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Influence of soil sample pretreatment on Portable X-ray fluorescence measurements

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ABSTRACT

Developing new technologies for field soil surveys has produced powerful new quantitative tools for in situ assessment of soil physicochemical properties. One such technology, portable X-ray fluorescence (PXRF) spectrometry, has shown great promise for assessing elemental concentrations in soils for a variety of applications. This study aims to determine the influence of four different methods (in-situ, dry, wet, and pre-treated) from samples collected in the SZIC campus forest Gödöllő in Hungary. Seven PXRF measurements on each genetic soil horizon of a soil profile within the study area were performed. Soil samples from the horizons were collected, vertically on soil profiles from bottom to top. A PXRF was used to scan soil samples in the laboratory while they were wet, dry, and pre-treated to assess 8 elements of magnesium (Mg), aluminum (Al), calcium (Ca), sulfur (S), manganese (Mn), iron (Fe), rubidium (Rb), and titanium (Ti). Traditional laboratory methods were used to prepare and treat the samples. Microsoft Excel 2007 was used to analyze the data. The results indicate that PXRF can be used to reliably determine (calcium (Ca), manganese (Mn), iron (Fe), rubidium (Rb), and titanium (Ti)) in all four states, showing no significant difference in the results of the measurements, while the determination of magnesium (Mg), aluminum (Al), and sulfur (S) proved to be less reliable. PXRF holds a lot of promise as a technique for assessing soil element concentrations in real time.

Keywords: PXRF, soil analysis, traditional laboratory methods, Hungary.

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Abbreviations and definitions

PXRF	Portable X-ray fluorescence
IUSS	International Union of Soil Science
WRB	World Reference Base
FAO	Food and Agriculture Organization
UNESCO	United Nations Educational, Scientific and Cultural Organization
RSGs	Reference Soil Groups
DSMorph	Digital soil morphometrics
GPS	Global Positioning System
HCL	Hydrochloric Acid
USDA	United States Department of Agriculture
MC	Moisture content
ER	Electrical resistivity
GPR	Ground penetrating radar
IR	Infrared spectroscopy
CEC	Cation exchange capacity
Mg	Magnesium element
U	Uranium element
Ag	Silver element
Rh	Rhodium
Ta	Tantalum
Au	Gold element
W	Tungsten element
kV	Kilovolt
K	Potassium element
YR	Yellow/ red
mm	millimeter
µm	micrometer
Al	Aluminum element
Ca	Calcium element
S	Sulfur element
Mn	Manganese element
Fe	Iron element

R	Rhenium element
Ti	Titanium element

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1 Introduction

Physiochemical laboratory analysis and traditional soil surveys are very important to increase our understanding of soil, but these techniques are expensive and time-consuming. Demand for soil data has been rising recently across a range of fields. Thus, more time- and money-efficient quantitative techniques for data collecting and soil analysis have been created (Zhu et al., 2011b). Portable X-ray fluorescence (PXRF) spectrometry is one of the many new methods that have proven effective in predicting soil qualities.

The use of portable X-ray fluorescence (PXRF) spectrometry in the field and laboratory has gained popularity with time. Common PXRF analysis targets are soils and Quaternary sediments. Several studies have shown that PXRF readings correlate strongly with information gathered by traditional techniques, including bench-top XRF (Hunt & Speakman, 2015). Numerous studies have demonstrated that PXRF equipment can produce reliable, precise, and repeatable data and that this data can be relevant to a broad range of environmental applications (Frahm & Doonan, 2013). Consequently, PXRF analyses are becoming more and more common among experts in the public sector as well as among ecologists, toxicologists, soil scientists, and geochemists (Horta et al., 2015).

Researchers are actively attempting, as with any new method, to determine its overall accuracy and to identify its main sources of error. Unfortunately, there is no universally accepted protocol for PXRF sample pretreatment for soil sample analyses. Sample pretreatment methods include air drying, homogenization, and fine gridding (Weindorf et al., 2016). This research will investigate the effects of these sample pretreatment methods on the results obtained, compared to when soil sample has not been pretreated in any way.

1.1 Justification

The laboratory measurement takes a lot of time and involves numerous sample preparations. This may lead to inaccurate results as the numerous preparation activities can lead to contamination of the samples. Human errors in the entire long preparatory activities can also lead to inaccurate results. Portable X-ray fluorescence (PXRF) spectrometry is a new technique for analyzing soil samples and it is more accurate, time and cost-efficient than laboratory measurement. This study aims to assess the element concentrations of four different states (in-situ, dry, wet, and post-treatment) from samples collected.

1.2 Objectives

The specific objective of this study is to:

- Determine the impact of various soil sample treatments on the results of measurements using portable XRF (PXRF).

The broad objectives are:

- To compare different measurement techniques in situ, wet, dry, and post-treatment by PXRF.
- To determine which measurement is a more accurate technique to determine certain elements using PXRF.

1.3 Research questions

- Does the temperature (in situ) at which measurement is done affect the results?
- Does grinding as a soil sample pre-treatment technique affect the results of the measurements?
- Does the duration of time between field sample collection to laboratory measurement time affect the result?
- Does the soil sample's moisture content affect the measuring results?

2 Literature review

2.1 Soil science

The 1800s saw the development of soil science as a field of study. The field has created a robust language that was required to describe and characterize the soil composition and its numerous properties. The evolution of several soil science subdisciplines has been impacted by semantics, or the science of meanings (Hartemink, 2016). Soil science comprises various fields, such as soil physics, soil chemistry, soil biology, soil survey, and its main branch, pedology (Rahman, 2018).

In different parts of the world, the term "pedology" has varied definitions, but the most common one is "the study of soil in the field, its classification, distribution, and formation," which includes a wide variety of observations, laboratory analyses, and inferences (Hartemink & Minasny, 2014). The Greek words 'Pedon' and 'logy' mean ground and study, respectively (Singer, 2005). Most international soil science collaborations have been driving soil classification standardization (Van Baren et al., 2000). For a long duration, pedology worldwide has standardized recording, sampling, and description (Clarke, 1936).

The soil profile is the central concern of pedology (Kellogg, 1974). A soil profile is a vertical section showing different soil layers from the surface to the unaffected parent material. The historical record of all soil formation processes provided by a study of the soil profile is important because it serves as the basic for pedagogical investigations. The soil profile is the basis for both the practical utility of soils and the key to soil classification (Matthews, 2014).

Soil classification is established to organize our knowledge about soils (Arnold, 2005). An overview of different soil characteristics is provided by soil names and classes (Kubiëna, 1953). In contrast to other fields (such as Phyto- and zoo taxonomy, and mineralogy), soil science has a variety of classification systems that coexist on a worldwide scale (Krasilnikov et al., 2010) and their number is even increasing. soil classification is interesting to collect soil that has a similar range of properties (chemical, physical, and biological) into units that can be mapped and geo-referenced (FAO, 2023). It is important to comprehend the classification system being used because there are numerous classification systems in use to fully appreciate the diversity of soils that exist on the earth. These systems offer data on their physical and chemical characteristics, which can be used to comprehend their possible specific applications (Padmanabhan & Reich, 2022). There are national soil classification systems in many countries, but there are two commonly used and applied throughout the world Soil Taxonomy (Soil Survey Staff, 1999) and the World Reference Base, IUSS Working Group WRB (2015).

