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## Sampling and analysis of soil and water: A review

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### Abstract

Within any project for the reclamation and cultivation of new areas, representative samples must be collected and tested in the laboratory in order to know their physical, chemical and fertility characteristics. Here comes the role of soil and water researchers in applying standard methods for sample collection and analysis. This study aimed to discuss the different methods used to collect soil and water samples from the field, as well as the standard methods used in soil and water testing laboratory. The physical properties of the soil are such as texture, structure, bulk and particle densities, porosity, etc. The chemical properties of the soil are such as soil reaction (pH), salinity (EC), cation exchange capacity, calcium carbonates, organic matter, etc. Water shares with soil many properties such as pH, EC, and soluble cations and anions. Fertility status of soil can be identified by analyzing macro and micro nutrients. These methods are very useful for workers in the field of soil and water analyzes, especially researchers and academics in fields related to the study.

**Keywords:** Soil analysis, water analysis, sampling, water quality, soil quality

### Introduction

Globally, there are many challenges regarding climate change and its' impacts of land degradation and food and water scarcity. Recently, and with increasing population and their needs of water and food, it was a necessity for finding fast and advanced solutions for dealing with these problems. Moreover, soil and water are considered as the main sources of life on the earth. Therefore, understanding of the nature of these resources is very important in order to knowing the possibility of their evaluation and management. This mission starts with selecting the area which needed to be studied for the objective of evaluating physical, chemical and biological characteristics. These characteristics describe mainly the soil and water qualities which located in a specific site under investigation, and also the fertility status as well as land capability for the agricultural production. Detecting the land use in a specific location is a very necessary for knowing the suitable approach which be followed for land evaluation. Different techniques are used for primary data collection in a specific location which needed to start the next stage. Among these techniques, the method of using recent remotely sensed imageries is commonly applied. Google Earth software is used for obtaining recent satellite data as imageries which used as a general guide for the new and un-surveyed areas. By using these images, visual interpretation of the study area which related to recognizing different land uses could be done. Furthermore, using software packages for remotely sensed imageries help in classifying the area to different classes of land use. These classes include uncultivated areas, desert land, newly reclaimed lands, cultivated areas, water bodies, urban areas, built-up areas, roads, etc. This type of classification is used for the purpose of knowing the next work stage which includes the field work and soil and water sampling. With the possibility of detecting the proposed locations of soil and water sampling, the next stage will be easier. After that, selecting the facilities and equipment for soil and water sampling could be known based on the nature of the site as well as soil types. The next stage is as field work and sampling, whereas a surveying visit is required for accurate and real primary data collection about the proposed sites. These data includes the nature of the area, cultivated crops, and method of cultivation, fertilization, and irrigation. Thereafter, collecting soil and water samples as well as corresponding information from the previous proposed sites will be possible. The stage of sampling includes soil profiles' digging or surface soil samples collection, which depends on the study objective. If the study aimed for land characterization, classification, or evaluation, soil profiles are necessary. Moreover, if the study aimed to soil surface or sub-surface diagnosis, only upper layers'

Sampling is applied. However, water samples are collected from resources for achieving crop irrigation purposes in the area as well as from the sources affected by salinization, pollution and degradation. Thus, soil profiles are drilled using suitable equipment and machines based on the nature of land and soil type. Soil samples are collected from each layer or horizon in the soil profile then put in packages or containers whereas all required data are written in attached labels. These data include geo-coordinates of sampling locations (recorded from GPS), cultivated crops' data, and method of cultivation, fertilization, and irrigation. The standard methods of soil profiles' description are followed depending on the methodology of United States of Geological Surveying (USGS). This description is very important for identification of soil genesis and classification, side by side with soil physical and chemical properties. After collecting samples, shifting to the soil testing laboratories is done. Soil preparation stage includes air drying, gridding, sieving and storing. Regarding water samples, the preparation process includes only filtration and purification from any suspended particles except if the study objective was to analyze these suspended particles. However, after this stage, samples will be ready for analysis against their physical and chemical properties. The soil physical properties include soil color, structure, texture, particle and bulk density, porosity, etc. Soil chemical properties include soil pH, total dissolved salts (EC), total content of calcium carbonates, organic matter, soil content of macro and micro nutrients in their different forms, soluble cations and anions, exchangeable cations, cation exchange capacity (CEC), etc. The required soil parameters to be analyzed are decided depending on the proposed objective of the study. Regarding water analysis, pH, total dissolved salts, soluble elements, as well as water content of pollutants, particles and microorganisms are needed. However, following the standard methods of soil and water analysis is mandatory either using conventional and mechanical methods or laboratory instruments. Depending on what previously described of the importance of soil and water analysis and with a brief about field surveying and sampling as well as laboratory analysis, this study aimed to explain and distinguish the most common methods of analysis used in soil and water laboratories.

