CHAPTER - I

- **FOOD ANALYSIS**

Outlines:

- **Introduction to food analysis:**
- Chemical composition of food
- Sampling and sample preparation
- Methods of food analysis

Introduction

Objectives:

- The end of the chapter students will explain of Chemical composition of food, Sampling and sample preparation for analysis and different Methods of food analysis.

❑What is Food analysis?

It is a discipline dealing with:

1. the development,
2. application and
3. study of analytical procedures

- For :

1. characterizing the properties of foods and
2. their constituents.

❑Use of the analytical procedures:

- **P**rovide information about a wide variety of different characteristics of foods:

✓ composition,

✓ structure,

✓ physicochemical properties and

✓ sensory attributes.

Why to analyze foods?

1. Food Safety
2. Quality control
3. Government Regulations and Recommendations
4. Research and Development

❑Who analyze foods?

- ✓scientists working in all of the major sectors of the food industry
- ✓food manufacturers,
- ✓ingredient suppliers,
- ✓analytical service laboratories,
- ✓government laboratories, and
- ✓university research laboratories.

Activity-1

- What are chemical composition of food?

❖Chemical composition of food:

- Food contains chemical molecules
- Chemical composition may be determined in laboratory
- Chemical substances found in the largest amounts in food:
  - Water
  - Carbohydrates
  - Fats
  - Protein
  - Ash/mineral/
  - Vitamin

❑Water

- All foods contain at least some water

- *Free water*
  - Held inside cells
  - Maintains properties of free water
  - May be removed by pressure

- *Bound water*
  - Is part of molecule structure
  - Reduced mobility
  - Does not retain properties of free water

❖ Water Activity:

- More *bound water*, then less *water activity*
- *Water activity:*
  - ➢ Ratio of the partial vapor pressure of water in a substance at a specified temperature to the standard state partial vapor pressure of pure water at same temperature.
  - ◦ The water activity scale extents from 0(dry) to 1.0(pure water).
- Foods more perishable if higher water activity:
  - ◦ Microorganisms need water
  - ◦ To reduce water activity:
    - Dry
    - Freeze
    - Add sugar or salt

Activity -2

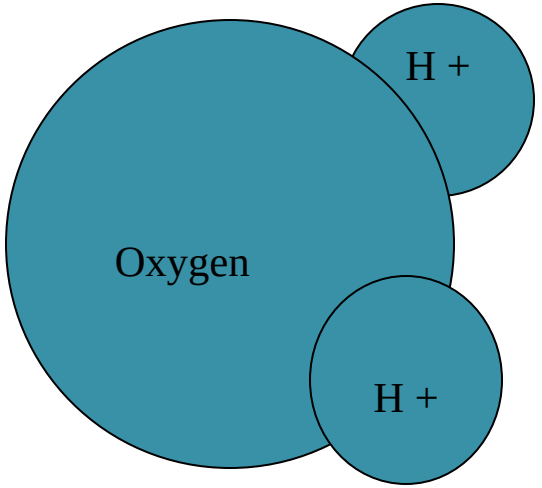
- What is the used of Water in Food Preparation?

❑Uses of Water in Food Preparation:

- Universal solvent
- Heat transfer
- Freezing
- Cleaning agent
- Promotion of chemical changes
  - Ionization of salt
  - Baking powder
- Hydrolysis reactions

❖Nature of Water

- H_2O
- Two hydrogen atoms bonded with *covalent* bonds to one oxygen atom
- Is dipolar
  - Negative on oxygen side
  - Positive on hydrogen sides



❑Water Hardness:

- **Types of hard water**
  - **Temporary**-It is due to the presence of bicarbonates of calcium and magnesium and can be removed by boiling/addition of lime/.
  - **Permanent-**It is due to the presence of sulphates, nitrates and chlorides of calcium &magnesiium.
  - It can be removed by addition of lime,addition of sodium carbonate and base exchange method.

