Study of soil Chemical analysis Methods

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Abstract
This Research Paper only provides a summary of some of the many methods of soil analysis, but these include more techniques in use. All methods must be fully validated and demonstrated to work across a range of matrices and monitored on a batch-by-batch basis using blanks and Analytical Quality Control (AQC) samples. In addition. According to the content of nutrients in the soil obtained by soil analysis and the requirements of a particular crop, it is easier to determine the amount of fertilizers needed to achieve high and high-quality yields. Quality soil analysis is the basis of fertilization planning (which fertilizers to use, how much and when to use) and thus the quality of the entire production cycle, which results in high quality and yield and better farm management decisions.
Keywords:- Soil analysis , Sampling depth , Previous applications of lime or fertilizers , Analytical methods , organic compounds , Inorganic compounds & Conclusion

I.   Introduction

Soil analysis is a set of different chemical processes that determine the amount of available plant nutrients in the soil, but also the chemical, physical and biological properties of the soil important for plant nutrition. Chemical analysis of the soil determines the content of plant nutrients; nitrogen (N), phosphorus (P_2O_5), potassium (K_2O), pH, humus content, total $CaCO_3$, available lime, organic matter, total sulfur (S), sodium, micronutrients and other physical properties (capacity, permeability, density, pH value).

Reasons for conducting a soil analysis
To determine the level of availability of nutrients or the need for their introduction.
Predict yield increases and fertilization profitability (poor soils do not always provide yield increases from fertilization due to possible limiting factors).
To provide a basis for calculating the required fertilization of each crop.
Evaluate the state of individual nutrients and at the same time determine how to manage nutrients.
Improve economic planning and decision-making for farmers.
when is the best time to do a soil analysis?
Taking soil samples for analysis is strongly recommended to be done after crop harvest and before any fertilization at optimum soil moisture. The soil must not be compacted, along the edge of the farm or where mineral fertilizers are unevenly dispersed, as the sample will not be representative of the entire field and the resulting data will not reflect the actual condition of the soil in the field. the whole field. In the case of permanent crops such as orchards and vineyards, the analysis is carried out regularly.

How to do sampling correctly
Samples are collected using an auger, but can also be collected using a shovel.
Important factors to consider when taking soil samples:
Sampling depth.
The depth of sampling is critical because tillage and soil nutrient mobility can affect nutrient levels in different soil zones. The depth of sampling depends on the crop, cultivation practices, tillage depth and nutrients to be analyzed.
Soil type.
In case one farm has different soil types, it is recommended to take different samples from different soil types separately.
Previous cropping.
The previous crop that occupied a particular field must be taken into account. If different crops were in different fields, separate samples should also be taken from these different fields and not a single sample from fields where different crops were grown.
Previous applications of lime or fertilizers.
In the case where different fertilization programs were used for different fields. The same should also be considered when taking soil samples for analysis by taking different soil samples separately for different fields. The slope or topography of the land.

Different soil samples should be taken if, for example, part of the field is muddy while the other part is on a gentle slope.

Soils are an extremely complex matrix to analyze, especially in contaminated sites – the actual soil matrix can vary from sand (silica) to limestone (calcium carbonate) to clay (composite minerals) or a mixture of many. Furthermore, the range of contaminants varies from completely harmless construction materials to toxic gas plant waste to highly toxic pharmaceutical waste/mercury/explosives etc.

Chemical analysis methods have to deal with this very wide range of materials and compounds, which is difficult, and the introduction of MCERTS (Environment Agency Monitoring Certification Scheme) has raised awareness of the challenges associated with reliable testing on such diverse matrices.

Preparation of soil samples

This is an absolutely critical step that is often overlooked in method reviews, but if the preparation is not done carefully, no amount of sophisticated equipment will improve the result.

Many tests cannot be performed on a dried and crushed sample because some of the parameters of interest will be lost, so soils must be initially mixed (and tested) in the wet state in which they were received, and these include:

Volatiles, leachate testing, phenols, ammonium nitrogen, sulfides, cyanides, most organics, hexavalent chromium.

Homogenization can be done using the classic cone and quarter technique, or using a jaw crusher to break up larger pieces or by kneading clay-type samples. Fibrous material may need to be cut or crushed. If the sub-sample can be dried, it is usually at 35-40 oC or 105 oC if moisture is required. Moisture content will be required for all tests performed on received soils as these must be adjusted back to dry weight for reporting purposes. For contaminated soils, it is not considered appropriate to remove anything from the sample as potentially hazardous material may cover the lumps (eg paint, electrolyte fluids) or form the whole lump (eg tar), but if any component is removed this must be noted in the final report to meet MCERTS requirements.