### Importance of surveying and testing

For agricultural purposes, soil and water samples are collected and analyzed for their characteristics. These properties such as mentioned in soil health card which issued by many different countries as a national project, whereas these characteristics are available N, P, K (Macro-nutrients); available S (Secondary- nutrient); available Zn, Fe, Cu, Mn, B (Microelements); and pH, ECe, and SOC. Based on soil testing, these analyses provide an advisory for fertilizers and amendments application. The soil sampling and testing is the most important stage in any land evaluation project. Many agencies were involved in soil sampling and testing such as agricultural departments in universities and institutes and also soil testing laboratories (NMSA, 2014) [29].

### Soil testing

To assess soil fertility and health attributes, soil testing is required to measure the physical, chemical, and biological health of the soil. Soil testing provides very useful

information about the changing productivity and fertility level of the soils from time to time. Soil testing enables farmers to assess the suitability of land for agriculture, identify and quantify the constraining factors of crop-productivity. Soil testing also provides recommendations for fertilization and amendments for improving soil health (Goyal, 2016) [18]. Unfortunately, the conventional method of soil sampling and laboratory analysis is limited in the spatial domain which also is expensive especially for a large number of soil samples. It is also time-consuming, laborious, requires a lot of preparation stages, and also requires large quantities of chemicals to determine the soil properties which can be harmful to the environment. Hence, the routine laboratory analysis of soil samples is restricted only for a limited number of soil samples (Zhu *et al.*, 2018) [47], which is the most reliable method for soil analysis (Dammate *et al.*, 2015) [15].

### The need for soil data

The importance of soil depends on being the fundamental natural resource for food, fiber and energy production. Soil is supporting and regulating the life on the planet. Soil can be described as a regulator of water movement, environmental filter of metals and nutrients, and a potential sink for carbon to mitigate the global warming. Soil structure, composition, and physico-chemical and biological characteristics are the main factors affect the soil functions but those factors are temporally and spatially variable either vertically or horizontally (Nocita *et al.*, 2015) [3]. Naturally, the soil is a heterogeneous system which consists of organic and inorganic materials in a complex matrix hardly to be fully comprehend and understood (Rossel, 2007) [3]. To match the human needs and the limited land resources, larger amount of spatial soil data needed as a step forward to the precision agriculture (Ji *et al.*, 2016) [2]. Qualitative and quantitative soil properties estimation is the major challenge for environmental monitoring (Schulz *et al.*, 2006) [39], whereas soil nutrients assessment is important for precision agriculture (Sarathjith *et al.*, 2016) [38]. Soil attributes identification is also useful for the productivity enhancement under the growing effects of climate change (Hicks *et al.*, 2015) [20]. There is an increasing demand from the soil science community for accurate regional, continental, and worldwide soil information and databases in agricultural and environmental fields of science. For that purpose, soil survey is the main source of information to assess the soil suitability (Sunita, 2016) [41]. There is a need for much surveying in regional, farm scale to achieve appropriate management of the soil (Hicks *et al.*, 2015) [20]. It is very costly and time consuming to collect and analyse a large number of soil samples for a specific purpose. Soil health for the continental scale depends on creating large, accurate, and cheap data-base (Garfagnoli *et al.*, 2013) [17]. For the same goal, to describe and analyse the soil, complex approaches should be followed (Nocita *et al.*, 2015) [3].