The World Reference Base (WRB) is an international soil classification system that evolved from the FAO–UNESCO Legend of the Soil Map of the World (Mantel et al., 2023). the main purpose of the World Reference Base for Soil Resources is to add scientific depth and background to the 1988 FAO Revised Legend by including the most recent findings about the world's soil resources and how they interact with one another (Nachtergaele et al., 2000). The WRB includes two hierarchical levels, the first level has 32 Reference Soil Groups (RSGs), while the second level has an unknown number, by combining the RSG with a set of principal and supplementary qualifiers the soils are named (Mantel et al., 2023)(Nachtergaele et al., 2000). The WRB is consistently evaluated and revised, with the most recent version being presented in Glasgow in 2022 at the 22nd World Congress of Soil Science (Mantel et al., 2023).

Soil Taxonomy is a natural classification system developed for mapping and classifying soils in the United States with the help of international soil experts to assist in organizing knowledge about soils through the National Cooperative Soil Survey (Ditzler & Hempel, 2017), this system is open structure to allow for modifies the classification system and accommodates new soil attributes and soil types (Padmanabhan & Reich, 2022). it includes a six-level hierarchical classification system: orders (the top category), suborders, great groups, subgroups, families, and series (the bottom category) (Learner, 2023). Most nations in the world use Soil Taxonomy as their primary system for classifying soils, and many more use it in conjunction with other systems and in technical reports. Additionally, for more than 50 years, Soil Taxonomy has been accepting alternative soil classification systems by acting as a standard forum for communication and technological exchange. To establish and apply soil taxonomy for soil survey, mapping, and interpretation, soil scientists from all around the world have made contributions. All the soil that exists on Earth is considered by soil taxonomy, making it quite simple to identify links between soil and landscape. This makes it possible to use the information gathered to develop appropriate decision support systems that lead to the promotion of good practices to preserve and improve soil quality.

2.2 Soil profile

Soil is both profiles and landscapes. A large trench (hole) dug between two and six meters vertically down into the earth will show different soil horizon layers. Viewing these layers from above provides a cross-sectional perspective of the earth's surface as well as the types of rocks and soils that form the soil profile (Balasubramanian, 2017) . A soil profile is a vertical cross-section of the soil layers at a particular location (Figure 2-1). A typical soil profile forms over a period of 1,000–100,000 years. The parent rock type, vegetation, terrain, and climate all have a significant impact on how the soil profile is formed (Hartemink et al., 2020).

Figure 2-1: Soil profile

(ISRIC, 2023)

2.3 Soil Horizon

The layers of a soil profile known as soil horizons are what identify different types of soil. Color, texture, roots, structure, fragmented rock, and any other distinctive feature that is significant are the basis for defining a horizon (Hartemink et al., 2020). The layers are categorized into three categories: bed rock, subsoil, and topsoil. Most of soils include of five main horizons. They are referred to as O, A, B, C, E, and R (Balasubramanian, 2017).

2.3.1 Master Soil Horizons

Letter and number combinations are used to name soil horizons. Soil profiles may contain any one of six common types of horizons. The capital letters O, A, E, B, C, and R appear in their names. We refer to these as master horizons. Each master horizon is depicted in (Figure 2-2) according to its relative location within a soil profile. Although most soils have three or four master horizons, here all six are displayed.

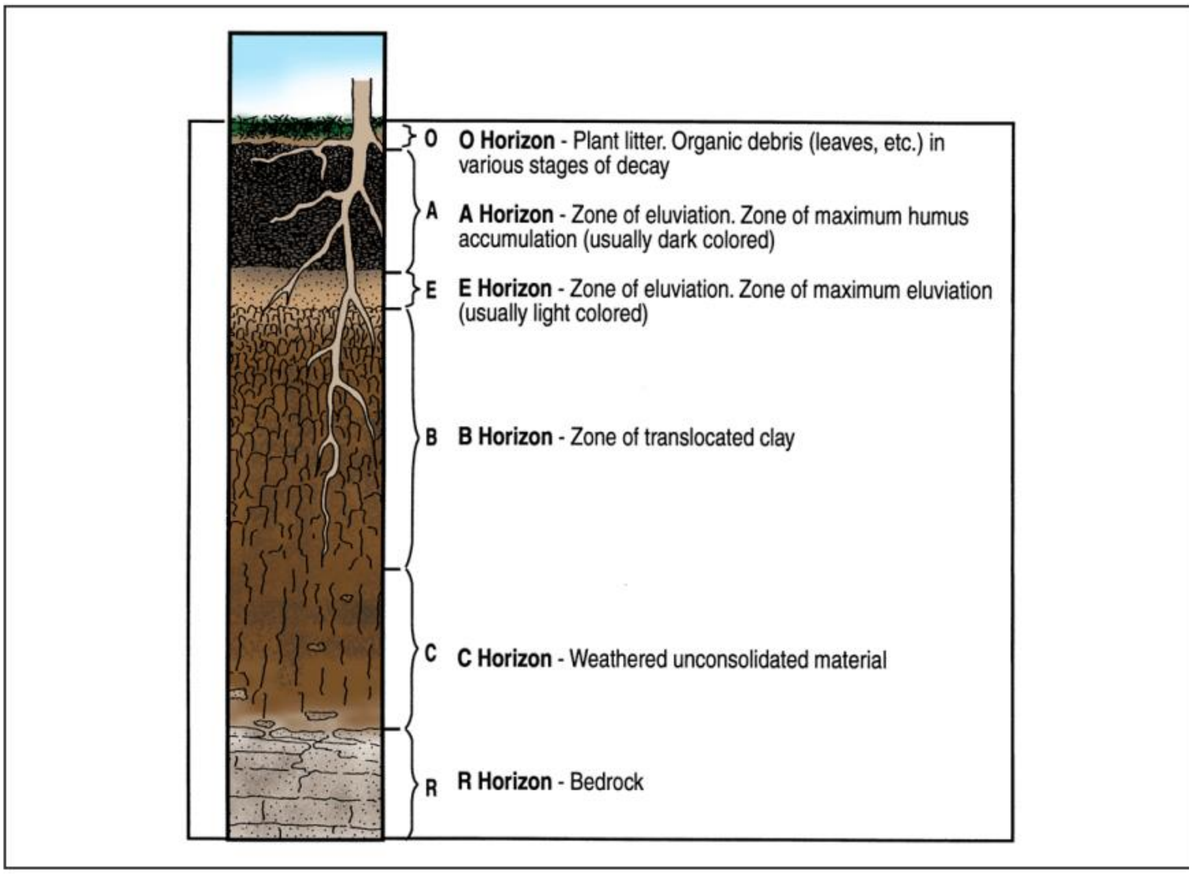

Figure 2-2: Master Horizons

(Hartemink et al., 2020)

2.3.2 Transitional Horizons

Rarely do master horizons change dramatically from one to the next. Rather, changes occur gradually across a zone that could be as thick as five to ten inches. Transitional horizons are the name given to these areas. In Missouri soils, there are three common ones: AB, BA, and BC. See (Figure 2-3).

