analysis is often the ultimate test for the acceptance or rejection of a particular food product, there are a number of disadvantages: it is time consuming and expensive to carry out, tests are not objective, it cannot be used on materials that contain poisons or toxins, and it cannot be used to provide information about the safety, composition or
nutritional value of a food. For these reasons objective analytical tests, which can be performed in a laboratory using standardised equipment and procedures, are often preferred for testing food product properties that are related to specific sensory attributes. For this reason, many attempts have been made to correlate sensory attributes to quantities that can be measured using objective analytical techniques, with varying degrees of success.

**Analytical Technique**

There are usually a number of different analytical techniques available to determine a particular property of a food material. It is therefore necessary to select the most appropriate technique for the specific application. The analytical technique selected depends on the property to be measured, the type of food to be analysed, and the reason for carrying out the analysis. Information about the various analytical procedures available can be obtained from a number of different sources. An analytical procedure may already be routinely used in the laboratory or company where you are working. Alternatively, it may be possible to contact an expert who could recommend a certain technique, e.g., a University Professor or a Consultant. Often it is necessary to consult scientific and technical publications. There are a number of different sources where information about the techniques used to analyse foods can be obtained:

Tabulated Official Methods of Analysis

A number of scientific organisations have been setup to establish certain techniques as official methods, e.g. Association of the Official Analytical Chemists (AOAC) and American Oil Chemists Society (AOCS). Normally, a particular laboratory develops a new analytical procedure and proposes it as a new official method to one of the organisations. The method is then tested by a number of independent laboratories using the same analytical procedure and type of equipment stipulated in the original proposal.
The results of these tests are collated and compared with expected values to ensure that the method gives reproducible and accurate results. After rigorous testing the procedure may be accepted, modified or rejected as an official method. Organisations publish volumes that contain the officially recognised test methods for a variety of different food components and foodstuffs. It is possible to consult one of these official publications and ascertain whether a suitable analytical procedure already exists or can be modified for your particular application.

**Journals**

Analytical methods developed by other scientists are often reported in scientific journals, e.g., Journal of Food Science, Journal of Agriculture and Food Chemistry, Journal of the American Oil Chemists Society, Analytical Chemistry. Information about analytical methods in journals can often be obtained by searching computer databases of scientific publications available at libraries or on the Internet.

**Equipment and Reagent Suppliers**

Many companies that manufacture equipment and reagents used to analyse foods advertise their products in scientific journals, trade journals, trade directories, and the Internet. These companies will send you literature that describes the principles and specifications of the equipment or test procedures that they are selling, which can be used to determine the advantages and limitations of each technique.

**Role of Internet**

The Internet is an excellent source of information on the various analytical procedures available for analysing food properties. University lecturers, book suppliers, scientific organisations, scientific journals, computer databases, and equipment and reagent suppliers post information on the web about food analysis techniques. This information can be accessed using appropriately selected keywords in an Internet search engine.
Developing a New Technique

In some cases there may be no suitable techniques available and so it is necessary to develop a new one. This must be done with great care so as to ensure that the technique gives accurate and reliable measurements. Confidence in the accuracy of the technique can be obtained by analysing samples of known properties or by comparing the results of the new technique with those of well-established or official methods.

One of the most important factors that must be considered when developing a new analytical technique is the way in which "the analyte" will be distinguished from "the matrix". Most foods contain a large number of different components, and therefore it is often necessary to distinguish the component being analysed for ("the analyte") from the multitude of other components surrounding it ("the matrix"). Food components can be distinguished from each other according to differences in their molecular characteristics, physical properties and chemical reactions:

- Molecular characteristics: Size, shape, polarity, electrical charge, interactions with radiation.
- Physical properties: Density, rheology, optical properties, electrical properties, phase transitions (melting point, boiling point).
- Chemical reactions: Specific chemical reactions between the component of interest and an added reagent.

When developing an appropriate analytical technique that is specific for a particular component it is necessary to identify the molecular and physicochemical properties of the analyte that are sufficiently different from those of the components in the matrix. In some foods it is possible to directly determine the analyte within the food matrix, but more often it is necessary to carry out a number of preparatory steps to isolate the analyte prior to carrying out the analysis. For example, an analyte may be physically isolated from the matrix using one procedure and then analysed using another procedure.
In some situations there may be one or more components within a food that have very similar properties to the analyte. These "interferents" may make it difficult to develop an analytical technique that is specific for the analyte. It may be necessary to remove these interfering substances prior to carrying out the analysis for the analyte, or to use an analytical procedure that can distinguish between substances with similar properties.

Selecting an Appropriate Technique

Some of the criteria that are important in selecting a technique are listed below:

- **Precision**: A measure of the ability to reproduce an answer between determinations performed by the same scientist (or group of scientists) using the same equipment and experimental approach.

- **Reproducibility**: A measure of the ability to reproduce an answer by scientists using the same experimental approach but in different laboratories using different equipment.

- **Accuracy**: A measure of how close one can actually measure the true value of the parameter being measured, e.g., fat content, or sodium concentration.

- **Simplicity of operation**: A measure of the ease with which relatively unskilled workers may carry out the analysis.

- **Cost**: The total cost of the analysis, including the reagents, instrumentation and salary of personnel required to carry it out.

- **Speed**: The time needed to complete the analysis of a single sample or the number of samples that can be analysed in a given time.

- **Sensitivity**: A measure of the lowest concentration of a component that can be detected by a given procedure.

- **Specificity**: A measure of the ability to detect and quantify specific components within a food material, even in the presence of other similar components, e.g., fructose in the presence of sucrose or glucose.
— **Safety:** Many reagents and procedures used in food analysis are potentially hazardous e.g. strong acids or bases, toxic chemicals or flammable materials.

— **Destructive/Nondestructive:** In some analytical methods the sample is destroyed during the analysis, whereas in others it remains intact.

— **On-line/Off-line:** Some analytical methods can be used to measure the properties of a food during processing, whereas others can only be used after the sample has been taken from the production line.

— **Official Approval:** Various international bodies have given official approval to methods that have been comprehensively studied by independent analysts and shown to be acceptable to the various organisations involved.

— **Nature of Food Matrix:** The composition, structure and physical properties of the matrix material surrounding the analyte often influences the type of method that can be used to carry out an analysis, e.g., whether the matrix is solid or liquid, transparent or opaque, polar or non-polar.

If there are a number of alternative methods available for measuring a certain property of a food, the choice of a particular method will depend on which of the above criteria is most important. For example, accuracy and use of an official method may be the most important criteria in a government laboratory which checks the validity of compositional or nutritional claims on food products, whereas speed and the ability to make nondestructive measurements may be more important for routine quality control in a factory where a large number of samples have to be analysed rapidly.

**References**