### Soil Test Crop Response (STCR)

The STCR approach is one of the most scientific approaches of nutrient application for crops by using the soil test values and targeted yield equations by considering the contribution of nutrients from the soil, manures, and fertilizers. It gives a real balance between applied nutrients and the available nutrients already present in the soil (Dey and Das, 2014) [12]. Quantitative fertilizer requirements based on this approach

are more quantitative, precise, and meaningful because combined use of soil and plant analysis (Mandal *et al.*, 2019) <sup>[26]</sup>.

### Soil health and fertilizer recommendation (SHFR)

Healthy soil is fit for purpose, easy to perform well, friable, holds water and nutrients and has good drainage capacity. It allows for healthy root growth and good crop establishment. Soil health cannot be determined by just measuring crop yield, water quality, or any other outcome. Soil chemical, physical and biological properties, should be taken into account to describe a function or perform the capacity of the soil. Thus, soil capacity can be measured from functional relationships obtained by surveying, observations, sampling, and remote sensing application (Moebius-Clune *et al.*, 2016) <sup>[27]</sup>. The decision on fertilizer use requires knowledge of the expected crop yield and response to the nutrient application. It is a function of crop nutrient needs, the supply of nutrients from indigenous sources, and the short-term & long-term fate of the applied fertilizer nutrients. Therefore, fertilizer recommendations based on soil fertility status is the first step towards precision farming (Dobermann *et al.*, 2003) <sup>[1]</sup>.

### Soil and water sampling

There are several techniques of soil sampling such as random, grid, and zone sampling. Random sampling whereas performed on uniform fields where a global positioning system is used to keep a record of soil and nutrients data. Grid sampling as unbiased technique evaluates the distribution of samples throughout the field whereas soil samples are collected from equal distances in a field. Zone sampling technique is used to understand soil fertility in which land is divided into different zones based on soil properties and crop characteristics.

Water sampling is a way of collecting water from different water levels for testing or analysis. The aim of sampling is to evaluate the water quality standards as well as describes water contamination level and other biological properties. The most common techniques of water sampling are random, systematic, haphazard, stratified and judgmental sampling. In random sampling method, the water body is divided into grids horizontally and vertically depending upon the depth then samples are selected randomly. Systematic sampling method divides water body into grids whereas samples are collected from points with an equal distance. Regarding haphazard sampling, it is preferable for a homogenous system which random sampling points are selected. In stratified random sampling, water body is divided into different strata according to former and later use of the area, nature of contaminants, etc., and then strata are subjected to further sampling techniques. Judgmental Sampling involves the prior knowledge of the water body and field observation where samples are collected from various points according to the probability of contaminants distribution. To collect irrigation water samples, clean bottles should be used to prevent accidental contamination during sampling. Usually, water samples are taken annually and semi-annually based on stability of water source (Acharya, (2018) <sup>[1]</sup>; Provin and Pitt (2002)) <sup>[33]</sup>.

### Soil and water samples' preparation

#### Preparation of soil samples for analysis

Wet soil sample should not be stored without drying as changes may occur in the chemical nature of certain ions

and organic matter. Samples are air dried at the laboratory temperature and relative humidity then stored. Fresh samples are used for some analysis such as for their content of ammonium and nitrate N, exchangeable K, acid extractable P and ferrous iron. However, results of soil analysis are expressed on oven dry weight basis. Therefore, the moisture percentage is determined by drying a small sample in an oven at 105 °C for 2 hours. Grinding process is done in a rubber pestle in an agate mortar, or by a grinder. The next process is sieving whereas soil samples are crushed then pass through a 2 mm sieve (about 10 mesh per inch) and discarding the rest. When the gravels in the soil exceeds 2% limit over a 2 mm sieve, their exact percentage should be recorded. Mixing process of soil sample should be done by rolling procedure whereas sample is placed on a piece of cloth then divide it to four quarters then divide the quarter to four quarters then choose any two or three divisions randomly and mix them to make a final sample. Final process of sample preparation is storing in a sample box (polythene bag as in inner lining) and a label with required data should be stored with sample (Acharya, 2018; Provin and Pitt (2002) <sup>[1, 33]</sup>.

