CHAPTER 11

METHODS OF ANALYSIS AND CHARACTERISATION OF SOIL SAMPLES

COLLECTION AND PREPARATION OF SOIL SAMPLES

Soil samples were collected from Ahmedabad, Amreli and Kutch districts of Gujarat state. Samples of soils were collected from virgin land at different places. The surface layer of 2 inches - 5 inches depth was removed and representative layers of the profiles were sampled after digging a pit of 3" x 2". The various portion of a particular depth interval were intimately mixed and about 50-60 kg of the sample was placed in a jute bag with labels. A record slip was kept in each bag, showing the location of the site, description of various profile features. About 1 Kg of the original sample was stored in wide-mouth glass bottle for observation of structure, colour etc. The remaining sample was kept in sun-shine for air drying and ground in wooden pestle and mortar without crushing the ultimate particles. The sample was sieved through 2.0 mm sieve (10 mesh standard), which was then used for chemical and mechanical analysis.
PREPARATION OF SOILS SATURATED WITH DIFFERENT IONS

The normal soil was treated with 2.0% solutions of chloride salts of sodium, calcium, magnesium and ammonium for preparation of Na, Ca, Mg and NH₄ saturated soils respectively.

The 2.0% salt solution and normal soil were stirred vigorously and kept over night. Next day the supernatent salt solution was decanted and the relevant salt solution was added and the process was repeated thrice. After the removal of solution, the soil was leached with distilled water till free from excessive chloride and finally washed with alcohol, dried in sun-shine, ground in the wooden pestle and mortar and stored in ground glass wide-mouth glass bottles.

ANALYSIS OF THE SOIL SAMPLES

The following determinations were carried out with the soil samples, adopting standard procedures.

1. SOIL REACTION

The pH was determined in a soil suspension. The soil suspension was prepared using 1:5 soil water ratio (W/w) and 1 hr equilibration (20). The suspension was stirred well just before immersing the electrodes (15). Soil reaction was measured potentiometrically on systronics pH meter Model 335. The electrode combination consists of a glass electrode and calomel reference electrode. The value
were measured within 60 sec. after immersing the electrode in sample.

The pH determination reported as pHw (1:5).

2. ELECTRICAL CONDUCTANCE

Electrical resistance of the saturation extract (15, 22) was measured with conductivity bridge (Systronics Model DRT 303). A dip type conductivity cell was used for soil suspension. The results were reported as $E_c$ in Mho/cm at 25°C, by the multiplication of the appropriate factor in relation to temperature from the standard tables (6, 19).

3. ORGANIC MATTER AND ORGANIC CARBON

Organic matter was estimated by the wet oxidation titrimetric method (15, 25, 26). The heat of dilution of $H_2SO_4$ with 1N $K_2Cr_2O_7$ was the sole source of heat. The organic carbon was oxidized with chromic acid. The unused chromic acid was calculated by back titration. Total organic matter was calculated by multiplying carbon content by the conventional factor 1.724 based on the assumption that soil organic matter is 58%.

CATION EXCHANGE CAPACITY

The soil, saturated with 1N ammonium acetate, was adjusted to pH 7.0 (22). After leaching excess $NH_4\cdot\text{Ac}$ from $NH_4$ saturated soil in a buchner funnel with 95% ethanol, the amount of exchangeable Ammonium ion was determined by Kjeldahl distillation (1). Ammonia distilled from $\Theta_6$
alkaline medium was absorbed in standard H₂SO₄ and excess acid back titrated with standard NaOH solution (15).

The NH₄OAc leachate was retained for determination of exchangeable cations.

**SODIUM AND POTASSIUM**

Exchangeable Na and K were determined after extraction with 1N NH₄OAc (pH7) (22) by Flame photometer (A Elico model cl-22A) using oxygen LPG flame. Potassium was determined at 766 nm wavelength and sodium at 589 nm (11).

**CALCIUM AND MAGNESIUM**

Calcium and magnesium were determined by complexometric titration method. The NH₄OAc leachate was titrated with 0.01 M EDTA, a sequestering agent which forms un ionized complexes with Ca and Mg ions. First the total concentration of Ca and Mg was obtained using Eriochrome black T dye as indicator (7). To get a pH of 10.0 (3) the NH₄Cl and NH₄OH buffer of pH 10 (13) was used. In a separate aliquot, Ca was determined with EDTA using murexide as an indicator after precipitating Mg as Mg(OH)₂ by adding 4N NaOH solution to increase the pH to 12 (7). The titration was performed immediately after alkali addition. Magnesium was calculated from the difference between the above two titrations.
ESTIMATION OF EXCHANGEABLE SODIUM & POTASSIUM PERCENTAGE

Sodium adsorption and potassium adsorption ratios were calculated as follows (23).

\[
\text{Sodium Adsorption Ratio (SAR)} = \sqrt{\frac{\text{Na}^+}{(\text{Ca}^{++} + \text{Mg}^{++})/2}}
\]

\[
\text{Potassium Adsorption Ratio (PAR)} = \sqrt{\frac{\text{K}^+}{(\text{Ca}^{++} + \text{Mg}^{++})/2}}
\]

where all the concentrations are expressed in meq./Litre.

