EXPERIMENT 3 DETERMINATION OF PERCENT ORGANIC MATTER CONTENT IN A SOIL SAMPLE

Structure

3.1 INTRODUCTION

You learnt about sampling of soil and determination of its acidity/ alkalinity by measuring its pH in the previous two experiments. In this experiment you will learn about the common method of determination of organic matter in soil. Organic matter (OM) constitutes about 5 % of the bulk soil volume and consists of substances of organic origin like, plants, animals and microbial residues, plant residues being predominant. These residues may be in decomposed or in undecomposed form. You have read in Unit 2 Sec 2.4 that the highly decomposed residues generate humus - a brownish black coloured material that contributes to the structure, water - holding capacity and cation exchange capacity (CEC) of soil. It is important to determine the soil organic matter as it is an index of its productivity.

Soil organic matter is estimated in terms of organic carbon (OC) content which in turn can be determined by dry decomposition of soil. In this method the carbon present in the soil is oxidised to CO_2 which is absorbed in a suitable absorbent and determined. Though accurate, it is quite a tedious method and is generally replaced by wet oxidation methods which provide a reasonably accurate estimate of organic carbon. In this experiment you will use a wet digestion method explained in the next section. An alternative procedure involves the use of a UV - VIS spectrophotometer and is beyond the scope of this course.

Objectives

After performing this experiment you should be able to:

- explain the significance of determination of organic matter as percent organic carbon content,
- explain the principle and procedure involved in the wet digestion method of determination of organic matter content,
- prepare the reagents required for the determination of carbon content in the soil,
- analyse a given soil sample for carbon content using wet digestion method and explain the chemical reactions involved, and
- interpret the value of organic carbon in terms of productivity rating of the soil.

3.2 PRINCIPLE

The wet digestion method, also called as **chromic acid digestion method**, makes use of oxidation of the soil sample on heating with potassium dichromate and sulphuric acid. A known amount of soil is oxidised by adding a known amount of standard potassium dichromate solution and sulphuric acid. Potassium dichromate is reduced in the process. After the oxidation, the excess potassium dichromate, not reduced by soil organic matter, is determined by titration with standard ferrous ammonium sulphate (FAS) solution using N- phenylanthranilic acid as indicator. Alternatively, 1% diphenylamine can be used as the indicator in presence of phosphoric acid and sodium fluoride.

The reactions involved may be written as:

i) Oxidation of organic carbon

Potassium dichromate in presence of H_2SO_4 gives nascent oxygen which oxidises carbon to carbon dioxide.

$$[K_2Cr_2O_7 + 4H_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3O] \times 2$$
$$[C + 2O \longrightarrow CO_2] \times 3$$

$$2K_2Cr_2O_7 + 8H_2SO_4 + 3C \longrightarrow 2K_2SO_4 + 2Cr_2(SO_4)_3 + 8H_2O + 3CO_2$$

ii) Titration of excess nascent oxygen (liberated by dichromate + sulphuric acid) with ferrous ammonium sulphate solution

Ferrous ammonium sulphate or Mohr's salt provides ferrous ions (ferrous sulphate) that get oxidised by the nascent oxygen in presence of sulphuric acid.

$$[FeSO_4 . (NH_4)_2SO_4 . 6H_2O \longrightarrow FeSO_4 + (NH_4)_2SO_4 + 6H_2O] \times 2$$
$$2FeSO_4 + H_2SO_4 + O \longrightarrow Fe_2(SO_4)_3 + H_2O$$

$$2FeSO_4.(NH_4)_2SO_4.6H_2O + H_2SO_4 + O \longrightarrow 2(NH_4)_2SO_4 + Fe_2(SO_4)_3 + 13H_2O$$

iii) Action of diphenylamine indicator

$2C_6H_5.NH.C_6H_5$ (colourless) $\longrightarrow$ $2(C_6H_5NHC_6H_4)$

$-H_2O$ | $+ O$

$C_6H_5\text{-}N\text{-}C_6H_4.C_6H_4\text{-}N\text{-}C_6H_5$

diphenylbenzidine (violet colour)