- Hard water and food preparation
  - Rehydration and softening of dried beans slowed
  - Alkalinity may affect color of vegetables
  - Promote cloudiness in tea
- Water may be softened

❖Measurement of Hardness of water:

- Hardness of water is expressed/measured in terms of milli equavilant /litre
- 1 milli-equavilant/lit=50mgs.of$CaCO_3$/litre
- Classification of hardness:
- Soft water----------=1m.Eq./litre
- Moderately hard water------=1-3 m.Eq./litre
- Hard water----------=3-6 m.Eq./litre
- Very Hard water----------->6 m.Eq./litre

Carbohydrates

- Simple
  - sugars
- Complex
  - starch and fiber
- Made of
  - Carbon (C)
  - Hydrogen (H)
  - Oxygen (O)
  - One molecule of H_2O for each atom of carbon

❖Chemical Classification

- Classified by number of basic sugar units linked together
  - Monosaccharide – One unit
  - Disaccharides – Two units
  - Oligosaccharides – 10 or fewer units
  - Polysaccharides – Up to 1000 units

Monosaccharides are :

- ✓ Glucose
- ✓ Fructose
- ✓ Galactose
  - ❖ Disaccharides
- Sucrose
  - Glucose + Fructose
- Lactose
  - Glucose + Galactose
- Maltose
  - Glucose + Glucose

❖Oligosaccharides

- ✓ Raffinose and Stachyos
- ✓ Not broken down by digestive tract
- ✓ Found in dried beans

❖Polysaccharides:

- Starch
  - Amylose
  - Amylopectin
- Dextrins
  - Produced when starch molecules are partially broken down by *enzymes, acid, or heat.*
    - Less thickening power than starch
- Glycogen
- Plant Fiber Components

❖ Plant Fiber Components:

- Called dietary fiber / roughage / bulk
  - Cellulose
  - Hemicellulose
  - Beta-glucans
    - Found in oats and barley
    - Associated with reduced risk of heart disease
  - Pectic substances
    - Pectin forms gels in jams, jellies, and preserves
  - Vegetable gums

Browning of Foods:

- Caramelization
  - Heating of sugars above melting point
- Maillard Reaction
  - Involves carbohydrate
  - Carbonyl group of sugar combines with amino group of a protein with removal of water. After additional reactions brown pigments are formed
    - i.e. browning of bread during baking

Lipids or Fats

- Insoluble in water
- Feel "greasy"
- Three major groups
  - Triglycerides
  - Phospholipids
  - Sterols

❑Triglycerides

- Account for 90-95 percent of fatty substances in food.
- **Composed of:**
  - 3 fatty acids
    - linked atoms of carbon with organic acid group
  - One molecule glycerol
    - 3 carbon atoms and three hydroxyl groups

❑Fatty Acids

- Most fatty acids in foods are combined in triglycerides.
- Fatty acids differ
  - Number of hydrogen atoms attached
  - Length of carbon chain
- Carbon chains
  - Usually even numbered

❖Types of Fatty Acids:

- Saturated fats
  - No double bonds between carbon atoms, so no more hydrogen can be added
- Unsaturated
  - Double bonds between some of the carbon atoms that can be broken to add hydrogen
- Monounsaturated
  - One double bond

❑Omega 3 fatty acids:

- ✓ Polyunsaturated fatty acids with double bond between 3rd and 4th carbon from the left on the structure.
- ✓ Found in fatty fish
- ✓ Protective for heart disease

❑Linoleic Acid:

- ✓ An essential fatty acid
- ✓ Cannot be made by the body – must be consumed in food.

❑Fat in Food Preparation:

- Tenderizing in baked foods
- Contribute to leavening
  - Creaming of fat and sugar
- Promote moistness
- Major components of salad dressings
- May be heated to high temperatures
  - Frying of foods
- Contribute flavor

❑Proteins:

- Essential nutrient
- In food preparation several important roles
  - Binding water
  - Forming gels
  - Thickening
  - Producing foams
  - Aiding browning
- Protein Contain:
  - Carbon
  - Hydrogen
  - Oxygen
  - Nitrogen
- Large molecules
  - Hundreds or thousands of amino acids joined with *peptide linkage*

❑Protein Quality:

- Amino acids used as building blocks for proteins
- Nine amino acids are essential for adult human nutrition
- Complete proteins include essential amino acids

- Isoleucine
- Leucine
- Lysine
- Methionine
- Phenylalanine
- Threonine
- Tryptophan
- Valine
- Histidine

Activity -3

- What are the Protein sources ?