Once a sub-sample of soil is weighed, then in most methods some form of liquid is added to the soil to extract the desired parameter of interest. For example, anions and pH require a 2:1 water extraction, metals use acid decomposition, cyanides require alkaline extraction, phenols use a mixture of methanol and water, and most organics require solvent extraction. Samples must be shaken, refluxed, or digested for a period of time, then filtered or centrifuged, and the liquid extract then analyzed with an analyte-specific instrument.

"During method validation, robustness and applicability to a range of matrices must be established, and this is usually done using certified reference materials" During the validation of a method, the robustness and applicability over a range of matrices must be determined, and this is usually done by using Certified Reference Materials (CRMs) or spike and recovery (spiking a range of soils with a known amount of a standard and then testing to see how much is recovered). The validation must cover the preparative stages of a method, as well as the instrumental analysis.

Analytical methods
Calibration

All analytical instruments require calibration, and this is usually performed by preparing a range of standards (five or six is common) at increasing concentrations, putting these through the instrument, which will then construct a calibration curve or graph. Samples, when run through the instrument, are read off this graph to give the concentration of the analyte within the sample. Alternatively, internal standards of a known concentration are added to the sample, and the concentration of compounds within the sample are calculated according to their response measured against that of the internal standard – this is more common in organic methods.

Methods fall into two main groups: spectroscopic methods for most inorganic analytes, and chromatographic methods for organic compounds.

Inorganic compounds
Anions

One of the most basic (and traditional) methods is colourimetric spectroscopy. This has been used by chemists for hundreds of years, although instrumentation is much more automated nowadays. This chemistry lends itself well to anions (compounds which are negatively charged and therefore attracted to an anode), and examples are:

Chloride, nitrate, nitrite, sulphate, phosphate, sulphide, ammoniacal nitrogen.

Most laboratories will use some form of automated spectrophotometer, such as a Kone or an Aqua. They are multi element analysers, so more than one compound can be determined in one run. All instruments will have an autosampler, and a system for sequentially adding the appropriate chemicals to develop the colour.

The final solution then passes into the spectrophotometer part of the system. The general rule (the Beer-Lambert Law) is that: 'the absorbance of light at a given wavelength is directly proportional to the concentration of the absorbing species', or in simpler words, the more intense the colour, the greater the concentration. These automated analysers are very efficient and can run up to two hundred samples per hour.

Metals

Metals comprise one of the most commonly requested groups of analytes in environmental analysis, and these are usually measured by an Inductively Coupled Plasma Optical Emission Spectroscopy (ICP – OES). The dried and crushed soil is weighed out (usually 5 g of sample), and then digested on a hot block with aqua regia (a 3:1 mix of concentrated hydrochloric and nitric acids) for up to two hours. The acid extract is filtered and then loaded into the autosampler rack on the ICP – OES, where it will be aspirated and pumped into the plasma (a very hot, ionised gas at 10,000oC). Here, energy causes electron excitation, and then as the sample passes out of the plasma, this energy will be emitted as the electrons return to their ground state. The energy emitted will be at specific wavelengths, according to the metals present in the sample, and this energy is detected by the spectrophotometer.

The beauty of the ICP is that it will measure several wavelengths at once, so over 20 metals can be analysed in about 4 minutes, ensuring this is a very efficient method. ICPMS can be used instead of ICPOES, but the samples require large dilutions due to the sensitivity of the instrument to the high acidic and dissolved solids content in the digests. An alternative method of metals analysis is atomic absorption, and there are several forms of this, but the downside is that only one element can be measured at once.

Sulphur compounds

There are several forms of sulphur containing compounds, and these have differing levels of importance in environmental analysis
• Total sulphur can be measured using an induction furnace, but as sulphur can exist in many forms, it will not give much information regarding risks on site
• Elemental (or free) sulphur is analysed using a solvent extraction, followed by HPLC (an organic method described later in this article). This is present on many ex gasworks sites and can cause a dermatitis like skin reaction
• Total sulphides can be analysed by acid digestion and ICP, but many of these are insoluble and relatively inert, so do not constitute an environmental hazard
• Acid Soluble Sulphide is considered more of an environmental risk, as if these compounds are present on a site with acid rain or groundwater, then hydrogen sulphide can be formed, which is highly toxic at low levels. These are analysed either by ion selective electrode, or acid digestion followed by colourimetric analysis of the impinger solution (which traps the evolved hydrogen sulphide)
• Acid Soluble Sulphate, sometimes referred to as total sulphate (which may not be strictly equivalent), is analysed using an acid digestion followed by ICP-OES. A value of 0.24% is considered a potential risk for concrete attack
• Water Soluble Sulphate, from a 2:1 water extract, is analysed either by ICP-OES or colourimetric spectrophotometry. This will determine if sulphate resisting cement is required by the construction company

