Chapter 2

Soil Sampling and Analysis
2.1 SOIL SAMPLE COLLECTION

Soil sampling is a useful farm management tool. According to an axiom, test results can be no better than the sample collected in the field. Proper collection of soil sampling is extremely important. Improper sample can result in erroneous recommendations. For the collection of soil sample, the importance of taking representative composite soil sample was kept in mind and variations in soil color, texture, slope, fertilization or cropping practice all were adequately considered. During sampling, the grass and other plant residues were removed from surface of the soil. The soil (0~30 cm depth) from 10-15 well described spots in fields were collected by using shovel or auger and stored in a bucket, broken the lumps, removed the stones and mixed well and placed this composite soil sample in the soil sample bag. The different soil samples were collected from different places of Aligarh district. Soil sample S₁ was collected from the field in the neighborhood of Sankra bus stand about two furlong from the river bank, S₂ was collected from the botanical garden Aligarh Muslim University Fort, S₃ from Jattari town in Khair tehsil, S₄ from the field of Malhepur village, situated on the Aligarh-Ramghat Road, S₅ from Talaspur village in koil tehsil and S₆ was collected from Narwari village in khair tehsil.

2.2 DETERMINATION OF PHYSICO-CHEMICAL PROPERTIES OF SOIL

The Physico-chemical properties of soil were determined as follows:

2.2.1 Determination of Mechanical Composition

The Mechanical Compositions of soil sample were determined by international pipette method [1].

Apparatus and Reagents Required

7,200 mesh sieves (BSS), graduated boiling tube (500 mL), constant temperature water bath, pipette, petridish, electronic balance, 30% hydrogen peroxide, 0.2 N hydrochloric acid, sodium oxalate solution (8 g L⁻¹).

Procedure

A 10 g of soil sample previously passed through 7 mesh sieve (BSS) was dispersed in water after treatment with 30% H₂O₂ to remove any organic matter and 0.2 N HCl and 50 mL sodium oxalate (8 g L⁻¹) as dispersing agent. The percentage of sand was
calculated from the weight of residues left behind on 200 mesh sieve (BSS). The suspension was then diluted to 500 mL and transferred to a graduated boiling tube, which was immersed in a constant temperature water bath at 25 ± 1°C throughout the course of pipetting. A 10 mL sample was pipetted out carefully in a petridish at specified intervals (4 min 15 sec and 7 hr 5 min) of time from a depth of 10 cm. It was dried and weighed. The percentage of clay was calculated from the weight of residues. The percentage of silt was calculated by subtracting the sum of percentage of all the fractions (sand plus clay) from 100.

2.2.2 Determination of Soil pH

The pH was recorded with Elico pH meter (model LI 120) with glass and saturated calomel electrodes assembly. A 1:2, soil : water ratio suspension was used for measuring the pH of the soil.

2.2.3 Determination of Electrical Conductivity (EC)

The electrical conductivity was measured at 30 ± 1°C with the help of Philips Conductivity Bridge and dip type cell. A 1:2, soil : water ratio suspension was used to measure the electrical conductivity.

2.2.4 Determination of Organic Matter

The organic matter of the soil was estimated by using Walkley and Black [2] method.

Reagents Required

Aqueous solution of 1 N potassium dichromate, conc. sulfuric acid, orthophosphoric acid (85%), 1% alcoholic solution of diphenylamine indicator, aqueous solution of N/2 ferrous ammonium sulfate.

Procedure

A 2 g of soil sample was taken in 500 mL conical flask. A 10 mL of 1 N potassium dichromate solution and 20 mL of concentrated sulfuric acid were added to it. The flask was shaken vigorously several times, allowed to stand for 30 min and thereafter a 200 mL of distilled water; 10 mL of orthophosphoric acid and 1 mL of diphenylamine indicator were added to it. The excess of unreacted potassium dichromate was titrated against standard N/2 ferrous ammonium sulfate solution till the violet color changed to purple and finally to green. Reagent blank determination
was also carried out in the same way. From the volume of ferrous ammonium sulfate solution used, organic carbon was calculated by using the expression:

\[
\frac{(\text{Blank titre} - \text{Actual titre}) \times 0.003 \times N \times 100}{\text{Weight of dry soil in g}}
\]

Where,

\[
N \text{ is the normality of ferrous ammonium sulfate solution.}
\]

The value of organic carbon was converted to organic matter by multiplying with the factor 1.724.

