B.Sc. Third Year V Semester

ANALYTICAL CHEMISTRY

Paper XIII [Applied Analytical Chemistry-I]

Environmental Analysis - II

Analysis of Soil

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Introduction

Analysis of Soil

Environment

Environment

- An ecosystem (also **called** as **environment**) is a natural unit consisting of all plants, animals and micro-organisms (biotic factors) in an area functioning together with all of the non-living physical (a biotic) factors of the **environment**.
- A natural body occur on the earth’s surface, supporting plants and consisting of mineral and organic materials. It characterized by relate horizons resulting from the interactions of climate and vegetation or various parent material over varying periods of time and modified by local relief.

What is soil ?

Unconsolidated mineral and organic material on the immediate surface of the earth that contains living and nonliving matter and serves as a natural medium for the growth of land plants.

Functions of soil for plants

anchorage • water • oxygen • nutrients

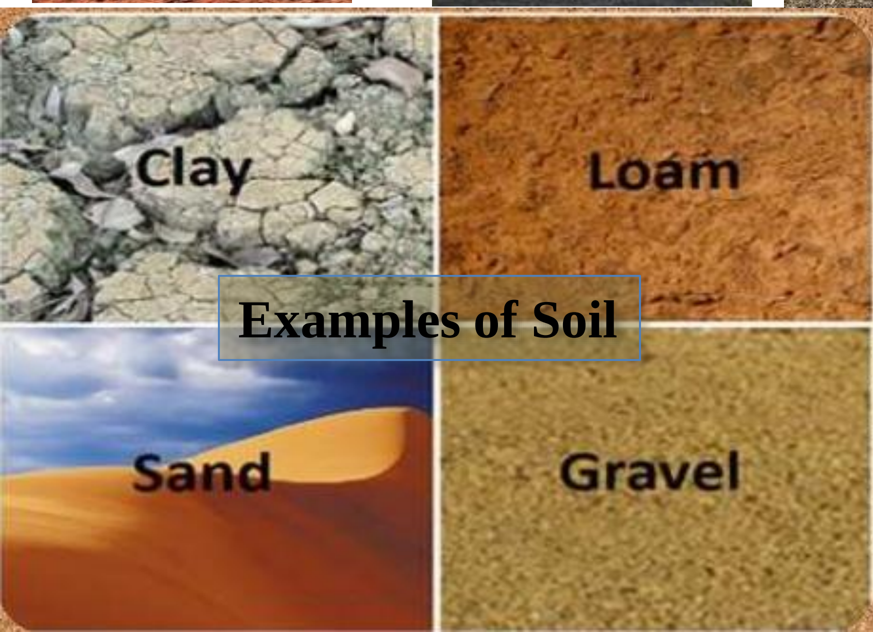
Soil Quality Indicators

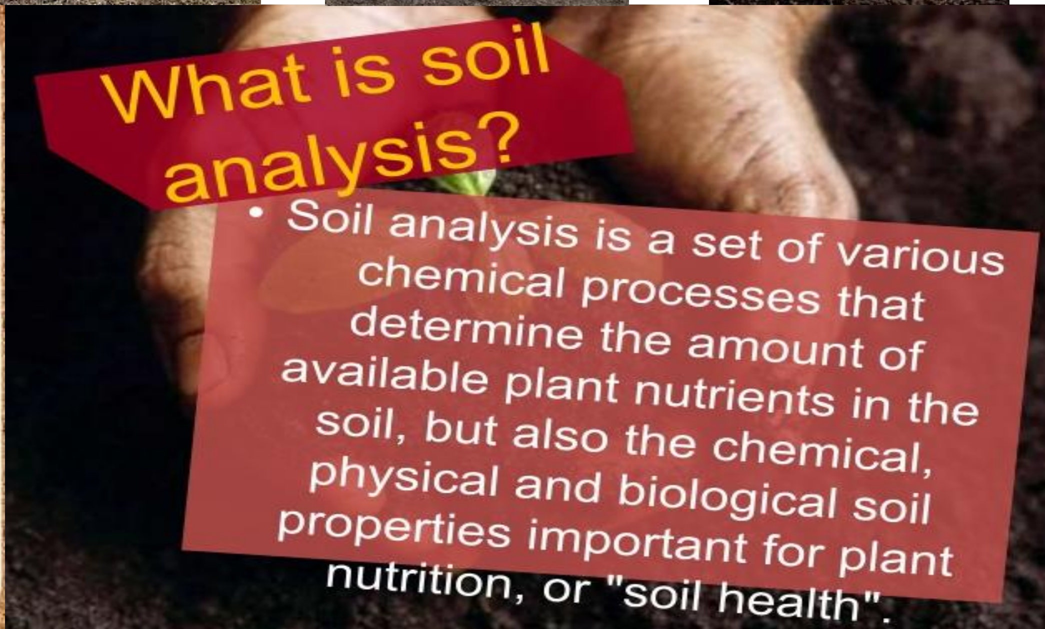
qualitative and/or quantitative techniques