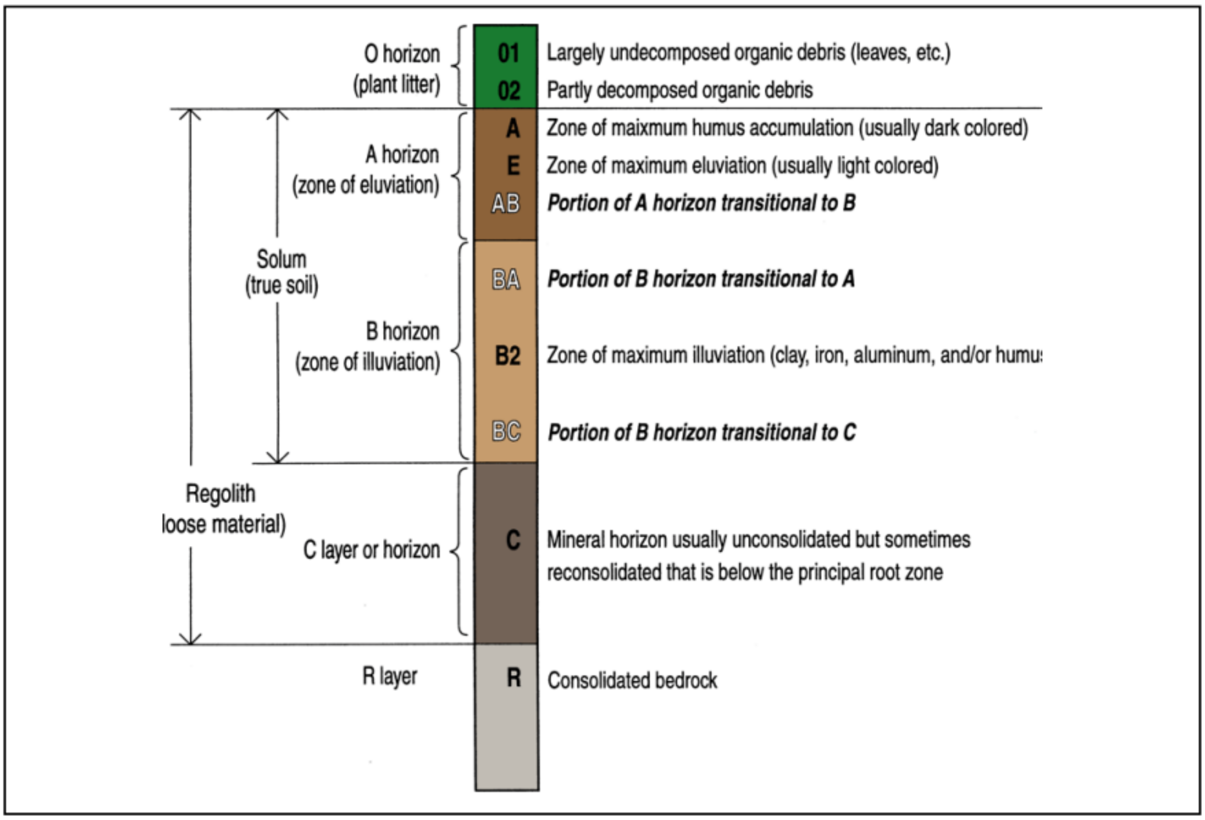

Figure 2-3: Transitional Horizons

(Hartemink et al., 2020)

2.4 Sampling

Generally, sampling can be defined as the process of selecting a subset of individuals from the total group upon which measurements will be made (Rochette & Bertrand, 2007). The measurements made on this sample will then be used to estimate the properties of the total population. In soil science, soil sampling means selecting soil samples from the field for analysis. Although it is nearly impossible to transfer the entire soil body into the lab, soil samples are the most practical technique for doing chemical analyses.

Choosing the most effective technique for selecting the samples that will be used to estimate the population's attributes is known as sampling design (Walworth, 2006). The sample population is made up of the sampled elements, which are chosen from the population according to the sampling design. As will be discussed below, there are various designs for soil sampling.

2.4.1 Simple Random Sampling

This is the simplest of all the methods in use. Sample selection is completely left up to chance without considering the variance in a soil population. Every sampling unit is given an equal, independent chance of getting drawn using this procedure. It is frequently an effective strategy in a highly homogeneous field (richard oliver (dalam Zeithml., 2021).

2.4.2 Systematic Sampling

Sampling is done in a systematic manner. For example, samples may be taken at 5 m intervals or only at the foot slopes and tops of hills. A third option is to sample the soil beneath every other tree in an orchard. This type of sampling can also be combined with simple random sampling, in which sampling sites are chosen at random from a soil map, and samples are drawn only from sites with even or odd numbers. Because the samples are distributed more evenly across the population, systematic sampling can often provide more accurate results than simple random sampling. However, if the soil population contains periodic variation and the interval between successive sampling sites coincides with the interval of the soil variations, biased samples may be obtained. As a result, before deciding to use this method, it is best to research the nature and occurrence of soil variability (richard oliver (dalam Zeithml., 2021).

2.4.3 Composite sampling

This is the most common and cost-effective sampling method, in which sub-samples are collected from randomly selected locations in a field and then composed for analysis. The composite sampling analytical results provide average values for the sampled area. The actual number of sub-samples depends on the size and uniformity of the field. In general, a larger or less uniform field should be sampled more intensively than a small and uniform field. A sampled area should have at least 5 sub-samples taken, and 15 to 25 are preferable (Walworth, 2006).

2.4.4 Soil Profile Sampling

Soil profile sampling is necessary for purposes such as physicochemical and mineralogical analysis of importance in soil genesis, morphology, and classification, or for chemical analysis needed for soil testing and studies in soil fertility. Plant roots penetrate different horizons, and each horizon provides a different chemical environment for the growing roots. To reduce time and expense in the analysis, the root zone in soils is commonly sampled. Frequently, this sample is composed of the A and part of the B horizon. Although this procedure violates the requirement of homogeneity, a root zone can be defined as one unit for the objective. In the study of soil genesis, morphology, and classification, soil types are usually selected to represent the soil volume. The use of such representative samples simplifies sampling procedures, analysis, and interpretation of results (Group, 2003).

2.5 Soil pit observations — digital morphometric

For a wide range of uses, including mapping, classification, land assessment, and pedological research, detailed soil observations are undertaken. In addition to the standard method of digging a soil pit, other methods of making observations include trenches, road cuttings, samplers, slice shovels, augers, and push probes. Reference and geographic location, the profile environment (climate, geology, etc.), a description of the site and region, and an explanation of the characteristics and qualities of the soil horizons should all be included in a description of a soil profile. The main objective of describing a soil profile is to preserve the image of the soil.