### Water samples' preparation

There are some precautions to be followed when preparing a water sample starting from shifting samples to the laboratory. Careless handling of a sample can produce highly misleading results.

To achieve accurate results, secure container should be used which prevent contamination of the sample. Therefore, resistant glass bottles or polyethylene containers are recommended. While water sampling from a tap or metal lines and valves, allow sufficient flow of water to wash out the system. After collecting sample, write all required data on a label that pasted in the container. If needed, store the sample in the refrigerator before taking it to the lab. A water sample needs to be shifted to the lab within a couple of days from collection. It is preferable to measure pH and EC immediately after sample collection. Sometimes, diluted nitric acid treatment is required for preventing contamination and microorganisms' activity for a short time (APEC, 2022) <sup>[2]</sup>.

### Soil physical analysis

#### Importance of the physical properties of the soil

Soil physical properties are very important for agricultural production and the sustainable use of soil. Soil air (oxygen) and water control nutrient absorption by growing plants depend on roots' ability to absorb soil solution and also soil supply to the roots. However, low hydraulic conductivity, affect negatively on the agricultural yield because of poor supply of water and oxygen to the roots (Candel *et al.*, 2018) <sup>[10]</sup>.

### Soil color

Soil color is the most property reflects the pedogenic environment and history of the soil. By soil organic matter and iron oxides, color can be identified, whereas organic matter darkens soil, and iron oxides produce a range of soil colors depending on oxidation state of iron. The common used method to determine soil color is a Munsell soil color charts. In this system, color is expressed in terms of hue (basic color), value (lightness or darkness), and chroma (intensity of basic hue). Color determinations are applied to

air-dried bulk soil samples.

### Soil texture (particle size distribution)

Particle size distribution is strongly related to other soil characteristics such as cation exchange capacity (CEC), water and nutrients' holding capacity, as well as organic matter content. To determine soil texture, three percentage values of soil particles (Sand (2.0-0.05 mm), Silt (0.05-0.002 mm), and clay (<0.002 mm)) content must be estimated. There are two commonly used methods for determining soil particles' distribution (hydrometer method and international pipette method). In both methods of soil texture analysis, soil samples are treated with 30% H<sub>2</sub>O<sub>2</sub> for removal of organic materials and HCl acid for removal of calcium carbonates then using sodium hexametaphosphate as a dispersing agent. Regarding hydrometer method (Bouyoucos, 1962) <sup>[9]</sup>, two reads of hydrometer and thermometer of soil suspension are recorded after 40 seconds and 2 hours of stirring. A correction factor is used to correct the read of hydrometer using temperature values. In an international pipette method (Richards, 1965) <sup>[36]</sup>, two times of pipette suction are done then dried in an oven to calculate the percentage of three soil fractions.

### Soil structure

Soil structure is controlling factor of modulating the flow and retention of water, solutes, gases, and biota in agricultural lands. Soil structure is important for land productivity and crop yield, and also regulates water infiltration, gaseous exchanges, soil organic matter and nutrient movements, root penetration, and susceptibility to erosion (Walia and Dick 2018) <sup>[43]</sup>. In soil structure, primary mineral particles form small clusters or (small aggregates) which form larger clusters (larger aggregates). However, organic matter acts as a cement that can help the formation of aggregates as well as soil organic carbon has an effect on aggregation in coarse-textured soils. Therefore, soil structure plays a vital role transportation of water, gases, and solutes in soil environment (Rabot *et al.*, 2017) <sup>[48]</sup>.

### Porosity

It is a main indicator of soil structural quality and it is essential for assessing the impact of adding organic matter to a soil system. Low soil porosity results from the loss of larger pores and the increase of finer pores (Barbera *et al.*, 2013) <sup>[5]</sup>. To determine soil porosity, pore size distribution must be characterized. The soil porosity also describes the situation of water and air movement as well as regulates the energy of these movements (Nimmo, 2005) <sup>[2]</sup>.