Nitrogenous compounds

The interrelationship of nitrogen compounds can be confusing, and these are defined as follows:
• Nitrate and nitrite (total oxidised nitrogen) – these will be analysed on a 2:1 water extract of the soil by colourimetric spectroscopy
• Ammoniacal nitrogen – this will include both ammonia (NH_3) and ammonium (NH_4), and again is either a water extract, or distilled as exchangeable ammonia
• Kjeldahl nitrogen is a measure of the ammoniacal nitrogen and organic nitrogen, and is analysed by a distillation and titration method
• Total nitrogen involves a stronger acid digestion, and includes all of the above
"ICP is a very efficient method it can measure several wavelengths at once, so over 20 metals can be analysed in about 4 minutes"
Cyanide compounds
• Free cyanide represents simple cyanide salts, such as potassium cyanide. These are very water soluble and extremely toxic, and are measured by a water extraction, followed by a specific, automated distillation/colorimetric analysis such as a Skalar
• Easily liberated cyanide is still measuring free cyanide, but uses a more acidic digestion to cope with difficult matrices

• Total cyanide requires a much more aggressive digestion, but this is also performed on the Skalar system. This will include the complex ferri and ferro cyanides
• Thiocyanate is a separate colourimetric analysis, and is not included in the total cyanide, but again can be performed on the Skalar
Organic methods
These are normally based on some form of chromatography, with common methods including:
• HPLC – High Performance (or High Pressure Liquid Chromatography)
• GCFID – Gas chromatography with flame ionization detection
• GCMS – Gas chromatography with mass spectroscopy detection
"chromatography is the separation of a complex mixture by partitioning between a stationary phase (column) and a mobile phase (liquid or gas)"
Chromatography is the separation of a complex mixture by partitioning between a stationary phase (column) and a mobile phase (liquid or gas). Separation occurs because of different properties of the compounds - either their mass (so larger molecules take longer to pass through) or their polarity (the charge associated with the compound - the larger it is, the more reactive the compound will be. the slower it will move through the column).
Water analysis

High performance liquid chromatography
Specialty phenols are analyzed by HPLC and provide results for phenol alone, xylenols, cresols, resorcinols and naphthols. Depending on the site, clients need to request only phenol, total phenols, or specialty phenols.
"Separation occurs because of the different properties of the compounds - either their mass or their polarity"
Gas chromatography with FID
This method is mostly used for petroleum hydrocarbons such as organic matter (GRO) and extractable petroleum hydrocarbons (EPH). The pattern of the chromatogram allows the analyst to identify the type of product in the soil.
Gas chromatography with mass spectroscopy detection
This method is used for a very wide range of compounds, some examples include:
Polyaromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs) and pesticides (chlorinated and phosphorylated and many others).
Mass spectrometry can be run in two ways, either in full scan, where the instrument is set to follow every peak, or in selected ion monitoring (SIM), where it will only look for specific target compounds and ignore anything else that may be present . If the client is unsure of which compounds to request, it is better to ask for a full VOC and/or SVOC plus TIC scan. Each of these will provide a target list of 60+ compounds plus up to ten tentatively identified compounds. If the likely compounds are known, then SIM-run methods are better to use, as these provide lower detection limits.

II.   Conclusion:-
        This overview only provides a summary of some of the many methods of soil analysis, but these include more techniques in use. All methods must be fully validated and demonstrated to work across a range of matrices and monitored on a batch-by-batch basis using blanks and Analytical Quality Control (AQC) samples. In addition, laboratories accredited to ISO 17025 (and MCERTS) must participate in proficiency testing programs where blank samples are tested at regular intervals to ensure that laboratory methods are fit for purpose. Environmental analysis today is complex, time-consuming and subject to strict quality procedures. More than 30% of the samples analyzed are QC or validation samples, which represents a high cost to the laboratory, but the data must be defensible. Laboratories are constantly looking to improve their methods and processes and further developments/legislation will provide the impetus to continue these improvements – this is an ever-changing industry.

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