Augers, pickaxes, spades, knives, spatulas, rock hammers, Munsell charts, notepads, water bottles, HCl, sample bags, tape measures, clinometers, compass, altimeters or GPS, and cameras are some of the standard field tools for describing soil profiles. These are used to sample soil for laboratory chemical and physical examination as well as to monitor and observe soil qualities and features. Soil orders are created by combining observed and measured soil qualities and horizons into classes.

Aerial photographs were initially used to remotely sense surface soil properties. Since the 1980s, spaceborne or aerial approaches have been used to assess surface soil properties, such as texture, iron, moisture, organic carbon, salinity, and carbonate content. Subsurface soil qualities can be deduced from such data, but most information on subsurface soil properties will need to come from either (i) measuring or sampling from a soil profile or (ii) utilizing ground penetrating equipment.

Soil characteristics, properties, and horizons use to describe and define the Reference Soil Groups and soil units of the WRB which determine soils and their interactions when they are combined (Nachtergaele et al., 2000). With populations that originated from places with less summer precipitation, both climatic and soil features were significant; soils with higher water-holding capacities tended to have lower water requirements for emergence (Smith et al., 2000).

Soil characteristics are single parameters that can be measured or seen in the field, in a lab, or by employing microscopic analysis methods such as, Soil horizons, Soil texture, Soil color, Soil structure, Soil moisture, and Carbonates, and Consistence (rupture resistance) (Hartemink & Minasny, 2014). All the primary characteristics that are measured and seen in a soil pit: horizons, texture, color, structure, moisture, carbonates, and consistency, are reviewed and described in this section.

2.5.1 Soil horizons

Soil horizons are distinguished layers of soil profile. From top to bottom, they are split into these layers, called "Master Horizons" layers: O Horizon, A Horizon, AB horizon, B Horizon, C Horizon, and R Horizon (Thompson et al., 2012). The letters and numbers used for the horizon identification transmit more information than just the location in the soil profile since they are interpretive symbols based on morphology and soil genesis (Bridges, 1993). V.V. Dokuchaev initiated the designation of soil horizons, and C.F. Marbut was one of the first to suggest utilizing horizons for classifying and distinguishing soils (Bockheim et al., 2005). The horizons of a soil reflect its processes and provide details about its past and present state (Hartemink et al., 2020). In addition, soil horizon plays important role to identify the various processes that contribute to soil development and to understand the various soil types. The classification of soil is also based on it (Sciencefacts, 2023).

2.5.2 Soil texture

Soil texture is one of the most crucial aspects of the soil (USDA, 2010). It refers to the percentage of soil's mineral fraction that is made up of sand, silt, and clay-sized particles (Figure 2-4) The largest particles are sand, which has a diameter of 2.0 to 0.05 mm. smaller silt particles range in size from 0.05 to 0.002 mm. The smallest clay particles are 0.002 millimeters (Mobilian & Craft, 2022). It is an extremely settled characteristic that influences soil biophysical properties. the soil texture is correlated with other soil properties such, soil quality and fertility in a long time, bulk density, permeability, and the soil porosity, which controls the water-holding capacity, gaseous diffusion, and water movement that determine the health of the soil, is also related to the soil texture (Upadhyay & Raghubanshi, 2020). For example, compared to soils with higher silt and clay concentration, sand-dominated soils have high permeability and poor water holding capacity (Mobilian & Craft, 2022).

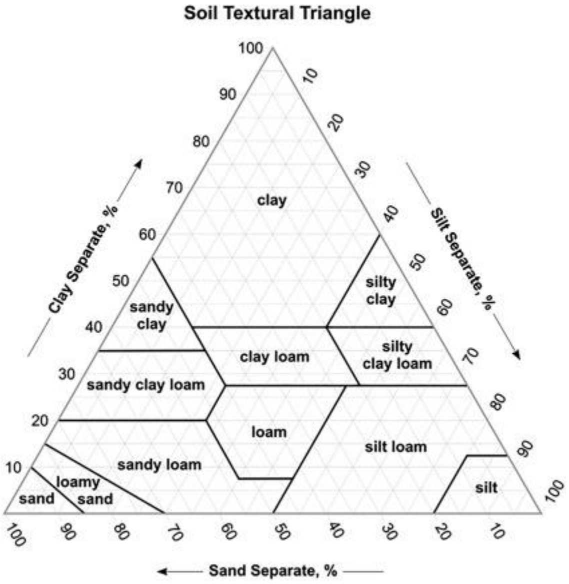

Figure 2-4*: Soil Textural Triangle*

(Moreno-Maroto & Alonso-Azcárate, 2022)

2.5.3 Soil color

It has been shown that the characteristic of soil that most represents its pedogenic environment and past is its color. More organic matter and iron than manganese determine the color of the soil. Higher organic matter content soils are darker and less reflective than lower organic matter content soils. (Morris et al., 2005), Manganese oxides (black), iron oxides (yellow, brown, orange, and red), and other oxides; alternatively, it can be because of the parent rock's color; consequently, native soil fertility has long been related to soil color (Figure 2-5). comparing with other methods Munsell soil color charts is the most used method to determined soil color (Rowe, 2005).

The Munsell color system uses the notations for hue (basic color), value (lightness or darkness), and chroma (intensity of basic hue) to record the color of the soil matrix of each horizon (Amerling et al., 2006). A symbol located in the upper right corner of each card in the soil color chart indicates that all of the colors on that particular card have the same hue. The colors gradually lighten vertically in visually equal stages as their value rises. They grow greyer to the left and have more chroma to the right when viewed horizontally. The chart shows the value and chroma of each color right next to the color. Value is represented by the first integer, and chroma by the second. The colors are organized in the chart to create three scales: (1) radial, or varying in hue from card to card; (2) vertical, representing value; and (3) horizontal, representing chroma (Owens & Rutledge, 2005).

Figure 2-5: Soil color

(Quanta Magazine, 2023)

2.5.4 Soil structure

The physical configuration of the soil's pores, particles, and organic matter is known as its soil structure. It is both shaped by and has an impact on numerous soil processes. For this reason, soil structural characteristics serve as critical indicators of soil functions (Fig 2-6) (Schlüter & Koestel, 2022).