In the wet oxidation method there is partial oxidation of the soil, therefore the values of carbon content obtained from this method are multiplied by a correction factor of 1.15. Further, since the organic matter (OM) on an average contains 58% carbon, the measurement of organic carbon (OC) content gives a rough estimate of soil organic matter and not its true value. Therefore, the percent of organic carbon is multiplied with another factor (100/58 or 1.724) known as **Bemmlen factor**, to get the true estimate of soil organic matter. These two factors (1.15×1.724) can be clubbed into a single factor of 2. i.e.,

1.15 is a correction factor applied to account for partial oxidation of the soil in wet digestion method.

$$\%\ OM = \%OC \times 2$$

The percentage of organic carbon determines the rating of the soil. A soil with percentage of organic carbon < 0.5 is rated as 'low' a value between 0.50 – 0.75 carries the rating as 'medium' while a soil with percentage of organic carbon > 0.75 is rated as the one of 'high' carbon content.

3.3 REQUIREMENTS

Apparatus

1. No. 10 (2 mm) sieve or mesh
2. 500 cm^3 Erlenmeyer flasks/ Conical flasks with ground glass top
3. Pipette
4. Burette
5. Reflux condenser
6. Analytical Balance

Chemicals

1. Conc. sulphuric acid (18 M)
2. Sodium fluoride powder
3. Orthophosphoric acid (85%)
4. Potassium dichromate solution (0.05 M) A.R
5. Mohr's salt solution (0.2 M)
6. Diphenylamine or N-phenylanthranilic acid

3.4 SOLUTIONS PROVIDED

You will be provided with these solutions. However, if required these can be prepared by the following procedures:

1. **Potassium dichromate solution (0.05 *M*):** Weigh 14.709 g of $K_2Cr_2O_7$ and transfer it to a 1000 cm^3 volumetric flask. Add 400 cm^3 of distilled water to the flask and dissolve $K_2Cr_2O_7$ by gently swirling the flask. Then carefully add 550 cm^3 of conc. H_2SO_4 to it in small lots mixing the solution after every addition. Make up the volume to the mark after allowing the solution to cool.

CAUTION!
Addition of H_2SO_4 to aqueous solution is highly exothermic, the flask would become quite hot, handle with care.

You would recall from the Chemistry Lab –1 quantitative experiments that H_2SO_4 is added to a $K_2Cr_2O_7$ solution to make the medium acidic as $K_2Cr_2O_7$ acts as an oxidising agent only in the presence of an acid.

2. **Mohr's salt solution (0.2 *M*):** Weigh 78.390 g of Mohr salt (ferrous ammonium sulphate [$Fe SO_4 .(NH_4)_2SO_4.6H_2O$]) and transfer it to a 1000 cm^3 volumetric flask. Add 500 cm^3 water and 50 cm^3 conc. H_2SO_4 to dissolve the salt. After cooling, the volume is made up to the mark. **This solution should be prepared fresh whenever required** (The Mohr's salt gets oxidised on keeping in air for long).

3. **Indicator solution:**
   N-phenylanthranilic acid : Weigh 20 mg of N-phenylanthranilic acid and transfer it to a 100 cm^3 volumetric flask. Dissolve in 0.2% aqueous solution (2 g per 1000 cm^3) of sodium carbonate.

   Or

   1% Diphenylamine : Dissolve 1.0 g of diphenylamine in 100 cm^3 of concentrated sulphuric acid containing about 15 % water.

3.5 PROCEDURE

You would have learnt to collect soil samples while performing Experiment 1 of this course. Let us recall the procedure used.

Soil Sample Collection

It should be kept in mind that soil samples taken from a lawn should be taken from the top 2 or 3 inches of soil. Samples from gardens or farm crops should be taken from the top 6 to 8 inches of soil. When analysing a large area of soil, samples taken from several locations should be mixed together. The steps given below should be followed to collect soil sample.

1. Use a clean trowel, soil sample tube, spoon, or knife to collect the samples.
2. Collect 4 or 5 heaping teaspoons of soil.
3. Place sample in plastic sampling bags.
4. Do not touch soil any more than is necessary.