These ratios were used to calculate the exchangeable cation percentage as follows (14).

\[
\text{Exchangeable Sodium Percentage ESP} = \left(\frac{\text{Exchangeable Na}^+ \text{ meq/100gm}}{\text{CEC meq/100 gm}}\right) \times 100
\]

\[
\text{Exchangeable Potassium Percentage ESP} = \left(\frac{\text{Exchangeable K}^+ \text{ meq/100 gm}}{\text{CEC meq/100 gm}}\right) \times 100
\]

SOIL COLOUR

The soil samples were matched against standard Munsell soil colour charts to obtain hue, value and chroma combinations (18). The colour values were obtained for both moist and dry samples.
MECHANICAL ANALYSIS

Particle size analysis of the fine skeleton of particles under 2 mm was performed by International Pipette Method (21). The sample was treated with H₂O₂ to kill the organic matter (9,10) while alkaline earth carbonates were removed by sufficient dil. HCl (20). Dispersion was carried out by an electric soil dispersion stirrer. Calgon (sodium Hexa meta phosphate with sufficient Na₂CO₃ to give pH of about 8.5 in 10% solution) was used as a dispersing agent. Volume of dispersed solution adjusted to 1 litre in a cylinder. Coarse sand was separated by 0.2 mm sieve (70 mesh). Silt and clay fractions were determined by pipette from upper 10 cm depth after requisite time correlated with temperature. Fine sand was determined by Beaker method.

The soil texture class was determined from a textural triangle of International classification of particles (2).

W.H.C., SₚGₐ, BULK DENSITY, PORE SPACE AND VOLUME EXPANSION

The values were determined by the keen Raekowski box experiment using the circular shaped boxes described by Coutts (16).

The dimension of boxes used in laboratory differs slightly from those determined by Coutts. Their height is the same (1.6 cm), but the internal diameter of 5.6 cm was chosen. The bottom is perforated with numerous holes 0.75 mm in diameter spaced 4 mm centers. Place a thin 5.5 cm filter paper (Whatman No.1 or 44) on the bottom of circular
boxes and fixed it in position by means of the split brass spring. The values were obtained as per the methods.

**CLAY MINERALS**

For identification of clay minerals, X-Ray diffraction method (4) was used. The diffraction pattern was recorded on counter diffractometer with a strip chart on Cu Kα X-Radiations (= 1.543 Å) on Philips X-Ray generator unit model PW 1730.

Oriented aggregates of the crystals enhance the basal reflections considerably. Oriented specimen are prepared by allowing a clay water suspension to settle onto a flat glass plate; then dry out it gradually (17,24).

The first and 2nd order reflections obtained for Kaolinite, Montmorillonite and Illite are as follows:-

<table>
<thead>
<tr>
<th></th>
<th>1st order</th>
<th>2nd order</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kaolinite</td>
<td>7.11 Å</td>
<td>3.75 Å</td>
</tr>
<tr>
<td>Montmorillonite</td>
<td>14 Å</td>
<td>7.0 Å</td>
</tr>
<tr>
<td>Illite</td>
<td>10 Å</td>
<td></td>
</tr>
</tbody>
</table>

Diffraction pattern graph of clay treated with Glycol is made. Value at 11.14 and 17.7 Å indicate vermiculite, chlorite and montmorillonite respectively.

A diffraction pattern is made of clay that have been heated to 500°C for several hour to distinguish Montmorillonite from Kaolinite.
HYDRAULIC CONDUCTIVITY

Hydraulic conductivity was determined by using Witt's apparatus (27), described below and shown in Fig. (2.1).

The tests with this apparatus provide data on vertical hydraulic conductivity and correlation between hydraulic conductivity and granular composition, porosity, minor structures etc. The data can be used for comparison with data obtained from other methods and as a basis for computation of direction and intensity of flow under natural conditions. The apparatus consists of a brass cylinder, 5 cm in diameter and 11 cm in height with two parts.

The planning of drainage and irrigation projects is based on geo-hydrological investigations. These investigations are carried out to obtain an insight into the present hydrological situation to obtain hydrological constants of soil and to find out what criteria must be used for a good functioning of the future systems. The hydrological constants to be determined comprises the hydraulic conductivity 'K' and the transmissibility 'KD' and the vertical resistance or a leakage factor 'C' of the aquifer.

INFILTRATION RATE

The infiltration rates of the soil samples were measured by the tube method described below.