### Bulk density

Bulk density (BD) determination does not need a specific expertise or expensive equipment. It depends on undisturbed soil sampling by soil cores (Arshad *et al.*, 1996) <sup>[3]</sup>. Bulk density is a ratio of the dry mass of solids to soil volume and expressed in grams per cubic cm (g/cm<sup>3</sup>). The BD ranges from about 1.0 to 1.5 g/cm<sup>3</sup> in fine textured soils, and from 1.3 to 1.7 g/cm<sup>3</sup> in sandy soils. The BD of organic soils is less than mineral soils. Bulk density and total pore space are readily altered by tillage operations. Bulk density as soil structure controls many processes in soil and it is an important indicator of soil quality, productivity, and compaction (Hu *et al.*, 2012) <sup>[21]</sup>.

### Particle density

Particle density (PD) is a ratio of soil mass and soil particles' volume (without soil pores' volume). To determine soil particles' volume, displaced water volume can be measured using a cylinder or a graded container. The PD is controlled by soil minerals, and soil organic matter. It varies from 2.60 to 2.75 g/cm<sup>3</sup> in fine mineral textured soils, while organic soils have a lower PD. The most common methods for determining PD is a graduated cylinder and the Pycnometer methods (Candel *et al.*, 2018) <sup>[1]</sup>.

### Hydraulic conductivity

It is directly related to the structure and vertically and horizontally water movement. In saturated condition, water movement is horizontal while vertical direction of the movement occurs in conditions of non-saturation (Gülser *et al.*, 2017) <sup>[50]</sup>.

### Water holding capacity

It is the ability of a soil to store the water to be available for plants. Water holding capacity is controlled by many factors such as rain, temperature, soil organic matter, texture, and structure. In rain-fed arid and semiarid agricultural regions, soil-water holding plays a vital role in productivity of crops (Blevins and Frye 1993) <sup>[4]</sup>. Infiltration, evaporation and soil roughness affect water storage whereas tillage process in soil surface modifies soil characteristics in order to porous space (shape and volume).

### Saturated Paste

This kind of soil treatment is a very important to assess soil salinity which controls plant growth whereas soluble cations (Ca, Mg, K, and Na) and anions (SO<sub>4</sub>, CO<sub>3</sub>, HCO<sub>3</sub> and Cl) can be extracted by this method. Boron (B) is also extracted to measure the toxicity. Some important indices such as Sodium Adsorption Ratio (SAR), as well as Exchangeable Sodium Percentage (ESP) can be estimated.

### Saturation Percentage

The added water volume to saturate the soil is used to calculate the saturation percentage (SP) whereas dividing the total amount of water added (mL) by the oven-dry weight of the soil (g) and multiplying by 100 (Black, 1965) <sup>[36]</sup>.

### Soil and water chemical analysis

#### Soil Reaction (Soil pH)

Soil pH is a negative log of the hydrogen ion activity whereas H-ion concentration in solution increases ten times when its pH is lowered by one unit. The normal soil pH range varies from 3 to 9. Soils are classified based on their pH values into strongly acid (pH < 5.0), moderately to slightly acid (5.0-6.5), neutral (6.5-7.5), moderately alkaline (7.5-8.5), and strongly alkaline (> 8.5). Soil pH controls availability of soil nutrients and solubility of toxic elements. At high pH, availability of P, and micronutrients, except B and Mo decrease (Sharma *et al.*, 2015) <sup>[40]</sup>. Acid soils are found in temperate and tropical areas where rainfall is substantial. Soils are generally alkaline (pH above 7.0) in semi-arid, dry land areas as a result of the presence of CaCO<sub>3</sub> (Calcareous soils, pH values of 8.0 – 8.5), and soils with excess Na have values over 8.5 (sodic soils). The soil pH is determined in 1:2 soil: water suspension by the potentiometric method using a glass electrode (Jackson,

1973) [22]. Regarding water pH, it is positively correlated with electrical conductance and total alkalinity. For determination of water pH, direct measurement of water samples is done using pH meter following the same methodology of measuring soil pH.