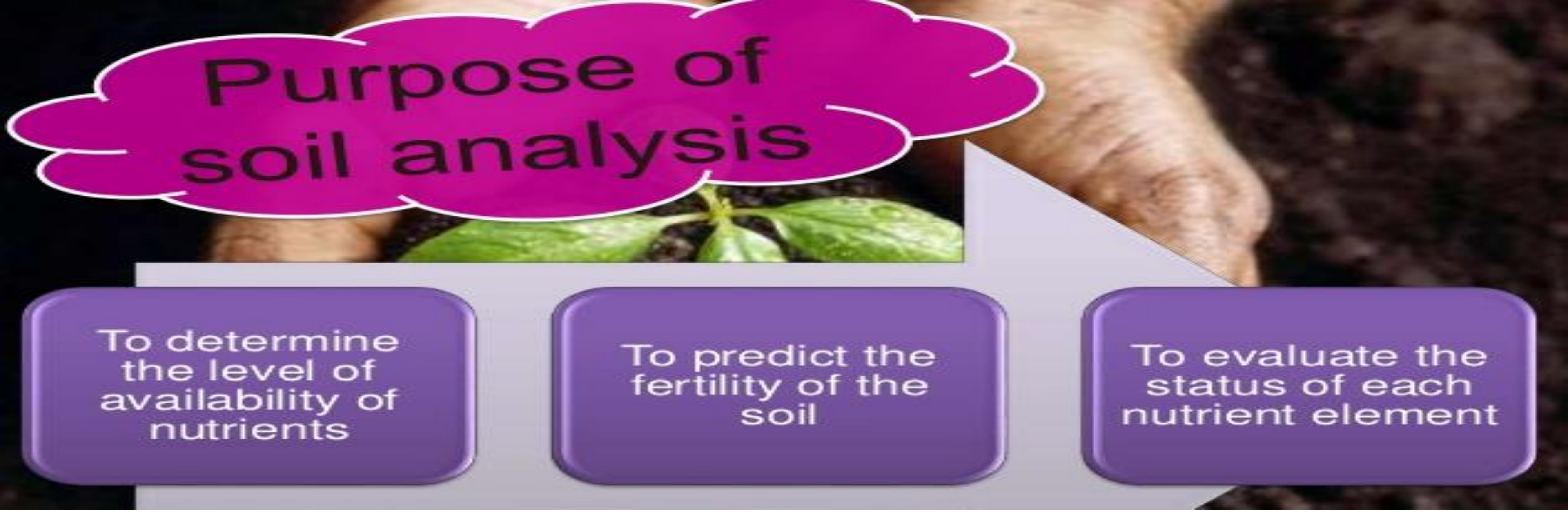
- **collect measurements**
- **evaluate patterns, if any**
- **compare results to measurements taken at different time/location**