2.2.5 Determination of Cation Exchange Capacity (CEC)

The cation exchange capacity of the soil was determined by the method of Jackson [3].

**Reagents Required**

- 0.05 N hydrochloric acid
- 1 N sodium acetate solution (pH 5)
- 1 N calcium chloride solution
- EDTA solution
- 1% alcoholic solution of eriochrome black "T"
- Buffer solution of pH 10
- 2% aqueous solution of sodium cyanide

**Procedure**

A 5 g soil sample was taken in a 100 mL conical flask. The soluble salts were washed out by treating the soil with 0.05 N HCl and finally with distilled water. It was further treated with 1 N sodium acetate of pH 5 for 30 min with intermittent stirring. This acidified sample was given five washings with 1 N standard calcium chloride solution. The excess salts were removed by washing with 80% aqueous acetone solution until the excess CaCl₂ was removed as indicated by a negative AgNO₃ test for chloride ion on the final washing. Finally the calcium ions were exchanged from Ca–soils by means of exchanging it with a neutral 1 N sodium acetate solution. The washing were collected and utilized in the determination of replaced Ca²⁺ from exchangeable sites and titrating it with a standard EDTA solution, using 10 mL of buffer solution (NH₄Cl-NH₄OH) of pH 10 and eriochrome black "T" indicator in the presence of 1 mL of 2% NaCN solution as masking agent for interfering ions. A reagent blank was also run simultaneously to avoid any error due to impurities. The blank reading was subtracted from the reading of calcium determination. From the volume of the EDTA
solution used, the value of cation exchange capacity was calculated by using the following expression:

\[
\text{Cation exchange capacity (meq 100 g}^{-1}\text{ soil)} = \frac{V \times N \times 100}{\text{Weight of soil in g}}
\]

Where,

- \(V\) is the volume of EDTA and \(N\) is the normality of EDTA solution used.

2.2.6 Determination of Exchangeable Cations

The exchangeable cations of soil were determined by method of Jackson [3].

Reagents Required

- 1 N aqueous solution of ammonium acetate, 6 N nitric acid, 30% H\(_2\)O\(_2\), 6 N hydrochloric acid, buffer solution (pH 10), eriochrome black “T”, mureoxide indicator, 10% potassium hydroxide, EDTA solution, standard solution of Na and K.

Procedure

A 50 g soil sample was taken into 250 mL conical flask and then 100 mL 1 N NH\(_4\)OAc solutions was added to it. The contents of the flask were shaken for 20 min and allowed to stand overnight. The soil contents were then transferred into a buckner funnel, in which moist Whatman filter paper (No. 42) was seated by using a gentle pressure. The soil was leached with an additional 400 mL NH\(_4\)OAc. The filtrate containing NH\(_4\)OAc extract of soil was evaporated to dryness on a steam plate. The dark color residue, containing organic matter was treated with 2 mL of 30% H\(_2\)O\(_2\) and 2 mL of 6 N HNO\(_3\), and heated to dryness on a steam plate. The dried organic matter free residue was then dissolved in 10 mL of 6 N HCl and diluted with distilled water. It was filtered through Whatman filter paper (No. 42) and the volume was made upto 100 mL. This solution was used for the determination of exchangeable Na\(^+\), K\(^+\), Mg\(^{2+}\), and Ca\(^{2+}\) in soil. Exchangeable calcium plus magnesium was estimated in the 10 mL of above solution by EDTA titration, using a half test tube of buffer solution (pH 10) and 4-5 drops of eriochrome black “T” indicator. Calcium was also estimated separately using mureoxide indicator with 10% KOH as recommended by Jackson [3]. The volume of EDTA solution for magnesium was calculated by subtracting the volume for calcium from the volume of calcium plus magnesium used. The
exchangeable sodium and potassium were estimated in the above solution using "Systronics" flame photometer. Standard curves (Fig. 2.1) of sodium and potassium were used for calculating the amounts of Na and K in the above soil solution.