### Electrical Conductivity (EC)

Soil salinity is a concentration of total soluble salts in the soil. To measure soil salinity, 1:1 or 1:5 soil: water ratio, w/v or soil paste extract is used. However, 1:1 soil: solution ratio or wider are more commonly used whereas the quantity of soil is limited. This test is rapid and salinity is measured by electrical conductivity (EC) meter (Richards, 1965) [36]. Total dissolved solids (TDS) can be calculated from EC values. Soil salinity as an important measurement affects crops in all stages of growing depending on the sensitivity of grown plants. However, values of 0 to 2 dS/m are safe for all crops; yields of very sensitive crops are affected between 2 to 4 dS/m; many crops are affected between 4 and 8 dS/m; while only tolerant crops grow reasonably well above that level. Water salinity also can be measured using the same methodology (EC meter).

### Total Calcium Carbonate

There are several methods for determining calcium carbonates in soil samples. Among these methods, using calcimeter to identify the carbonates' amounts is a common. Given weight of soil is reacted with an excess of acid whereas, carbon dioxide gas is released and shift the colored solution by a volume in equal to released gas. This value as well as laboratory temperature is used to calculate the carbon dioxide mass which used then to calculate total carbonates in soil. Other method for determining carbonates is a back-titration method. It depends on that hydrochloric acid is able to dissolve sample's carbonates then the remaining acid is back-titrated against sodium hydroxide (Sharma *et al.*, 2015) [40].

### Soil organic matter

Soil organic matter (SOM) represents roots, plant material, and soil organisms in different decomposition stages. It has influence on soil aggregation, nutrient reserve and its availability, moisture retention, and biological activity. Soil Organic Carbon (SOC) ranges from dominant in peat soils under humid-cold regions to absent in some desert soils. In cultivated regions, soils may have 3-4% SOM, while semi-arid soils' SOM are less than 1.5%. Losses after ignition (weight change) or wet chromic acid digestion are widely used methods of determining SOM. However, Walkley & Black method is the most common procedure involves reduction of potassium dichromate by organic carbon compounds and subsequent determination of the unreduced dichromate by oxidation-reduction titration with ferrous ammonium sulfate as described by Jackson (1973) [22]. After that, data are converted to SOM percentage using a constant factor (OM contains 58% organic carbon).

### Cation exchange capacity

Soils have negative charges which attract cations (potassium, sodium, calcium, magnesium, ammonium, etc.). Exchangeable cations can be leached or uptake by crops. Cation exchange capacity (CEC) is affected by soil pH, clay content, and organic materials. Several methods are used for determining CEC (Rhoades, 1982) [35]. Saturation with

(NH<sub>4</sub>) cation is the first stage of the procedure, then removal by washing of excess cation, and subsequent replacement of the adsorbed (NH<sub>4</sub>) by another cation such as (Na) and then measuring sodium using flame photometer (Richards, 1965) [3]. Cation exchange capacity is expressed as (cmol (+)/kg) and ranged between 1.0 and 100 cmol (+)/kg, least for sandy soils and most for clay soils. Similarly, higher CEC values reflect the dominance of 2:1 clay minerals, and lower values reflect the presence of 1:1 clay minerals.

### Gypsum

Precipitation with acetone is a commonly used method for determining soil contents of gypsum (CaSO<sub>4</sub>.2H<sub>2</sub>O) as described by Richards (1965) [36]. Method was modified and procedure is found in the FAO bulletin on gypsiferous soils (FAO, 1990) [16].

### Soluble carbonates and bicarbonates

Carbonates are indicated when pH is about 8.3 whereas It can be measured by titration with HCl using phenolphthalein indicator. Carbonates convert into bicarbonates in pH below 8.3 which can be titrated using HCl acid and methyl orange indicator when pH touches 4.0 (Jackson, 1973) [22].

### Soluble sulphates

It is measured turbid-metrically by spectrophotometer (420nm) whereas barium chloride is used for producing turbidity (barium sulphates) and mixture of Gum acacia and calcium chloride is used to prevent the settling of turbidity (Williams and Steinbergs 1959) [45].