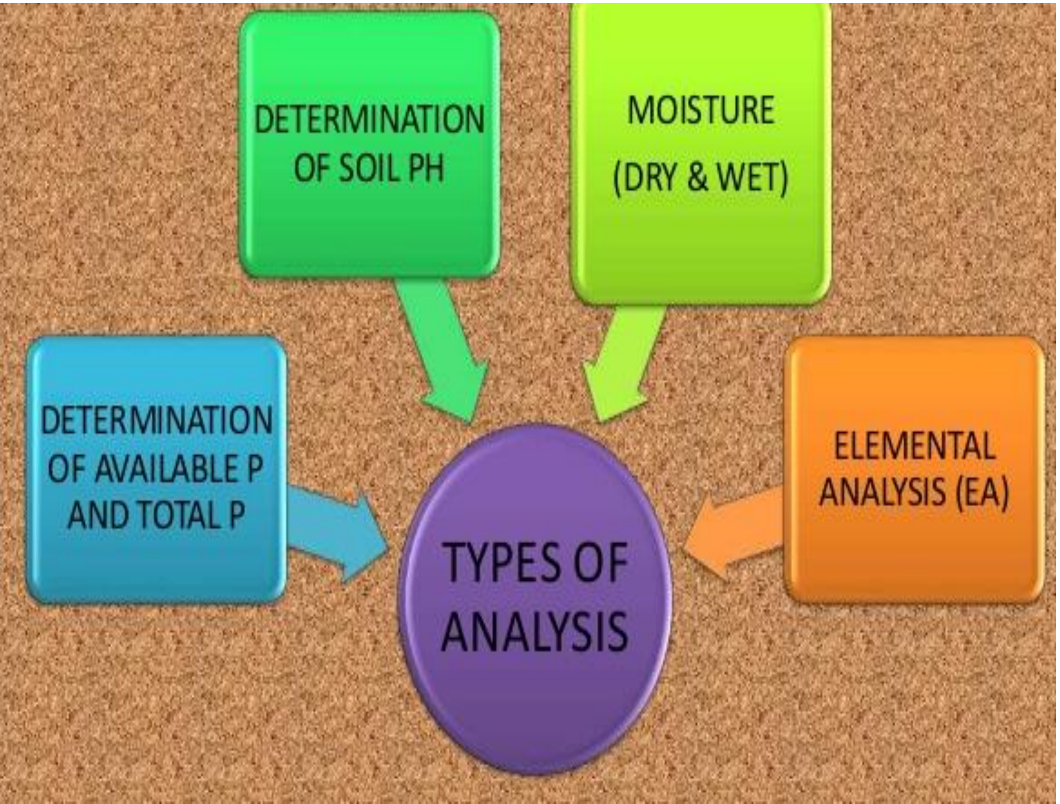
Types of Soil

Examples of Soil

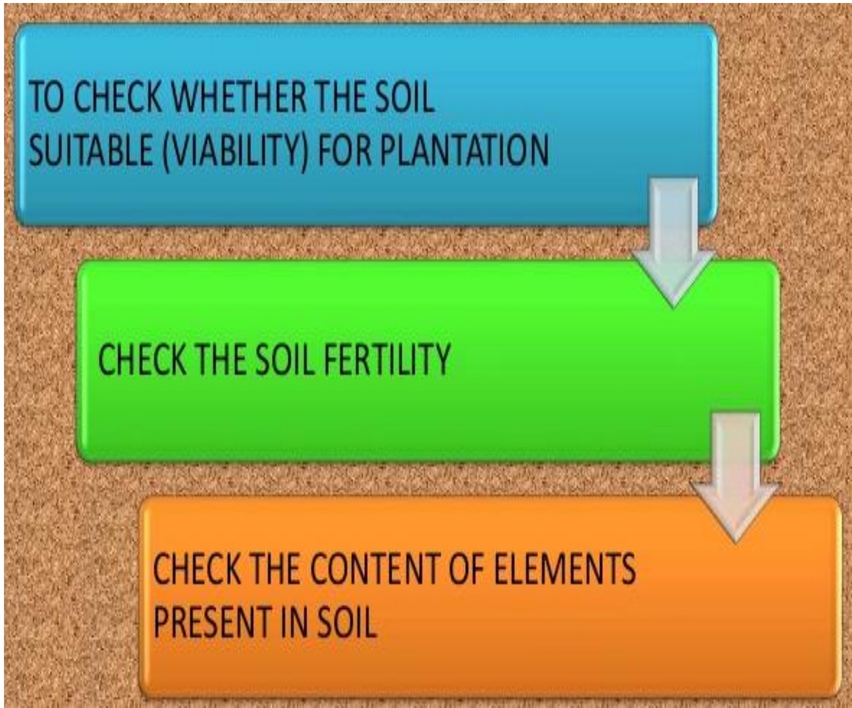





Purpose of soil analysis
To determine the level of availability of nutrients
To predict the fertility of the soil
To evaluate the status of each nutrient element


DETERMINATION OF SOIL PH
MOISTURE (DRY & WET)
DETERMINATION OF AVAILABLE P AND TOTAL P
ELEMENTAL ANALYSIS (EA)
TYPES OF ANALYSIS

WHY ???


TO CHECK WHETHER THE SOIL SUITABLE (VIABILITY) FOR PLANTATION
CHECK THE SOIL FERTILITY
CHECK THE CONTENT OF ELEMENTS PRESENT IN SOIL

Analysis of soil

Soil Sampling/ Preparation

1. pH
2. Moisture Content
3. Bulk Density
4. Specific Gravity
5. Water Holding Capacity
6. Loss of Ignition
7. Total Nitrogen
8. Nitrate Nitrogen
9. Organic Matter
10. Potassium & Sodium

Methods of Soil Sampling/ Preparation


Registration
• The soil samples received.
• The quantity of samples were checked.
• The soil samples were registered and put on the labelled box to dry it out.


Drying
• After being registered, the soil samples were dried out at a well air-ventilatio


Crushing and sieving


Weighing
pH:
Total P:
1 g of sample

Analysis of Soil

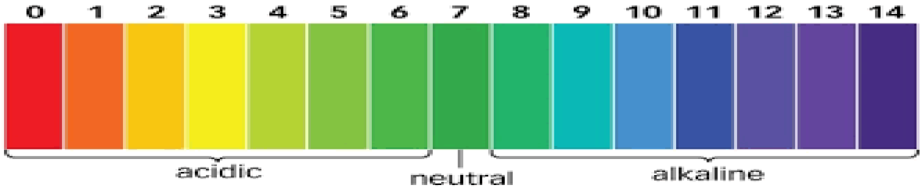
1. pH
2. Moisture Content
3. Bulk Density
4. Specific Gravity
5. Water Holding Capacity
6. Loss of Ignition
7. Total Nitrogen
8. Nitrate Nitrogen
9. Organic Matter
10. Potassium & Sodium

1. pH

The definition of pH is the negative logarithm of the **hydrogen ion (H+) activity** in a given solution.

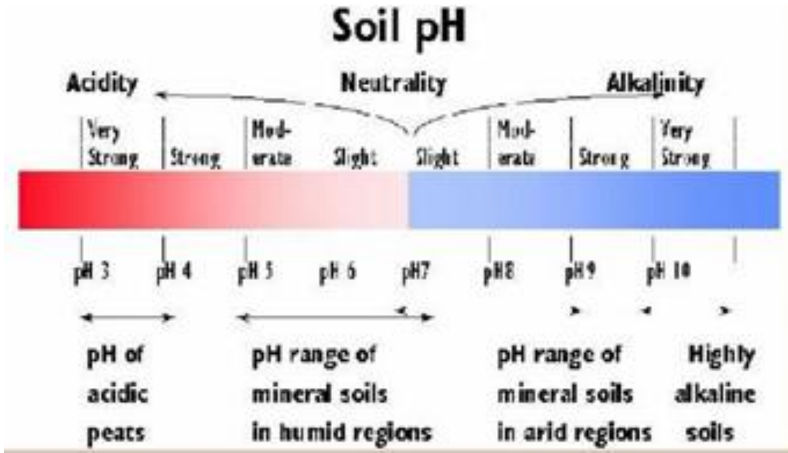
- The range of the **ph scale** is from 0 to 14.

$$pH = -\log aH^{+}$$



Method:

Weight 10g of air-dry soil / sediment. To this add 100ml distilled water for making a suspension of 1:10 w/v dilution. Find out the pH of suspension by as follows method and give the dilution while reporting the result.