In order to understand the nature of infiltration rates continued over a short period, the following method was used,
which is a modification of the technique used by Dettman and Emerson (12). Of course, it is known that as the size of the tube is small, the measurement cannot be considered highly accurate, but in order to obtain comparative infiltration rates for different soils (disturbed soil samples) this method was adopted.

The soil was filled in a hard glass test-tube of length 20 cm and diameter 2.6 cm with an arrangement of transverse hole to maintain 2.0 cm height of water over the soil filled by stroke method. In the lower part, sand was filled by placing cotton plug in the hole below. The level of water was maintained constant by adding water or solution from the reservoir.

Table No.2.1 shows the characterisation chart for the soil samples.

Fig. No.2.1 shows the apparatus for the laboratory determination of hydraulic conductivity.

Fig. No.2.2 shows the infiltration rate tube.

Fig. No.2.3 shows X-Ray diffraction patterns for soil samples.
<table>
<thead>
<tr>
<th>Place of sampling</th>
<th>Depth cm</th>
<th>% Coarse sand 2-0.2</th>
<th>% Fine sand 0.2-0.02</th>
<th>% Total sand 2-0.02</th>
<th>% Silt 0.02-0.002</th>
<th>% Clay 0.002</th>
<th>Texture class</th>
<th>Clay Mineral</th>
<th>WHC %</th>
<th>Bulk Density %</th>
<th>Pore space %</th>
<th>Vol Exp. %</th>
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</thead>
<tbody>
<tr>
<td>AHMEDABAD</td>
<td>0-10</td>
<td>20.43</td>
<td>55.73</td>
<td>76.10</td>
<td>1.02</td>
<td>22.80</td>
<td>SCL</td>
<td>K-M</td>
<td>25.07</td>
<td>1.24</td>
<td>32.35</td>
<td>2.67</td>
</tr>
<tr>
<td>AMRELI</td>
<td>0-10</td>
<td>2.44</td>
<td>35.01</td>
<td>37.45</td>
<td>16.22</td>
<td>45.57</td>
<td>CLAY</td>
<td>M</td>
<td>63.33</td>
<td>1.09</td>
<td>59.8</td>
<td>12.26</td>
</tr>
<tr>
<td>ADIPUR (KUTCH)</td>
<td>0-10</td>
<td>3.42</td>
<td>34.92</td>
<td>37.95</td>
<td>12.75</td>
<td>48.87</td>
<td>CLAY</td>
<td>K-M</td>
<td>43.25</td>
<td>1.25</td>
<td>51.44</td>
<td>7.45</td>
</tr>
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### Table 2.1 (Contd.)

#### CHEMICAL ANALYSIS

<table>
<thead>
<tr>
<th>Place of sampling</th>
<th>Depth cm.</th>
<th>% Organic carbon</th>
<th>% Organic matter</th>
<th>% Carbonate as CaCO₃ &lt; 2mm</th>
<th>pH 1:5 Mmho</th>
<th>Ec 1:2.5 Mmho</th>
<th>Ca⁺⁺ Meq/100 gm Soil</th>
<th>Mg⁺⁺ Meq/100 gm Soil</th>
<th>Na⁺ Meq/100 gm Soil</th>
<th>K⁺ Meq/100 gm Soil</th>
<th>CEC Meq/100 gm Soil</th>
<th>ESP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ahmedabad</td>
<td>0-10</td>
<td>0.14</td>
<td>0.25</td>
<td>1.25</td>
<td>8.50</td>
<td>0.46</td>
<td>12.12</td>
<td>10.14</td>
<td>1.25</td>
<td>0.11</td>
<td>23.63</td>
<td>5.26</td>
</tr>
<tr>
<td>Amreli</td>
<td>0-10</td>
<td>0.28</td>
<td>0.48</td>
<td>6.28</td>
<td>8.80</td>
<td>1.17</td>
<td>32.30</td>
<td>17.21</td>
<td>8.45</td>
<td>2.40</td>
<td>60.32</td>
<td>13.96</td>
</tr>
<tr>
<td>Adipur (Kutch)</td>
<td>0-10</td>
<td>0.28</td>
<td>0.46</td>
<td>1.41</td>
<td>8.71</td>
<td>0.20</td>
<td>22.18</td>
<td>1.24</td>
<td>4.20</td>
<td>4.20</td>
<td>28.43</td>
<td>14.77</td>
</tr>
</tbody>
</table>
Apparatus for the laboratory determination of hydraulic conductivity (Constant head method).

1. Tap
2. Constant level measuring tank with overflow.
4. Nylon cloth.
5. Siphon.
X-RAY DIFFRACTION PATTERN

ADIPUR KUTCH SOIL
(K1)

AMRELI SOIL
(M)

AHMEDABAD SOIL
(KMI)
REFERENCES