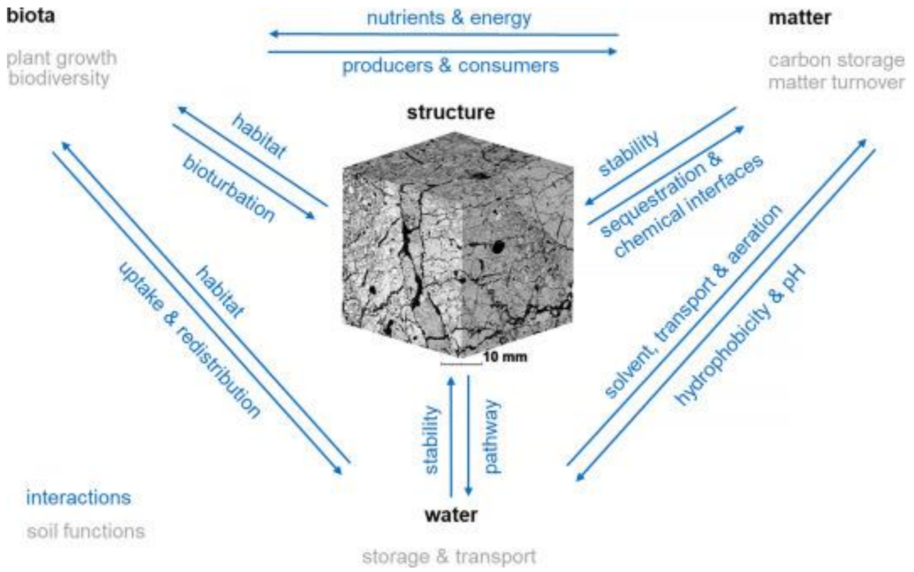

Figure 2-6: Soil structure

(Schlüter & Koestel, 2023)

The amount of nutrients that are available for uptake by roots, as well as the lateral and down-profile growth of roots, are significantly influenced by the structure of the soil. Nutrient intake is influenced by the position of the roots and the characteristics of the local microenvironment. Many methods have been employed to describe the characteristics of the soil and the roots, As an illustration, consider (1) X-ray-computed micro-tomography to identify the characteristics of soil aggregates and mesopores in the 27–67 µm range, for characterizing macro- and microporosity down to 19 µm pore resolution at interfaces of texture-contrast soils and for 3D imaging of roots in columns of undisturbed soil; (2) X-ray absorption combined with phase-contrast imaging; (3) detailed 2D X-ray imaging to describe the soil structure and root characteristics in intact soil cores, which gave a satisfactory match with simulation models; and (4) observations of the electrical capacitance of roots of plants cultivated in fields. Because it can photograph particles in the nanometer size range with sub micrometer spatial resolution and can be combined with high spectrum resolution for Spectro microscopy studies, X-ray microscopy is increasingly the method of choice (Foecke et al., 2022).

2.5.5 Soil moisture

The term "water content" refers to the moisture content of the soil, which indicates how much water is present in the top 1-2 meters of the soil. (“Soil Moisture,” 1977). By means, the relationship between the amount of water in a section and the quantity of solids in the soil sample, given as a proportion, for example, is the moisture content in soils (percentage %) (Smith et al., 2000). Since it is the primary source of water for both natural vegetation and agriculture, it is of greatest significance (“Soil Moisture,” 1977). Water must therefore be handled carefully. The highest method of water withdrawal in agriculture is irrigation. Plants should only be watered when necessary, because of this. Because of this, monitoring soil moisture is crucial (Nwogwu et al., 2018).

In general, a soil's capacity to retain water is influenced by several variables, including organic matter content, soil type, depth, compaction level, salt, pollutant presence, porosity, temperature, and humidity. This division is based on the proportions of clay, silt, and sand. The largest is sand, followed by silt, and clay is the smallest. based on the soil texture triangle, as shown in (Figure 2-4). The soil types listed below were determined by the US Department of Agriculture (USDA). The texture and amount of organic matter in the soil determine how much water it can hold. The soil may hold water based on the particle size's surface area. The surface area increases with decreasing particle size. Soil may store more water since it has a bigger surface area. The amount of organic matter in the soil has an impact on how much water it can store. The ability to store water increases with an increase in organic matter and vice versa (Nwogwu et al., 2018). There are many techniques to measure soil moisture; (i) Classical techniques and (ii) Modern techniques.

2.5.5.1 Classical Soil Moisture Measurement Techniques:

Farmers have traditionally utilized these methods to determine the soil moisture content (MC). These consist of (i) the Feel approach (ii) and the Appearance method. In the past, when technology was less advanced, farmers would measure the amount of water in the soil with their hands. They attempted to estimate the water content from the physical characteristics of the soil. These techniques were imprecise approximations.

2.5.5.2 Modern Soil Moisture Measurement Techniques

New technologies are developed along with new sensors and meters as technology advances. These techniques are quite effective and yield reliable results. There are three methods that are often used nowadays to determine the moisture content of soil. These are (i) In situ or point measurements; (ii) Soil-water models, and (iii) remote sensing methods.

2.5.6 Carbonates

Calcium carbonates in soils can result from the residues of the limestone parent rock material or can be pedogenic ally derived as in soils of arid and semi-arid areas. Carbonates influence soil pH, nutrient availability (particular micro-nutrients), and the flocculation of particles. Pedogenic carbonates are particularly important as they are closely associated to the carbon cycle and can be used in assessment of carbon stocks. Determination of carbonates concentration is achieved by dripping 10% HCl on the soil matrix. The observed degree of effervescence is then recorded in the levels of none, very slight or violent. PXRF device can also be used to determine presence of carbonates in the soil from the field, as an in situ method (Hartemink & Minasny, 2014).

2.5.7 Consistence (rupture resistance):

Rapture resistance, also called consistency or fliability depends on soil moisture content and is determined manually in the field. It is described as loose and soft to extremely hard and rigid. Rapture resistance refers to the degree of cohesion and adhesion of the soil material. It influences several soil functions and has implications for engineering applications and soil tillage. Like soil structure, rupture resistance is influenced by various individual soil properties, and not many pedological studies have attempted to assess it in other ways than the traditional field method, the drop-shatter test.

2.6 Digital soil morphometrics

The advancements in proximal soil sensing have significantly increased the measurement of soil properties in the field. A variety of modalities, including proximal or remote, in situ and ex-situ (field and laboratory), non-invasive or intrusive, mobile, or stationary, have been used to characterize proximal soil sensing. Proximal soil sensing in digital soil morphometrics is limited to stationary in situ and ex-situ observations of soil form and characteristics with depth (Bellon-Maurel & McBratney, 2011). DSMorph is generally understood to be the "application of tools and techniques for measuring, mapping, and quantifying soil profile attributes and deriving continuous depth functions (Hartemink & Minasny, 2014)." Thus, DSMorph is at the very forefront of technology and innovation in soil science and promises to provide field soil description, a subject that has been relatively technology stable, if not technology averse, for decades, a much-needed scientific and technological revamp. There are countless pedological, edaphic, and environmental uses for DSMorph tools and approaches (Zhang et al., 2021).

Most soil profile characteristics, such as soil color, soil structure, and coarse fragments, may be determined quickly and quantitatively utilizing proximal sensing techniques (Bellon-Maurel & McBratney, 2011). The number of soil characteristics that may be measured in the field is expanding, and this range is influenced by technology as well as environmental factors like light and soil moisture as well as the heterogeneity of the soil under investigation. Rapid evaluation and interpretation of observations, as well as the absence of sample collection and lab-related expenses, are benefits of in situ soil property assessments. Cluster analysis of proximally sensed data, such as that obtained from portable X-ray fluorescence spectroscopy, visible-near-infrared sensors, and digital images, can be used to carry out more objective and quantitative horizon delineation procedures (Zhang et al., 2021). To cover soil profile variance and minimize sample effort, sophisticated profile sampling systems have been devised. New quantitative and objective measurements of soil profile features and their variability are provided by digital soil morphometrics.