2. Determination of Moisture Content

Water present in the soil serves the following purposes :

(*i*) It acts as a solvent.

(*ii*) It acts as a transporting agent.

(*iii*) It maintains texture and compactness of soil, therby making it habitable for plants and animals.

Soil gets moisture from infiltration of precipitated water and irrigation. Its constent in the soil at any time is more or less dependent upon the water holding capacity of soil. However, it drains through percolation, evaporation, and uptake by plants.

Material

Oven ; and chemical balance.

Method

Collect a fresh homogenized sample of soil. Now weigh it. Then dry it in an oven at 105°C until we get a constant weight. Cool it in a desiccator. Finally record the final weight of sample.

Calculation:

$$\% \text{ of Moisture content} = \frac{X_1 - X_2}{X_1} \times 100$$

Where, X_1 = Initial weight of sample in grams

X_2 = Final weight of dried sample

3. Determination of Bulk Density

- **Definition:** Bulk density is an indicator of soil compaction. It is calculated as the dry **weight** of soil divided by its **volume**. This volume includes the volume of soil particles and the volume of pores among soil particles. Bulk density is expressed in **g/cm3.**

One may define the bulk density of the soil as the dry weight of a unit volume of it. It may be expressed as g/cm^3. Generally the bulk density has been found to range from 1.1 to 1.5 g/cm^3 for medium to fine textured soil and from 1.2 to 1.65 g/cm^3 for coarse textured soil. However, it has been slightly higher in case of alkaline saline soils. The soils having high bulk density have been found to be inhibitive to root penetration, and have low permeability and infiltration. The bulk density has been inversely related to pore space of soil.

Material

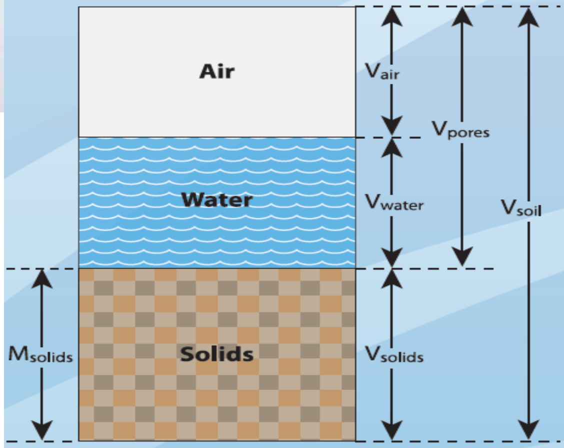
Oven ; measuring cylinder ; and chemical balance.

Method

Collect the sample and dry it in an oven at 105°C until we get its constant weight. Now put a little dried soil to a measuring cylinder and record the volume. Now find out the weight of this volume of soil on a balance.

Calculation. It is found out by the following equation :

$$\text{Bulk density} = \frac{\text{Weight of soil (g)}}{\text{Volume of soil (cm}^3)} = \ldots g/cm^3$$



4. Determination of Specific Gravity

- **Specific gravity is** also called relative density, ratio of the density of a substance to that of a standard substance. .

$$\text{Specific gravity} = \frac{\text{Density of the object}}{\text{Density of water}} = \frac{\rho_{\text{object}}}{\rho_{H_2O}}$$

The specific gravity of the soil has been directly related to its bulk density. It has been used as an index of some aspects of soil quality as in case of bulk density.

Material

Oven ; glass bottles ; and chemical balance.

Method

Collect the soil sample and dry it in an oven at 105°C until we get a constant weight. Fill a pre-weighed glass bottle of known volume with dried soil and note down its weight. Fill it with distilled water and record its weigh.

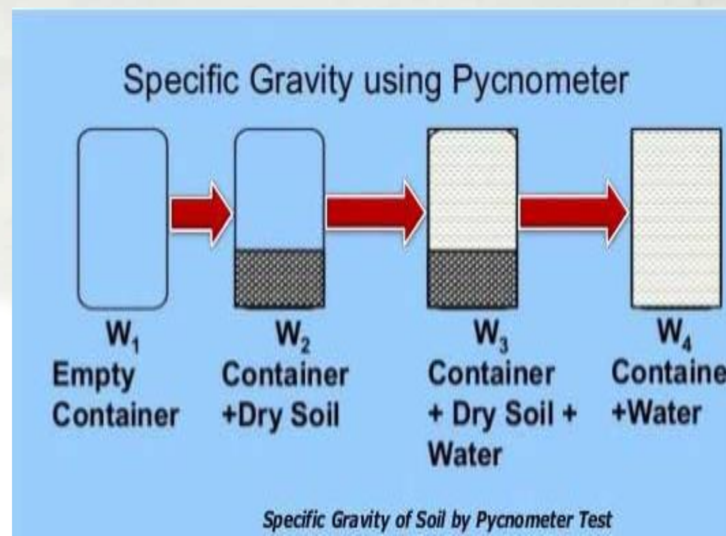
Calculation

$$\text{Specific gravity} = \frac{x_2 - x_1}{x_3 - x_1}$$

where x_1 = weight of empty bottle

x_2 = weight of empty bottle + soil

x_3 = weight of empty bottle + water



5. Determination of Water Holding Capacity

❑ **Soil water holding capacity** is a term that all farms should know to optimize crop production. Simply **defined** soil water holding capacity is the amount of water that a given soil can hold for crop use. The larger the surface area the easier it is for the soil to hold onto water so it has a higher water holding capacity.