  In 3min.

❑Protein sources are:

- Meats, Fish, and Poultry
- Eggs and Dairy
- Nuts
- Dry legumes
- Cereal grains – in lesser amounts etc.

❑Ash:

- Ash: total mineral content; inorganic residue remaining after ignition or complete oxidation of organic matter
- Minerals:
  - Macro minerals (>100 mg/day)
    - Ca, P, Na ,K, Mg, Cl, S
  - Trace minerals (mg/day)
    - Fe, I, Zn, Cu, Cr, Mn F,
  - Toxic mineral
    - lead, mercury, cadmium, aluminum

- SAMPLING AND SAMPLE PREPARATION:

Population vs. sample

- ***A population:*** is the whole set of measurements or counts about which we want to draw conclusion.

  - The whole of the material whose properties we are trying to obtain an estimate.

- ***Sample:*** is a sub set of a population, a set of some of the measurements or counts that comprises the population.
- ***Laboratory Sample:*** fraction of population sample used in the final laboratory analysis.

❖Sampling:

- Definition: a defined procedure whereby a part of a substance is taken to provide, for testing, a representative sample of the whole or as required by the appropriate specification for which the substance is to be tested.

❑Random Sampling strategy:

Random: to eliminate questions of bias in selection. **Three types**.

1. **Simple**: any sample has an equal chance of being selected.
2. **Systematic**: first sample selected randomly and subsequent samples taken at arranged intervals

most commonly used procedure

Examples

- solid material in motion (conveyor belt): periodically transfer portion into a sample container.

Cont'd…

3.Stratified: The lot is **subdivided** and a simple random sample selected from each stratus.

Example:

- Material lots delivered at different times: take proportional weights of material from each lot.

❑Selective Sampling:

- Selective: screens out or selects materials with certain characteristics usually attempted following test results on random samples.

Examples:

- contaminated foods: attempt to locate the adulterated portion of the lot.

❑A Composite Sample:

- Composite: portions of material selected in proportion to the amount of material they represent.
- The ratio of the components taken up to make the composite can be in terms of bulk, time or flow.

- Reduces the cost of analysing large numbers of samples.
- Not a sampling technique; it is a preparatory technique after the samples have been taken.

❑Sampling Plan:

Sampling Plans: Clearly written document that contains precise details about:

- Sample size
- The locations from which the sample should be selected
- The method used to collect the sample
- The method used to preserve them prior to analysis

Cont'd…

DECISIONS TO BE MADE:

- Manual vs. automatic sampling
- Sampling frequency
- Sample sizes
- Sampling locations
- Individual vs. composite samples
- Training
- Sampling strategy
  - Random selection
  - *Stratified* random selection
  - *Systematic* stratified selection

❑Factors Affecting a Sampling Plan:

- Purpose of inspection

  -acceptance/rejection, variability/average

- Nature of the product

  -homogenous, unit, cost

- Nature of the test method

  -Critical/minor, destructive, cost, time

- Nature of the population

  -uniformity, sublot

❑Developing a Sampling Plan:

- Number of samples selected

  -Variation in properties, cost, type of analytical techniques

- Sample location

  -random sampling vs systematic sampling vs judgment sampling

- Manner in which the samples are collected

  -manual vs mechanical device

❑The Bottom Line in Sampling:

- Depending upon the nature of the material to be analyzed, you must determine a method of taking small subsamples from a large lot (5,000 lb blender, 20 sack on a truck etc) that accurately reflect the overall composition of the whole lot.
- An inaccurate sample of a large lot may actually be worse than no sample at all.

❑Preparation of Laboratory Samples:

- You may have taken as much as 10 lbs of sub-samples from a lot that now needs to be further reduced in size;

  -Make the sample homogeneous by mixing and grinding and then more sub-sampling.

  -Be aware of any changes that might occur between sampling and analysis and take proper action (e.g. enzymatic action, microbial growth etc).

  -Properly label the final sample with name, date/time, location, person and other relevant data.