2.2.7 Base Saturation (%)

It is defined as the percentage of total CEC occupied by basic cations (Na\(^+\), K\(^+\), Mg\(^{2+}\), and Ca\(^{2+}\)).

\[
\text{Base saturation} \, (\%) = \frac{\text{Total bases} \, (\text{meq} \, 100\, \text{g}^{-1} \, \text{soil})}{\text{CEC} \, (\text{meq} \, 100\, \text{g}^{-1})} \times 100
\]

The availability of Na\(^+\), K\(^+\), Mg\(^{2+}\) and Ca\(^{2+}\) to plants increases with increasing BS (%).
Fig. 2.1 Standard curves for sodium and potassium by flame photometer
2.2.8 Determination of Available Nitrogen

Available nitrogen was determined by the method of Hesse [4].

2.2.8.1 Determination of Ammonium Nitrogen

Reagents Required

- **2 M Potassium chloride solution**: A 150 g KCl was dissolved in 800 mL distilled water and boiled with 10 g magnesium oxide for about 15 min, until any ammonia present was expelled. The solution was then cooled and filtered before making the volume upto the mark.

- **Magnesium oxide**: Magnesium oxide was heated at 650°C for two hr in an electric muffle furnace and allowed to cool in a desiccator over solid KOH and stored in a tightly stoppered glass bottle.

- **Mixed indicator**: Mixed indicator solution was prepared by dissolving together a 0.1 bromo cresol green and 0.07 g methyl red in 100 mL of ethanol.

- **Boric acid solution**: A 20 g of boric acid was dissolved in 900 mL of hot distilled water. After cooling the solution, 20 mL mixed indicator solutions was added to it. And then a 0.1 M NaOH solution was added drop wise until the color is changed to reddish purple.

- **M/70 hydrochloric acid solution**.

Procedure

A 5 g soil sample was taken in 100 mL glass stopper conical flask. A 50 mL of 2 M KCl solution was added in it. The flask was shaken vigorously for an hr and the soluble contents were extracted through Whatman filter paper (No. 42). A 10 mL solution from extract was taken in distillation flask and diluted with 50 mL distilled water. A 0.5 g magnesium oxide was added through a long-stemmed funnel into the flask. Ammonia was distilled into a solution of 5 mL of boric acid containing mixed indicator through a condenser until the final volume of the distillate was reached to about 30 mL. The boric acid was then titrated with M/70 HCl by using a micro burette until the green color changed to pink. A blank was also run in the same way. The available ammonium nitrogen was calculated using the relation:
1 mL of M/70 HCl = 0.2 mg of nitrogen.

2.2.8.2 Determination of Nitrite Nitrogen plus Nitrate Nitrogen

Reagents Required

- All the reagents used in the determination of ammonium nitrogen.
- Devarda alloy: Finally grounded and passed through a 0.15 mm sieve.

Procedure

After distillation of ammonium nitrogen, the stopper of the distillation flask was removed and a 0.2 g of Devarda alloy and 50 mL of distilled water were added to it. The distillation flask was stoppered and then ammonia was distilled in a fresh portion of 5 mL boric acid solution until 30 mL distillate was collected and then it was titrated with M/70 HCl. The value of nitrite nitrogen plus nitrate nitrogen was calculated in the same manner as described in the determination of ammonium nitrogen.

2.2.8.3 Determination of Nitrate Nitrogen

Reagents Required

- All the reagents used in the determination of nitrite plus nitrate nitrogen.
- 2% sulphamic acid solution.