When we saparate the soil absolutely with water, water fills all the proes between the particles of soil and no air space exists as in the case of aquatic sediments. Such a soil is said to be at its maximum water holding capacity or saturation.

Material

Perforated circular soil boxes filter paper Whatman No. 1) ; Petri dish ; chemical balances and oven.

Method

Collect the soil sample and crush it. Dry this sample in an oven at 105°C. Keep a filter paper (Whatman No. 1) inside the perforated bottom of the circular soil box. Then weigh the box. Now fill it with dried soil sample. Then, find out the weight of box filled with dried soil. Keep the box in Petri dish of 10 cm diameter, having water, for about 12 hours, so that water enters the box and gets saturated the soil. Remove the box out of water, wipe it dry on the outside, and find out its weight.

Calculations

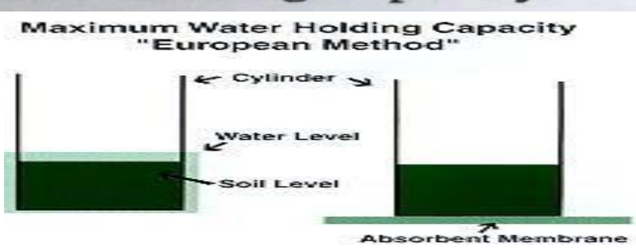
$$\text{WHC } (\%) = \frac{(W_3 - W_2) - (W_2 - W_1)}{(W_2 - W_1)} \times 100$$

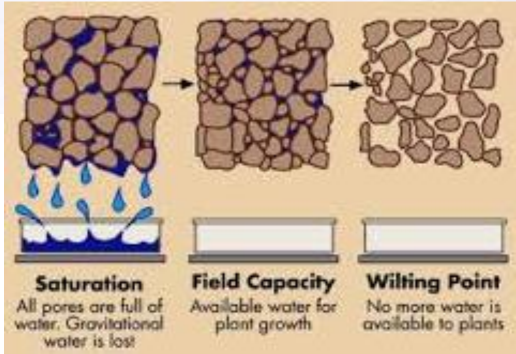
where W_1 = Weight of empty box

W_2 = Weight of box with dried soil

W_3 = Weight of box with water saturated soil

WHC = Water holding capacity.



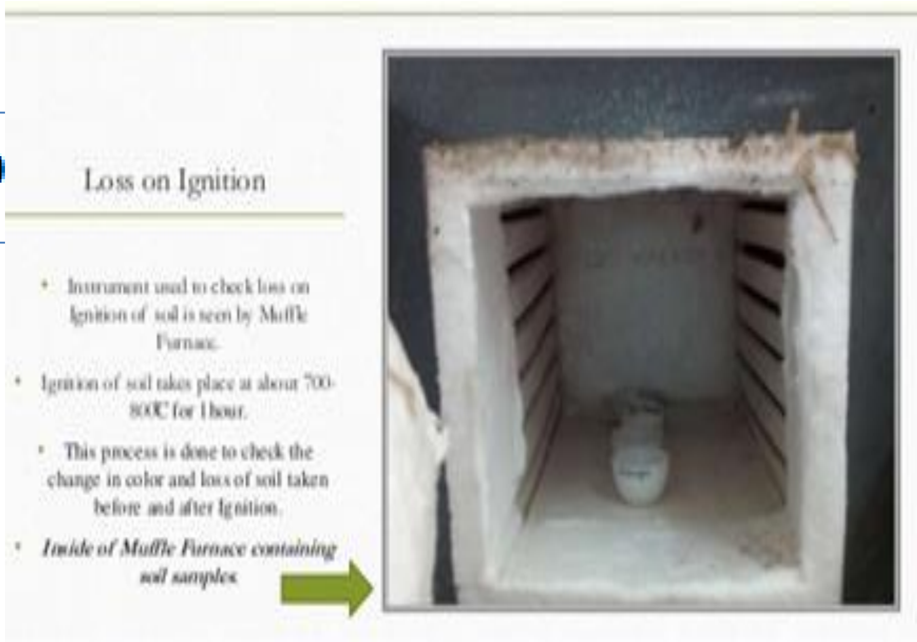


6. Determination of Loss of Ignition

- **Loss** on **ignition** (LOI) is one of the most widely used methods for measuring organic matter content in **soils** but does not have a universal standard protocol. A large number of factors may influence its accuracy, such as furnace type, sample mass, duration and temperature of **ignition** and clay content of samples.

Method

- Weigh the empty crucible that the sample is to be placed in and record it. Place the sample in the empty crucible and weigh the crucible again with the sample in it. Place the sample in the drying oven or blast furnace as required. Set the oven or furnace to the desired temperature at 700 – 800 ^{0}C in for over night. Allow the oven and sample to cool down before removing the sample from the oven. Weigh the crucible with the sample again. Subtract the empty crucible weight from this new weight and that is the sample's dry weight.