Various sensors and instruments have been evaluated to quantify distinct characteristics. It is more difficult to quantify compound characteristics (horizons, structure) than single characteristics (soil moisture and clay content). Soil horizons, soil texture, soil color, and soil moisture are the main characteristics of soil that have been evaluated in the field using digital morphometrics. There have been no attempts to measure soil structure in the field using non-traditional methods. Using the cone penetrometer, electrical resistivity (ER), or ground penetrating radar (GPR), several studies concentrated on soil horizons. None of these methods require a soil pit: GPR and ER are non-invasive methods, while the penetrometer is an invasive method that can be used to identify specific soil horizons. Redoximorphic features are characterized by their uneven and spotty nature, which makes them ideal for digital morphometric assessment using vis-NIR. Infrared spectroscopy (IR) may have the greatest potential of all the sensors. The use of infrared sensors in a soil pit is limited by signal interference from non-soil sources, the uneven and variable nature of the soil surface, and sample preparation issues (Ben-Dor et al., 2009). Since soil moisture can also be detected by the vis-NIR sensor, algorithms have been developed to extract the moisture from the spectra (Ben-Dor et al., 2009).

2.7 Portable X-ray fluorescence spectrometer:

One of the most vital resources for humanity is soil. The soil's chemical and granulometric properties determine the dynamics of water, climate, life, forests, carbon, and other elements. They are essential to the production of food in addition to their significance to the ecosystem. suggesting that soil health is essential or there will probably be environmental issues in several places. The determination of soil's analytical value is implied by the research of its constituents, such as carbon, nutrients, and clay. because evaluation and monitoring of the chemical and physical characteristics of soil depend on soil analysis (Demattê et al., 2019).

Particularly on contaminated sites, soils present an exceptionally difficult matrix to analyze. The actual composition of the soil might range from sand (silica) to clay (complex minerals) to limestone (calcium carbonate), or a combination of many. Furthermore, the variety of contaminants is diverse, ranging from relatively safe building materials to highly poisonous pharmaceutical waste, mercury, explosives, and gasworks waste (AWE Magazine, 2023).

One of the techniques used in laboratories the most is traditional laboratory analysis. These methods typically have a lot of drawbacks, though. Analyses take a long time—between three and fifteen days—to produce results, which is insufficient given the urgency of the situation. Traditional laboratory analysis has been around for a while, but it involves a variety of chemical substances. The use of these chemicals substances consumes a lot of money, as the annual expenditure for traditional analysis can reach millions of dollars annually. We have to emphasize that this world problem. The most affected are developed nations with well-established agricultural systems, particularly those in development (Latin America and Africa) with ample area for agricultural growth. In this regard, it took numerous writers years of research to recognize proximal sensing as a potentially useful method that can bring light on the problem (Demattê et al., 2019). To gather soil information, modern instruments now include sensors that are smaller, faster, more accurate, more intelligent. Take portable x-ray fluorescence (PXRF) as an example.

The quantification of a portion of the inorganic elemental component is done using the portable X-ray fluorescence (PXRF) technology. It is applicable to a variety of industries, such as agricultural, environmental, metalworking, plastics, textiles, and archeology, for both qualitative and quantitative study (Weindorf et al., 2014). On the other hand, because (PXRF) is quick, incredibly affordable, portable, and non-destructive, its application has grown (Jha et al., 2021). There are certain distinct benefits that portable X-ray fluorescence analysis has that no other analytical method can match. These benefits come from the technique's multi-element capacity as well as its non-destructive nature, but most significantly, it comes from the operator's instant access to information about a sample's chemical makeup in the field. As a result, the operator has access to information in real-time that, in comparison to traditional sampling and laboratory analysis, can help solve the problem for which analytical results are needed much more quickly (Haschke, 2014).

One of the few analytical methods that can perform in-situ analysis and give a field operator access to the chemical composition of a sample in a short amount of time (30–120 s) is portable x-ray fluorescence, which carried over the benefits of XRF (Corbeil, 2009)s. The non-homogeneity of efficiency for various elements is the main cause for concern with PXRF performance (Migliori et al., 2011) (VanCott et al., 1999). When we say, "in situ," we imply that the analytical tool is brought to the sample and brought into contact with it. An analytical measurement is performed, and the operator can access the result right away. The idea of "interactive sampling and analysis" was born because the operator might utilize this data to determine what to analyze next. Consequently, PXRF falls under a unique class of "hand held" instrumentation (Haschke, 2014).

The use of portable X-ray fluorescence (PXRF) spectrometry for pH determination (Sharma et al., 2014), Cation exchange capacity (CEC) (Sharma et al., 2015), The potential of integrated field spectroscopy and spatial analysis for enhanced assessment of soil contamination (Horta et al., 2015), Water analysis (Pearson et al., 2017), Analysis of metal-laden water (Pearson et al., 2018), Elemental assessment of vegetation (McGladdery et al., 2018), Quantification of trace arsenic in soils (Parsons et al., 2013), Applications for applied geochemistry (Lemière, 2018), Assess the soil elemental composition (Borges et al., 2020).

2.7.1 PXRF Instrumentation

Due in large part to developments in semiconductor detector technology and downsizing, portable XRF is one of the instrumental methods that has grown quickly in recent years. PXRF is comprised of an excitation source, a detector, a sample positioning facility, and a pulse processing and analysis facility, just like conventional XRF apparatus. On the other hand, mobility necessitates that an ergonomic instrument minimizes mass and power consumption in the selection and integration of these components.

Elements from Mg to U in the periodic table can be identified and measured using portable X-ray fluorescence spectrometry. Since the primary X-rays incident on the sample, any incident X-ray photon that has energy greater than the binding energy of the core-shell electron will be absorbed by an atom. A core-shell/subshell vacancy (i.e., an excited state of the atom) is left behind when incident photons are absorbed because the core-shell/subshell electron, also referred to as a photoelectron, is emitted. Characteristic or X-ray fluorescence (XRF) and Auger electron emissions are the two competitive atomic de-excitation processes that produce XRF emissions following absorption. It is implied by both processes that an electron moves from an upper subshell to a lower one within the atom. Since the Auger electron and XRF photons both have distinct energies that characterize the electron transition in the given atom, they form the basis of spectroscopic methods for atom identification. However, because of the high attenuation of electrons in matter, XRF spectroscopy methods are more practical than Auger electron spectroscopic methods. By observing the de-excitation transitions, which are specific to each element, XRF can be used to identify the elements present in the sample. The primary X-ray radiation in contemporary portable instruments is produced in a vacuum-tight tube that houses a cathode (Figure 2-7). Miniaturization was made possible by the field-emission X-ray tubes' small coin-size and far simpler design than the large Coolidge-type tubes used in industrial or diagnostic X-ray imaging applications. The electrons are accelerated toward a metal target anode (such as Ag, Rh, Ta, Au, W, and others) by a high voltage of 20–60 kV. X-rays corresponding to the distinctive K and L line fluorescence of the target atoms as well as a lower intensity continuum are produced when the electrons collide with the metal target.