### Soluble calcium

It can be measured using complex-metric titration with EDTA solution using murexide powder indicator and a fixed volume of 4N Sodium Hydroxide (Jackson, 1973) [22].

### Soluble magnesium

Same titration is followed with EDTA solution in the presence of eriochrome black T indicator and buffer solution (made from Ammonium Chloride and Ammonium Hydroxide) as described by Jackson (1973) [22].

### Soluble sodium

For determining sodium, flame photometer is used whereas known concentrations of sodium ion (1 to 100 mg/liter) are used for calibration. The samples having higher concentration of sodium are suitably diluted with distilled water and the dilution factor is applied to the observed values (Jackson, 1973) [22].

### Soluble potassium

It is also measured by flame photometer whereas 1 mg to 5 mg/ltr concentrations of potassium is used to generate a calibration curve of measurement. Extracts with higher concentrations are suitably diluted with distilled water and the dilution factor is applied to the observed values (Jackson, 1973) [22].

### Soluble chloride

To determine chloride in samples, titration is used of a known volume of sample with standardized silver nitrate solution using potassium chromate solution indicator. The formation of red color is the end point and the consumed

volume of silver nitrates' solution is used for calculating chloride concentration (Jackson, 1973) <sup>[22]</sup>.

### Soil fertility parameters

#### Nitrogen

The Kjeldahle procedure of total nitrogen determination includes digestion and distillation. The soil is digested in concentrated sulfuric acid with a catalyst mixture. The distillate is collected in saturated boric acid, and then titrated with dilute sulfuric acid. This method determines ammonium-N, most of the organic-N forms, and a variable fraction of nitrate-N in soil. Available nitrogen is determined by the alkaline potassium permanganate method as described by Subbaiah and Asija (1965) <sup>[41]</sup> using Kjeldahl flasks.

#### Phosphorus

It is measured using spectrophotometer whereas yellow colour is developed from the action of phosphor-molybdates complex formation under strong acidic conditions. The intensity of colour is directly proportional to the concentration of phosphorus in the sample (Black, 1965) <sup>[8]</sup>. Phospho-complexes are reduced by weak reducing agents such as ascorbic acid or tartaric acid (potassium antimony tartrate).

The soil extraction by sodium bicarbonate as described in a procedure of Olsen *et al.* (1954) <sup>[49]</sup> and What an able and Olsen (1965) <sup>[44]</sup>. It is a common method of determining available phosphorus. It is suitable for alkaline and calcareous soils.

#### Total Phosphorus

The procedure is includes soil samples' digestion with a strong acid to dissolve all insoluble inorganic and organic P forms of minerals then determination colorimetrically using spectrophotometer (Olsen and Sommers, 1982) <sup>[3]</sup>. Recent techniques of measuring total phosphorus may be used whereas ICP is a valid tool of measurement.

#### Potassium

Soils contain large amounts of total potassium (1 to 2%) as in minerals' matrix. A very small fraction (about 1%) is presented as available to plants (soluble and exchangeable). The soluble potassium can be extracted using distilled water then measured by flame photometer. Available form of potassium can be extracted using neutral ammonium acetate method then also measured using flame photometer (Jackson, 1973) <sup>[22]</sup>. The exchangeable form is gotten from subtraction. For fertilizers' recommendations, extractable-K values of less than 100 to 150 ppm are considered as deficient in potassium.

#### Sodium

Soluble sodium fraction can be measured by flame photometer in soil water extract (saturated paste preferable). Available sodium is determined in the same extraction of available potassium whereas neutral ammonium acetate solution is used for extraction. Exchangeable sodium is normally the dominant portion of total extractable Na. It can be detected by difference between available and soluble forms (Jackson, 1973) <sup>[22]</sup>.

#### Calcium and magnesium

They are usually determined in a neutral normal ammonium

acetate extract of soil as available form. Soluble Ca and Mg are obtained by extracting the soil by water or from a saturated paste. Measurement of these forms is done by titration with EDTA or by atomic absorption spectrophotometer after the removal of ammonium acetate and organic matter (Sharma *et al.*, 2015) <sup>[40]</sup>.