Calculation:

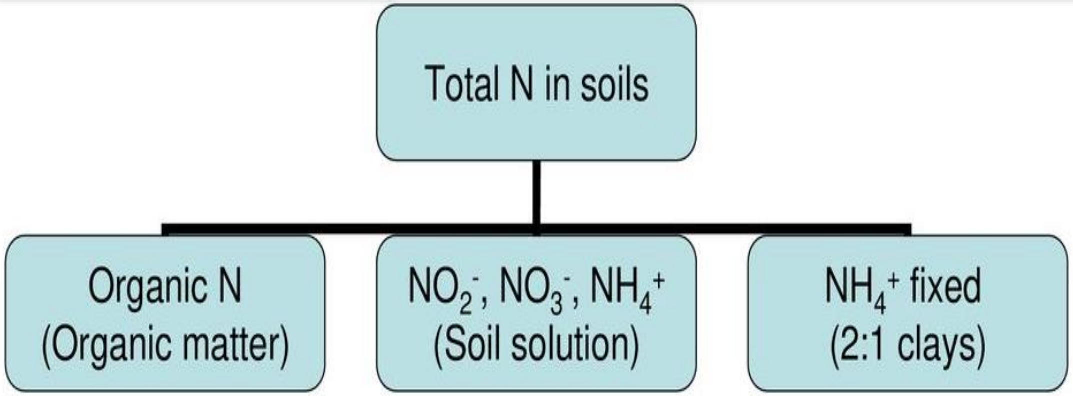
$$\% \ loss \ of \ ignition = \frac{x_1 - x_2}{x_1} \ X \ 100$$

Where,

X_1 = Initial weight of sample in grams

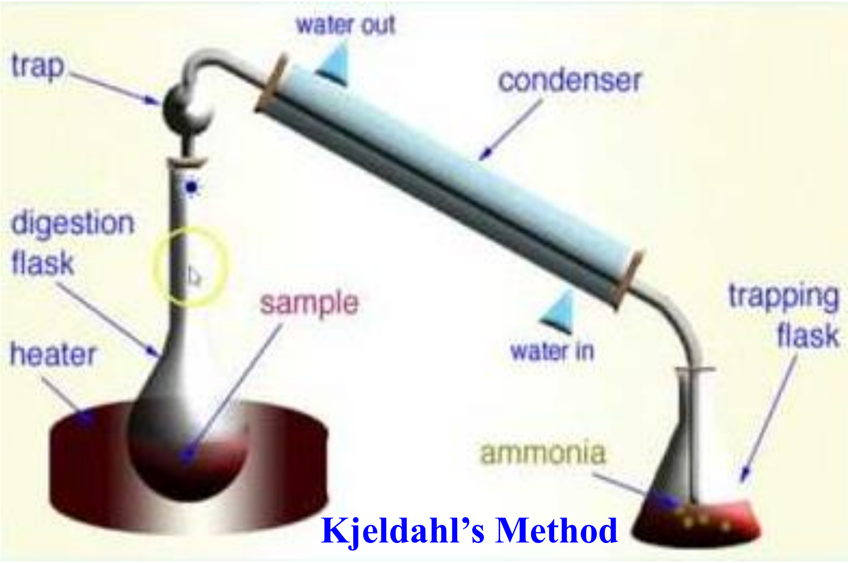
X_2 = Final weight of sample after ignition

7. Determination of Total Nitrogen by Kjeldahl's Method



- NO_2^- is seldom present in detectable amount
- Amount of NO_3^- in agricultural soil is usually low
- In soils with high amount of 2:1 type of clay minerals fixed NH_4^+ may account for about 10% of total N.

Total N ≈ Organic N + NH_4^+ (in soil solution)



Kjeldahl's Method (1833)

- ❑ Samples are digested with **H_2S0_4** to convert **N** in the sample to **$(NH_4)_2S0_4$**.
- ❑ Determination of the **NH4$^+$** in the digest by distillation and titration.
- ❑ Wet oxidation process

Digestion

1. $$\text{Organic N} + H_2SO_4 \xrightarrow[(Na_2SO_4)]{(Se\text{-}CuSO_4)} (NH_4)_2SO_4 + H_2O + CO_2$$

2. H_2SO_4 – Oxidizing agent used to oxidize organic N to $(NH_4)_2SO_4$.

3. Na_2SO_4 – salt used to rise the temperature of the digestion.

4. Se-$CuSO_4$ - catalyst used to speed up the oxidation rate.

❑ Kjeldahl's Method:

1. Digestion

- Weigh 10gm soil into 500ml Kjeldahl flask.
- Add10ml distilled water and leave to stand for 10 minutes.
- Add the catalyst, Add 2 tablets Na_2S0_4.
- Add 20ml Conc. H_2S0_4.
- Digest until clear and colourless (1-1.5hrs).
- Further digest for 30-45 minutes.
- Allow the flask to cool, Decant or filter the fluid into a100ml volumetric flask washing the sand with altogether 50ml distilled water, small at a time.
- Make up to the mark with distilled water.

2. Distillation

i) Free NH_4 is liberated from the solution by steam distillation in the presence of excess NaOH:

$$(NH_4)_2SO_4 + 2NaOH \xrightarrow{heat} 2NH_3 \; Na_2SO_4 + 2H_2O$$

ii) The NH_3 evolved is collected in excess boric acid to form ammonium-borate complex.

$$NH_3 + H_3BO_3 \longrightarrow NH_4^+ : H_2BO_3^- + 2H_2O$$

1. Transfer an aliquot of 10ml of the liquid into the Kjeldahl apparatus
2. Add 15 ml of the strong NaOH.
3. Distil over steam for 10 minutes into 10 ml of the Boric acid in a 500 ml conical flask.