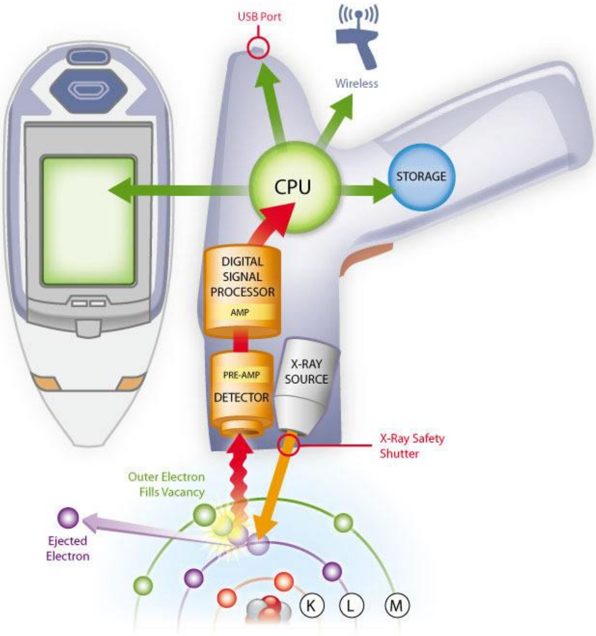

Figure 2-7 Functional schematic of a portable PXRF instrument

(López-Núñez, 2022)

3 Materials and methods

3.1 Profile description Gödöllő university forest

Figure 3-1 (Profile description Gödöllő university forest, 2023)

0 – 8 cm (O horizon): undecomposed or partially decomposed organic material

8 – 23 (A horizon): 10YR 3/2 moist color, Sandy loam texture, weak subangular blocky structure, no carbonates, pH = 4-5

23 – 48 (AB horizon): 10YR 4/3 moist color, Sandy loam texture, weak subangular blocky structure, no carbonates, Ph = 4-5

48 – 76 cm (Bt horizon): 10YR 4/6 moist color, Sandy clay loam texture, weak prismatic structure, no carbonates, pH = 5

76 – 87 cm (BC horizon): 10YR 4/6 moist color, sandy loam texture, single grain/structureless, pH = 6

87 – 115 cm (Ck horizon): 10YR 6/4 moist color, loamy sand texture, single grain/structureless, extreme carbonate reaction, pH = 8

Table 1: Lab data, data source: data archives of the Department of Soil Science

Master horizons	**Depth** (cm)	**pH** H2O	**OM** (%)	**CaCO3** (%)	**% Sand** 2-0.05 mm	**% Clay** <0.002 mm
O	0-8		12.00	nd	nd	nd
A	8-23	4.9	2,48	0	72.9	9.7
AB	23-48	4.8	0,60	0	75.5	14.9
Bt	48-76	5.3	0.68	0	68.2	26.8
BC	76-87	5.6	0.65	0	70.3	19.8
Ck	87- 115	8.6	<0.5	32	85.1	3.8

3.2 Field methods

Samples were collected from soil profiles pit in Gödöllő university forest, in December 2023 at -2℃ temperature. The profiles were characterized based on WRB/FAO classification system. The horizons were O, A, AB, Bt, BC, and Ck (Figure 3-1). Samples were collected by hand, vertically on soil profiles from bottom to top and scanned with PXRF in situ. They were further used for laboratory and PXRF characterization. The PXRF measurements were performed by using the Olympus Vanta portable X-ray fluorescence spectrometer.

3.3 Laboratory methods

In the laboratory, samples were scanned with PXRF in three conditions:

1. As received from the field (sample wet).
2. Air dry sample dry.
3. Grounded sample (sample pre-treatment) to pass a 2 mm sieve, grinding of the samples was facilitated using a mortar and pest.

4 Data analysis

Descriptive statistical parameters including mean, standard deviation, median, minimum, maximum, and range for each variable were obtained with the data analysis add-in of Microsoft Excel 2007. Boxplot, which shows the concentration of each element in the various horizons was also drawn.

5 Result and discussion

5.1 Magnesium element

The results of the pre-treated samples of magnesium are displayed in (Figure 5-1). The O horizon (0-23 cm), A horizon (8-23 cm), AB horizon (23-48 cm), Bt horizon (48-76 cm) do not have any recorded values of magnesium, but the BC horizon (76-87 cm) and Ck horizon (87-115 cm) have. The results of the wet samples of magnesium are displayed in (Figure 5-2), where values for magnesium are found in the O horizon (0-23 cm), A horizon (8-23 cm), AB horizon (23-48 cm), Bt horizon (48-76 cm), and BC horizon (76-87 cm) but not in the Ck horizon (87-115 cm). The results of the dry samples of magnesium are displayed in (Figure 5-3), where values for magnesium are found in the O horizon (0-23 cm), AB horizon (23-48 cm), Bt horizon (48-76 cm), BC horizon (76-87 cm) and Ck horizon (87-115 cm) but not in the A horizon (8-23 cm). The results of the Mg post-treatment samples are displayed in the results of the Mg post-treatment samples are displayed in (Figure 5-4). This is different from the earlier instances where the Mg levels were recorded in all horizons O horizon (0-23 cm), A horizon (8-23 cm), AB horizon (23-48 cm), Bt horizon (48-76 cm), BC horizon (76-87 cm), and Ck horizon (87-115 cm).

These results show that PXRF is not able to detect magnesium; it has limitations in terms of light major element identification, particularly for magnesium (Interdonato et al., 2022). In the absence of a vacuum or He atmosphere, which cannot be produced in the field, Mg is very poorly determined by PXRF (Bloise & Miriello, 2022).