#### Boron

Boron (B) is a non-metal element that found in small amounts in igneous, sedimentary and metamorphic rocks. It is considered as an essential micronutrient for plant growth (needed for cell division and the growth of young shoots). The dominant form of boron in soils is borates and boron-silicate minerals. There are many effective methods for analyzing boron samples. The hot-water extraction procedure, introduced by Berger and Truog (1939) <sup>[6]</sup>, modified later is still the most popular method for measuring soil available boron in alkaline soils. Water soluble B, the available form of B, is extracted from the soil by water suspension. Boron in soil extracts is measured calorimetrically using reagent Azomethine-H (Bingham, 1982) <sup>[7]</sup>. Also, B can be analyzed by colorimetric methods using reagents such as Carmine, and most recently by Inductively Coupled Plasma (ICP) and Atomic Emission Spectrometry (AAS). The colorimetric method, using reagent Azomethine -H, is preferable because the use of AAS poses some limitations as B is not a metal. Where soil B levels are less than 0.5 ppm, deficiency is likely to occur for most crops. However, where levels are greater than about 5 ppm, toxicity may occur. The normal B concentration in plant tissue is reported to be between 20 and 100 µg/mg dry matter of mature leaves. It is essential in sugar translocation and in the synthesis of hormones and protein in plants.

#### Available micronutrients

Micronutrients are required in small quantities by grown plants. These nutrients are essential for crop growth. Solubility of micronutrient cations decreases with an increase in soil pH. Methodology of determining micronutrients includes extraction using suitable solution then measurement using ICP or AAS instruments. The Diethylene Triamine Pentaacetic Acid (DTPA) test of Lindsay and Norvell (1978) <sup>[25]</sup> is commonly used for evaluating fertility status with respect to micronutrient cations, i.e., Fe, Zn, Mn, and Cu. The DTPA method is an important and widely used chelating agent, which combines with free metal ions in the solution to form soluble complexes of elements (Estefan, 2013) <sup>[15]</sup>.

#### Heavy Metals

For extracting heavy metals from soil samples, aqua regia and tri-acid digestion methods are used to decompose soil particles. Heavy metals such as Cadmium, Lead, Mercury, Chromium, Selenium, Nickel as well as Cobalt, Molybdenum, and Arsenic can be measured in this resulted solution. After that, these elements are measured by AAS and ICP (Estefan, 2013) <sup>[15]</sup>.

#### Dissolved Oxygen

It is an important parameter whereas its correlation with water gives a direct / indirect influence of bacterial activity, photosynthesis, availability of nutrients, and stratification (Premlata Vikal, 2009) <sup>[32]</sup>. Increase of temperature and

microbial activity, dissolved oxygen decreased (Kataria, 1996) [24]. Winkler's method as titration procedure is used to measure DO in water samples after five days of incubation at 20 °C. The difference between initial and final DO gives the amount of the consumed oxygen by the bacteria. Chemical Oxygen Demand (COD) is expressed by unit of mg/ltr. It is the amount of dissolved oxygen consumed to the process of chemical oxidation of the organic materials in water. Biological Oxygen Demand (BOD) is the amount of dissolved oxygen that consumed in biochemical decomposition of organic compounds and the oxidation of inorganic materials (e.g., iron, sulfites).

**Table 1:** Rating limits of available soil nutrients (Yurembam *et al.*, 2015) [46]

Nutrient	Low	Medium	High
N (kg/ha)	<280	280-560	> 560
P (kg/ha)	<10	10-25	>25
K (kg/ha)	<108	108-280	>280
S(mg/kg)	<10	10-20	>20
Fe (mg/kg)	<4.8	4.8-8.0	> 8.0
Mn (mg/kg)	<2	2-4	>4
Zn (mg/kg)	<0.6	0.6-1.2	> 1.2
Cu (mg/kg)	<0.2	0.2-0.4	> 0.4

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