3. Titration

The amount of NH_3 liberated and captured by boric acid is determined by titrating with 0.1N HCl

$$NH_4^+ : H_2BO_3^- + HCl \longrightarrow NH_4Cl + H_3BO_3$$

1. Add 8 drops of the mixed indicator.
2. Titrate with 0.IN HCl till colour changes to grey and then suddenly flashes to pink.
3. Carry out blank determination with distilled water.

4. Calculation

$$Kjeldahl's\,Nitrogen\left({}^{mg}/_{g}\right) = \frac{(V_1 - V_2)\,X\,N\,X\,14}{x}$$

$$Kjeldahl's\,Nitrogen\,(\%) = \frac{(V_1 - V_2)\,X\,N\,X\,1.4}{x}$$

Where,
V_1 = Vol. of titrant is used against **sample** in ml.
V_2 = Vol. of titrant is use against distilled water (**blank**) in ml.
N = Normality of titrant **(0.1N).**
x = Wt. of **soil** is used in **grams**.

8. Determination of Nitrate by Laboratory Method

❑ **Nitrate**-nitrogen (NO_3-N) measures the amount of available nitrogen in the **soil** that can be absorbed immediately by plants. To **determine** the amount of nitrogen fertilizer needed to meet crops' demand, the NO_3-N concentration is being subtracted from the nitrogen requirement of the crop.

Material

Laboratory reagents (in addition to those used for nitrate estimation in water as given below :

(a) *Extraction reagent.* (a) Weigh 12.5 g of copper sulphate. Dissolve it in distilled water to make 100 ml of solution. (b) Weigh 0.6 g of silver sulphate. Dissolve it in distilled water to make 100 ml of solution. Mix 20 ml and 100 ml of solutions (a) and (b) respectively. To this add distilled water to make 1 l of nitrate extraction reagent.

(b) *Calcium hydroxide.* Dry, powdered.

(c) *Magnesium carbonate.* Dry, powdered.

Method

Dry the soil/sediment in air. Now weigh 50 g of it and put it in an Erlenmeyer flask (500 ml). To this add 250 ml of extraction reagent (reagent A). Shake for 15 minutes. To this add 0.4 g of calcium hydroxide (reagent B), shake for 5 minutes. Then add 1 g of magnesium carbonate (reagent C). Now filter the contents through filter paper (Whatman No. 50). Then measure the total volume of filtrate. Find out the nitrate content in filtrate by using phenol disulphonic acid method discussed for water.

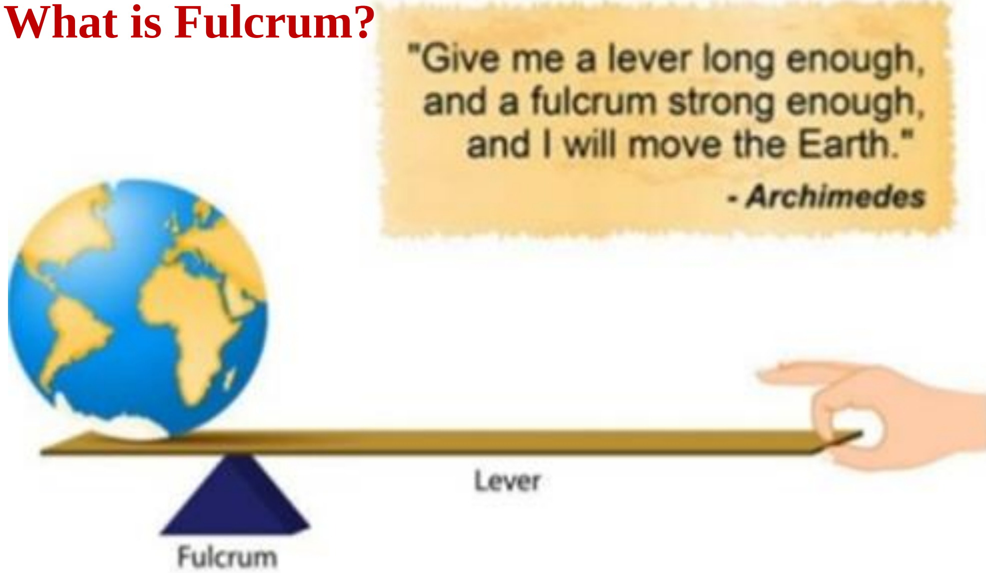
Calculation

$$NO_3—N\ (mg/g) = \frac{Y \times V}{1000 \times X}$$

where, $Y = NO_3—N$ determined in filtrate (mg/l) ; V = total volume of filterate (ml) ; and X = weight of dried soil/sediment used (g).

9. Determination of Organic Matter

What is Fulcrum?