❖ Solids sample pre-treatment:

- Grinding of solids
- Sample drying
- Leaching and extraction of soluble components
- Filtering of mixtures of solids, liquids and gases to leave particulate (solid) matter

- Methods of food analysis

❖Moisture Determination:

- Moisture or water is the most common component in foods.
- The two most common moisture considerations in food is that of total moisture content and water activity.
- The total moisture content of foods is generally determined by some form of drying method. whereby all the moisture is removed by heat and moisture is determined as the weight lost.

Cont'd…

The moisture content of a food material is defined through the following equation:

$$\%\,\text{Moisture} = (m_w / m_{sample}) \times 100$$

Where: m_w is the mass of the water and m_{sample} is the mass of the sample.

Moisture Determination Methods:

1. Direct Methods:

- Air oven Drying
- Vacuum oven drying
- Freeze Drying
- Distillation
- Chemical Desiccation
- Thermo Gravimetric method
- Gas Chromatography

2. Indirect methods:

- Refractometer method
- Infrared Absorption Spectroscopy
- Near infrared Refraction Spectroscopy
- Microwave absorption method
- Conductivity methods

Activity-4

- What is oven Drying method work?

  3min.

Cont'd…

- These methods rely on measuring the mass of water in a known mass of sample.

Types of Oven:

1. Convection Oven

– Greatest temperature variations - because hot air slowly circulated with out the aid of fan, air movement is obstructed further by pans placed in the oven.

Cont'd…

2. Forced Draft Oven:
   - The least temperature differential across the interior (< 1°C).
   - Air is circulated by a fan that forces air movement throughout the oven cavity.
   - Drying period 1 – 24 hr, depending on food sample and its pretreatment.

Cont'd…

3. Vacuum oven:

- Sample is placed in oven under reduced pressure thereby reducing the boiling point of water.
- Drying under reduced pressure (25 – 100mm Hg) and temperature (30-250°C)
- Able to obtain a more complete removal of water and volatiles without decomposition within a 3 – 6 hr.

Cont'd…

- 4. Microwave Oven
- Uses microwave as a heat source; Very fast method.

Cont'd…

5. Infrared lamp drying:
   - Uses infrared lamp as a heat source; Very fast
   - Advantages: rapid and inexpensive
   - This is because the IR energy penetrates into the sample

Cont'd…

6. **Moisture Analyzer:**
   - Using a digital balance,
   - the test sample is placed on an aluminum pan and
   - the constant temperature is applied to the test sample.
   - Instrument automatically weighs and calculates the % of moisture or solids

❖Method of Protein Analysis:

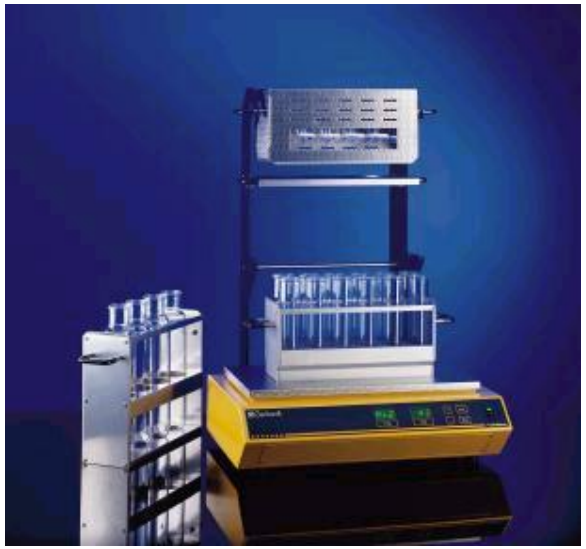
- ✓ Kjeldahl – measures the amount of nitrogen in a sample.
- ✓ Lowry- measures the tyrosine/tryptophan residues of proteins.

Activity-5

- What are the three basic steps to determine % nitrogen protein in kjeldahl method?

5min.

Cont'd….



1.Digestion

$$\text{Protein} \xrightarrow[\text{Heat, catalyst}]{\text{Sulfuric acid}} (NH_4)_2SO_4$$

(ammonium sulfate)

$$\text{Protein N} \rightarrow NH_4^+ + H_2SO_4 \rightarrow (NH_4)_2SO_4$$

Cont'd….