Determination of Percent Organic Matter Content in a Soil Sample

5. Place collected sample on a piece of paper or plastic for several hours or overnight to dry.
6. Remove all stones, leaves, roots, and other foreign material.
7. Crush soil so that all lumps are smaller than 1½" in diameter.
8. Pass through a mesh depending upon the fineness of soil required.

Perform the following steps to determine the percent organic matter content in a soil sample.

1. Weigh 0.1 to 0.5 g of air dried sample of < 10 - mesh soil (ground and passed through 2 mm mesh) and transfer it to a 500 cm^3 Erlenmeyer flask or conical flask and add 15 cm^3 of 0.05 M $K_2Cr_2O_7$ solution to it.
2. Fix a reflux condenser to the flask and boil the contents of the flask for 30 min and leave it for cooling. In an alternative method the heat generated by the dilution of sulphuric acid is used for accelerating the rate of the reaction.
3. Put some distilled water from the top of the condenser so as to wash it into the flask.
4. Add 3-4 drops of N-phenylanthranilic acid indicator solution to this and titrate the violet coloured solution with 0.2 M Mohr's salt solution at room temperature. Add a few more drops of the indicator solution towards the end point. At the end point the colour changes from violet to bright green.
   (*If the above indicator is not available then you may add 5-6 drops of diphenylamine indicator, 5 cm^3 of orthophosphoric acid and a pinch of NaF powder. At the end point diphenylamine becomes colourless and of the solution in the flask changes to green due to ferrous ions.)*
5. Record the volume of ferrous ammonium sulphate solution used in the titration.
6. Perform a blank titration following the same procedure without using soil and record the volume of ferrous ammonium sulphate solution used for it.

H_3PO_4 and NaF make the colour change distinct and end point reliable by forming complex with the Fe^{3+} ions generated during the titration.

3.6 OBSERVATIONS AND CALCULATIONS

Mass of the soil sample taken = X g

Volume of 0.2 M ferrous ammonium sulphate (FAS) solution used for the blank titration = A cm^3

Volume of 0.2 M FAS solution used for titrating the excess of $K_2Cr_2O_7$ in sample titration = B cm^3

Volume of 0.2 M FAS solution used for the oxidation of carbon. = $(A - B)$ cm^3

Thus volume of 0.05 $K_2Cr_2O_7$ used for the oxidation of carbon (see margin) = $2(A - B) / 3$ cm^3

$M_F V_F = 6\ M_D V_D$
$\Rightarrow V_D = M_F V_F / 6\ M_D$
$\Rightarrow V_D = 0.2\ V_F / (6 \times 0.05)$
$\Rightarrow V_D = 2/3\ V_F$

Amount of organic carbon in X g of the soil = $(A - B) \times 2/3 \times 0.0009 \times 1.15 = Z$ g
(1.15 is the correction factor for incomplete oxidation of the organic carbon)

1 cm^3 of 0.05 M $K_2Cr_2O_7$ = 0.0009 g organic carbon

Percent organic carbon in the soil = $Z \times 100 / X = Z'$ = ------ %

Percent organic matter = $Z'' = Z' \times 1.724$ %

(100/58 = 1.724 is the Bemmlen factor discussed earlier)

3.7 RESULTS

Percent organic carbon calculated by titration method in the given soil sample (on dry weight-basis) was found to be

$= Z' =$ ---------- %

Percent organic matter calculated by titration method in the given soil sample on dry weight-basis was found to be

$= Z'' =$ ---------- %

The rating of the soil sample in terms of percent organic carbon in soil was found to be -

low/medium/high (delete the incorrect options)

3.8 PRECAUTIONS

- Use clean and dry apparatus.
- Solution of ferrous ammonium sulphate should be freshly prepared .
- All reagents should be prepared in distilled water.
- During the titration the change in colour should be carefully observed.
- Add conc. H_2SO_4 in small lots mixing the solution after every addition.
- While adding sulphuric acid care should be taken that the flask does not become too hot.