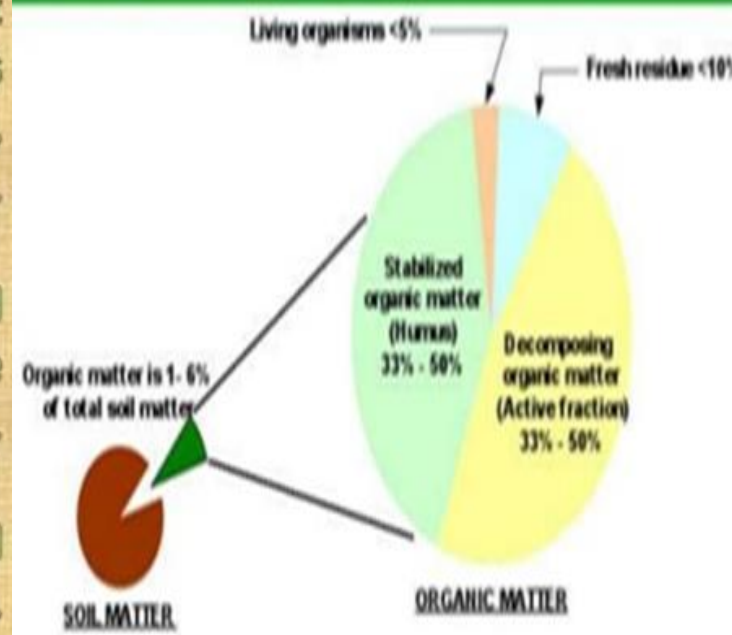
What is Soil Organic Matter?

Soil organic matter (SOM) is the organic matter component of soil, consisting of plant and animal residues at various stages of decomposition, cells and tissues of soil organisms, and substances synthesized by soil organisms.

SOM exerts numerous positive effects on soil physical and chemical properties, as well as the soil's capacity to provide regulatory ecosystem services.

Particularly, the presence of SOM is regarded as being critical for soil function and soil quality.

Components of Soil Organic Matter



Material

(*a*) *Potassium dichromate solution* (1*N*). It is prepared by dissolving 49.04 g of potassium dichromate in distilled water to get 1 *l* of solution.

(*b*) *Sulphuric acid* (concentrated).

(*c*) *Phosphoric acid* (concentrated).

(*d*) *Sodium fluoride*. Dry, powdered.

(*e*) *Diphenylamine indicator*. It is prepared by dissolving 0.25 g of diphenylamine in 10 ml of distilled water and 50 ml of concentrated sulphuric acid is gradually added to it.

(*f*) *Ferrous ammonium sulphate solution* (0.5 *N*). 20 ml of concentrated sulphuric acid, is added to 800 ml of distilled water. Then dissolve in it 196.1 g of ferrous ammonium sulphate. Further add more distilled water to make the volume 1 *l*.

Method

Dry the soil-sediment in air. Now weigh 0.5 g of it. Then put it in 500 ml Erlenmeyer flask. To this add 10 ml of potassium dichromate solution (reagent A) and gradually 20 ml of sulphuric acid (reagent B). Keep it as such for about half an hour. To this add 200 ml of distilled water, 10 ml of phosphoric acid (reagent C), 0.2 g of sodium fluoride (reagent D), and 1 ml of diphenylamine indicator (reagent E). Then contents are titrated against ferrous ammonium sulphate solution (reagent F). At the end point the dull green colour gets changed through turbid blue to the brilliant green.

Run a distilled water blank simultaneously.

Calculation

$$\text{OM (mg/g)} = \frac{6.791}{X}\left[1 - \frac{V_1}{V_2}\right] \times 10$$

$$\text{OM (\%)} = \frac{6.791}{X}\left[1 - \frac{V_1}{V_2}\right]$$

$$\text{Carbon (\%)} = \frac{6.791}{X \times 1.724}\left[1 - \frac{V_1}{V_2}\right]$$

In the above relation, OM = organic matter ; X = weight of soil/sediment taken (g) ; V_1 = volume of titrant used against sample (ml) ; and V_2 = volume of titrant used against distilled water blank (ml).

10. Determination of Sodium & Potassium

❑ Sodium

Material

(*i*) All reagents which are needed for determination of sodium in water ; (*ii*) filter paper (Whatman No. 50) ; and (*iii*) other reagents, such as given below :

(*a*) *Ethyl alcohol.* Absolute and 40%.

(*b*) Ammonium acetate solution.

Method

Preparation of soil extract. Dry the soil/sediment in air. Now put 50 g of it in a flask. To this add 100 ml of 40% of ethyl alcohol (reagent A). Then shake well, wait for about 10 minutes, and filter the suspension through filter paper (Whatman No. 50). Then wash the soil residue on filter paper with 40% ethyl alcohol and finally with absolute ethyl alcohol. Now put the residue into a beaker. To this add 100 ml of ammonium acetate solution (reagent B). Now stir, and keep it overnight. Filter the supernatant through filter paper (Whatman No. 50). Then collect the filtrate (soil extract). Measure out the total volume of soil extract.

Find out the sodium content in extract by using the method of sodium determination in water.

Calculation

$$\text{Sodium (mg/g)} = \frac{X \times V}{W \times 10000}$$

where, X = sodium content of soil extract (mg/l) ; V = total volume of soil extract (ml) ; and W = weight of air-dry soil/sediment which is taken for extraction (g).

❑ Potassium

Material

(*i*) All reagents, needed for the estimation of potassium in water ; (*ii*) filter paper (Whatman No. 50) ; and (*iii*) other reagents, such as given below :

(*a*) *Ethyl alcohol.* Absolute and 40%.

(*b*) Ammonium acetate solution.

Method

The soil extract is prepared by the method as described for determination of Sodium. Determine the potassium in extract as it has been determined in water.

Calculation

$$\text{Potassium (mg/g)} = \frac{Y \times V}{X \times 10000}$$

Here, Y=potassium content of soil extract (mg/l) ; V=total volume of soil extract (ml) ; and X=weight of air-dry soil/sediment which is taken for extraction (g).

……SUGGESTIONS ?

Created by, Dr. Subhash Lonkar

Thank you…..