2.Neutralization and distillation

$(NH_4)_2SO_4 + 2NaOH \rightarrow 2NH_3 + Na_2SO_4 + 2H_2O$

$NH_3 + H_3BO_3 \rightarrow NH_4^+ : H_2BO_3^- + H_3BO_3$

(boric acid) (ammonium-borate complex)

Kjeldahl distillation unit

Color change

Cont'd…

3.Titration (direct titration)

$$H_2BO_3^- + H^+ \rightarrow H_3BO_3$$

(HCl)

$$\%N = \frac{x \text{ moles}}{1000 \text{ cm}^3} \times \frac{(v_s - v_b) \text{ cm}^3}{m \text{ g}} \times \frac{14 \text{ g}}{\text{moles}} \times 100$$

$$\%\,\mathrm{N} = N\,\mathrm{HCl} \times \frac{\text{Corrected acid volume}}{\text{g of sample}} \times \frac{14\,\text{g N}}{\text{mol}} \times 100$$

%Crude Protein = F*%N

❑Crude Fat Analysis:

- Fats are soluble in organic solvents but insoluble in water.
- Solvent Extraction Methods:
- Sample preparation:
  - Particle size reduction increases extraction efficiency
  - Pre drying sample to remove water is common.

Actvity-6

- What are solvent selection parameters to determine crude fat?

4min.

Solvent selection:

- high solvent power for lipids
- low solvent for other components
- easy to evaporate
- low boiling point
- nonflammable
- nontoxic
- good penetration into sample
- single component
- inexpensive
- non-hygroscopic

❖Common Solvents:

❖Ethyl ether:

-best solvent for fat extraction,

-more expensive,

-fire hazard,

-hygroscopic

❖Petroleum ether:

✓cheaper,

✓more hydrophobic,

✓ less hygroscopic

◦ Hexane – is a clear colorless liquid with a petroleum like odor, crude oil extraction

Types of Fat Analysis:

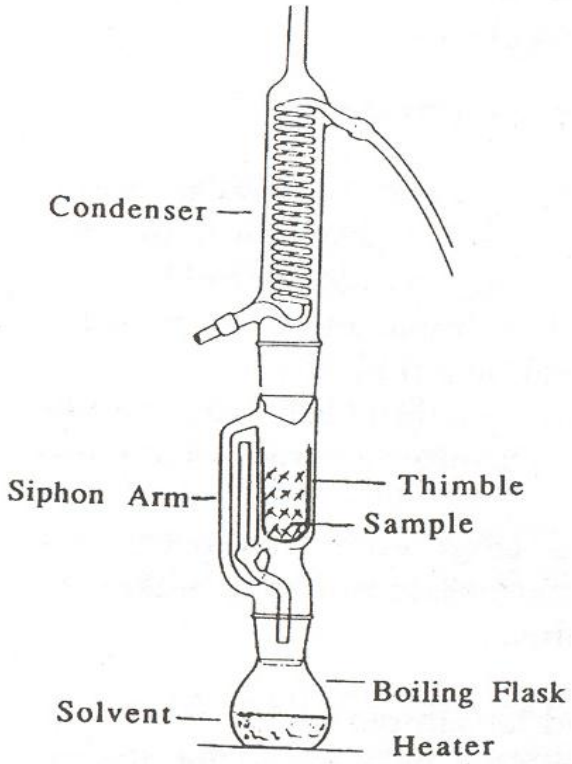
- Extraction Methods
  - ✓ Continuous – Goldfish
  - ✓ Semi-Continuous- Soxhlet
  - ✓ Discontinuous- Mojonnier
- Instrumental Methods
  - ✓ Dielectric
  - ✓ Infrared
  - ✓ Ultrasound

Solvent Extraction Methods:

- Continuous extraction: Goldfish method
  - ✓Principle: Solvent continuously flowing over the sample with no build-up
  - ✓Advantages: it is fast & efficient.
  - ✓Disadvantages: channeling – not complete extraction.

cont'd…

- Semicontinuous extraction: Soxhlet method
  - Principle: Solvent building up in extraction chamber for 5-10 min before siphoning back to boiling flask.
  - Advantages: no channeling
  - Disadvantages: time consuming



Cont'd..

- Discontinuous extraction: Mojonnier method (wet method extraction)
  - Principle: a mixture of ethyl ether and petroleum ether in a Mojonnier flask
  - Advantages: no prior removal of moisture
  - Disadvantages: constant attention.