Procedure

The experimental procedure was partly the same as described in the determination of available ammonium nitrogen. After distillation of ammonia nitrogen the stopper of the distillation flask was removed and 1 mL of a 2% aqueous solution of sulphamic acid was added to the flask. The distillation flask was swirled for few sec to destroy the nitrite and then 0.2 g Devarda alloy and 50 mL distilled water were added in it. It was stoppered and ammonia was distilled in a fresh portion of boric acid solution. About 30 mL distillate was collected in each case and titrated with M/70 HCl. The value of nitrate nitrogen was calculated in same manner as described in the determination of ammonium nitrogen.

2.2.8.4 Determination of Nitrite Nitrogen

The value of nitrite nitrogen was calculated from the following expression:
Nitrite nitrogen = (Nitrite nitrogen + Nitrate nitrogen) – Nitrate nitrogen.

2.2.9 Determination of Available Phosphorus

Available phosphorus was determined by the method of Olsen [5]

Reagents Required
- 0.5 M sodium bicarbonate solution (pH 8.5).
- Draco G 60, phosphorus free charcoal.
- Chloromolybdic acid: (15 g ammonium molybdate + hydrochloric acid + 650 mL warm distilled water).
- Stannous chloride solution: (10 g SnCl₂ . 2H₂O was dissolved in 25 mL conc. HCl, then 0.5 mL of this solution was dilute with 66 mL distilled water).
- Standard phosphorus solution.

Procedure
A 5 g soil sample was taken in a 150 mL conical flask and one teaspoonful of charcoal was added to it. The sample was then treated with 100 mL of 0.5 M sodium bicarbonate solution. The content of the flask was shaken for 30 min on a mechanical shaker and kept over night. It was then filtered through a Whatman filter paper (No. 42).

A 5 mL of the clear soil extract was taken in 25 mL of measuring flask and 5 mL of chloromolybdic acid solution was added and the contents were thoroughly mixed. After shaking, 1 mL of stannous chloride solution was added and the volume was made up to 150 mL with distilled water. A blue color was developed. A blank was also run in the same way. The absorbance of the solution was recorded after 5 min with Bausch and Lomb using spectronic ‘20’ at a wave length of 660 nm. The concentration of phosphorus was determined by means of the standard curve (Fig. 2.2) already prepared for this purpose.
Fig. 2.2 Standard curve for phosphorus by spectrophotometer
2.2.10 Determination of Available Metals in Soil

The available metals present in soil was determined by the method of Tandon [6]

Apparatus and Reagents

- Double beam atomic absorption spectrophotometer (GBC-902).
- **DTPA extracting solution**: To prepare 1 L DTPA extracting solution, 13.1 mL reagent grade TEA, 1.967 g DTPA (AR Grade), 1.47 g of CaCl₂ and HCl were added in distilled water maintaining the pH of solution to 7.3.
- Standard solutions of metals (Cd, Cu, Fe, Mn, Mo, Ni and Zn).

Procedure

The extraction of metals from the soil was made by taking 10 g of soil with 20 mL of DTPA extracting solution for 2 hr with intermittent stirring. The suspension was then filtered through Whatman filter paper (No.42). Available Cd, Cu, Fe, Mn, Mo, Ni and Zn were determined directly from these extract by using double beam atomic absorption spectrophotometer (GBC-902).

2.2.11 Determination of Extractable Metals in Soil

1 g of air dried soil was taken in a conical flask and 100 mL of NH₄OAc was added. The contents were shaken at definite intervals and the flask was kept overnight. The contents were filtered and the residue was again mixed with 50 mL of NH₄OAc. The contents after shaking well were again filtered and the total filtrate was evaporated to dryness and later diluted with 10 mL 1 N HNO₃. The prepared solution was used for the determination of extractable metals by GBC-902 atomic absorption spectrophotometer [3].
REFERENCES