Instrumental Methods

- Dielectric method
  - Principle: low electric current from fat
- Infrared method
  - Principle: Fat absorbs infrared energy at a wavelength of 5.73 μm
- Ultrasound method
  - Principle: sound velocity increases with increasing fat content

II. Nonsolvent Wet Extraction Methods

- Gerber Method for Milk Fat
- Principle:
- it uses sulfuric acid and amyl alcohol.
- The sulfuric acid: digests proteins and carbohydrates, releases fat, and maintains the fat in a liquid state by generating heat.

Butyrometer

❖CARBOHYDRATE ANALYSIS

- Next to water, carbohydrates are the most abundant food component
- %Total carbohydrate=100% - (H_2O + ash + fat + protein)
- Types of carbohydrates include;
  - monosaccharide: glucose, fructose, galactose
  - disaccharide: sucrose, lactose, maltose
  - oligosaccharids: raffinose
  - polysaccharide: starch, cellulose

❑Methods for Determining Ash:

- The **'ash content'** is
- A measure of the total amount of minerals present within a food,
- whereas the '**mineral content'** is
- A measure of the **amount of specific inorganic components** present within a food, such as **Ca, Na, K, Mg** and so on.

Activity- 7

- Why **ash & mineral** content of foods is determined?
    - 5min.

Cont'd…

- Determination of the **ash & mineral** content of foods is important for a number of reasons:
  - **Nutritional labelling**
  - **Quality:** The quality of many foods depends on the concentration & type of minerals they contains
  - **Microbiological stability:** High mineral contents are sometimes used to retard the growth of certain microorganisms
  - **Nutrition:** Some minerals are essential to a healthy diet whereas others can be toxic (e.g., lead, mercury, cadmium…)
  - **Processing:** It is important to know the mineral content of foods during processing because this affects the physicochemical properties of foods.

❑Cont'd….

- Dry ashing
  - high temperature
- Wet ashing
  - oxidizing agent and/or acid
- Low-temperature plasma ashing
  - dry ashing in partial vacuum at low temperature

Cont'd…

- *Ash content:*
- is a measure of the total amount of minerals present within a food,
- Whereas
- *Mineral conten:*
- is a measure of the amount of specific inorganic components present within a food, such as Ca, Na, K and Cl.

❖Muffle furnace and crucibles:

A

B

❑Definition of Fiber:

- Dietary fiber is defined as lignin plus plant polysaccharides that cannot be digested by human enzymes.

❑**The major components of dietary fiber are:**

- ✓ cellulose,
- ✓ hemicelluloses,
- ✓ pectins,
- ✓ hydrocolloids, and
- ✓ lignin.

❑Methods of Dietary Fiber determination:

- Dietary fiber is estimated by two basic approaches:

1.gravimetrically or

2. chemically.

1. Gravimetrically :digestible carbohydrate, lipids, and proteins are selectively solubilized by chemicals and/ or enzymes.

- Then, undigestible materials are collected by filtration, and
- the fiber residue is quantitated gravimetrically

2.Chemically;

- a) digestible carbohydrates are removed by enzymatic digestion,
- b) fiber components are hydrolyzed by acid, and
- c) monosaccharides are measured.
- d) the sum of monosaccharides in the acid hydrolysate represents fiber.

❑Methods of analysis of vitamins:

- Classification of vitamin analyze

1. Bio analyze
2. Microbiological analyze
3. Physicochemical analyze

1. Bio analyze

- Involve humans and animals
- Used only or the analysis of Vitamins B_{12} and D
- Analysis of Vit D (AOAC Method 936.14)
- Test organisms usually rats

2. Microbiological analyze:

- Limited to the analysis of water-soluble vitamins(B1,B2,B3,…)
- Sensitive and specific to each vitamin
- Principle:
- The growth of microorganisms is proportional to their requirement for a specific vitamin

3. Physicochemical analyze:

- Include:
  - Spectrophotometric
  - Fluorometric
  - Chromatographic
  - Enzymatic
  - Immunological
  - Radiometric methods
  - Application Vitamins A, E, C, B_1(Thiamin), B_2(Riboflavin), etc.

Activity

- Summarized chapter

- THANK YOU!!!