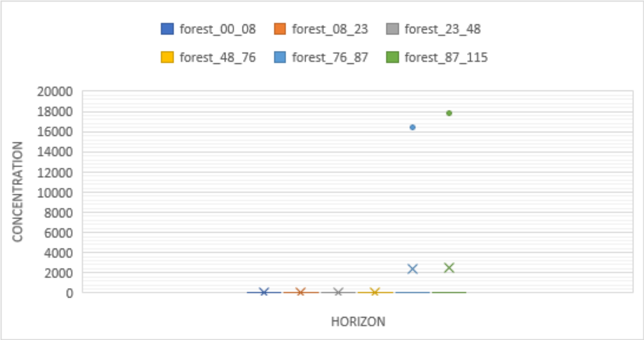

Figure 5-1: Magnesium (ppm): pre-treatment

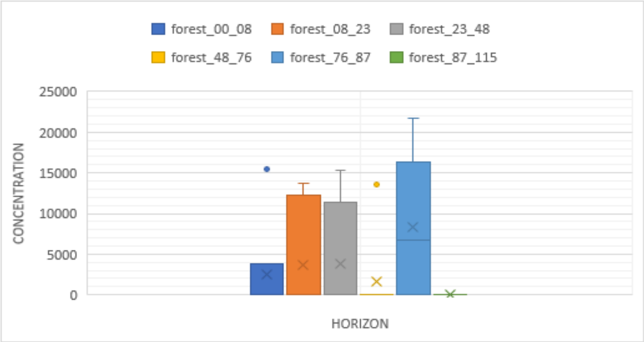

Figure 5-2: Magnesium (ppm): wet

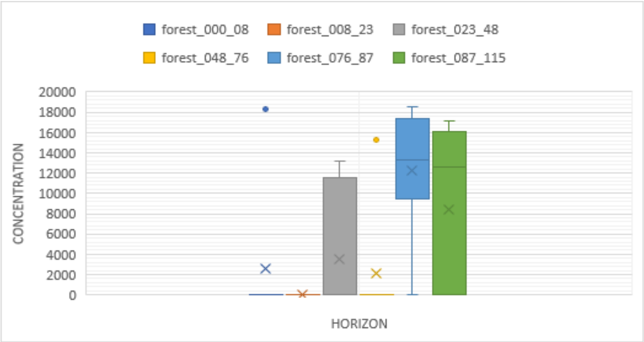

Figure 5-3: Magnesium (ppm): dry

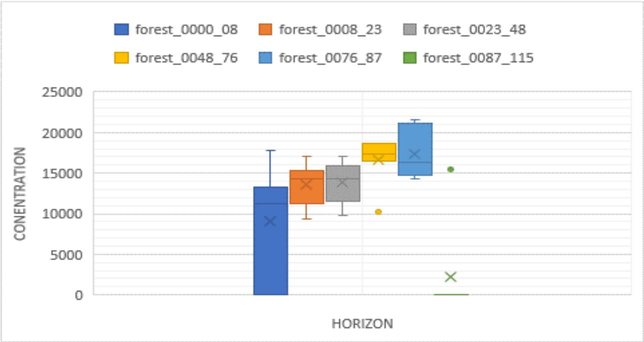

Figure 5-4: Magnesium (ppm): post-treatment

(Magnesium element analysis, own work)

5.2 Aluminum element

As we have seen, the PXRF can measure the aluminum content of each status; however, the aluminum concentration is different between different statuses. The results of the pre-treated samples of Al are displayed in (Figure 5-5). The concentrations in the O horizon (0-23 cm), AB horizon (23-48 cm), Bt horizon (48-76 cm), and BC horizon (76-87 cm) are nearly identical with other statuses, while the concentrations in the A horizon (8-23 cm) and the Ck horizon (87-115 cm) are different with other statuses. The results of the wet samples of Al are displayed in (Figure 5-6), where the concentration is nearly constant throughout all horizons except for the AB horizon (23-48 cm). The results of the dry samples of Al are displayed in (Figure 5-7), where the concentration is nearly constant throughout all horizons apart from the O horizon (0-23 cm). The results of the post-treatment samples of Al are displayed in (Figure 5-8), where the concentrations in the O horizon (0-23 cm), AB horizon (23-48 cm), and BC horizon (76-87 cm) are similar, while they are almost similar in the O horizon (0-23 cm) and Bt horizon (48-76 cm). The amount of aluminum in the Ck horizon (87-115 cm) has noticeably increased.

These results indicate that PXRF is not able to measure aluminum and that it has limitations when it comes to measuring Al elements. According to soil science, there is a loss of aluminum in AB horizon (23-48 cm), which could not be seen in the earlier figures. The primary characteristic of the AB horizon, which is a mineral horizon, is the eluvial loss of silicon, iron, aluminum, and/or silicate clay, or some combination of these (Hartemink et al., 2020).

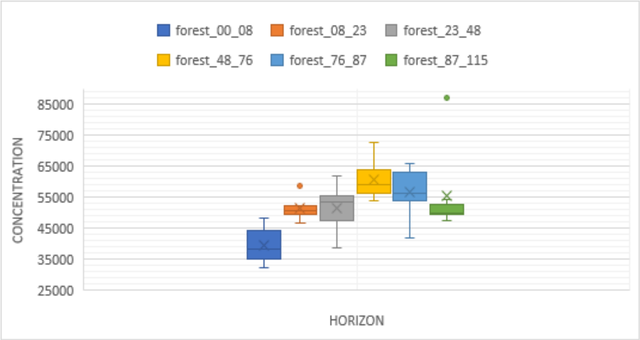

Figure 5-5: Aluminum (ppm): pre-treatment

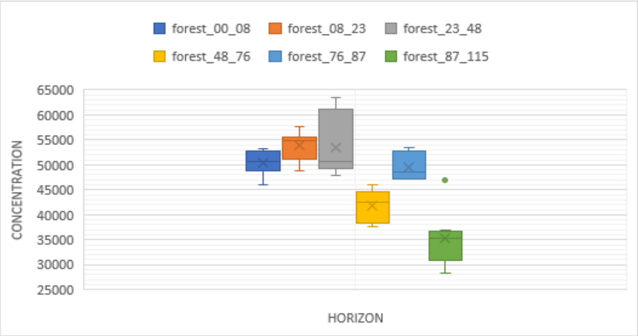

Figure 5-6: Aluminum (ppm): wet

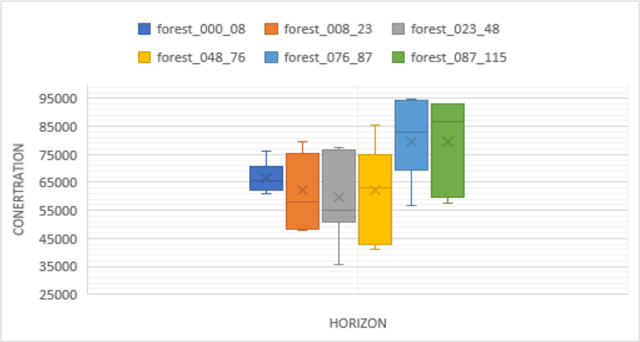

Figure 5-7: Aluminum (ppm): dry

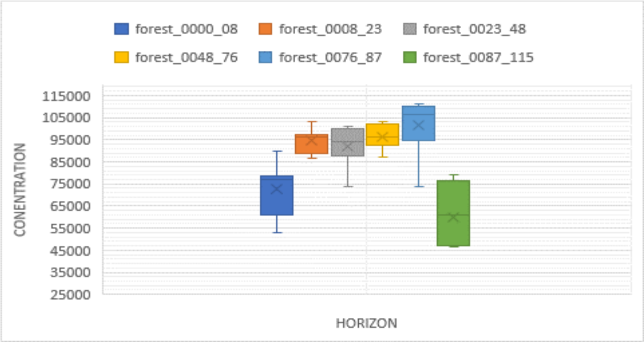

Figure 5-8: Aluminum (ppm): post-treatment

(Aluminium element analysis, own work)

5.3 Calcium element

The results of the pre-treated samples of calcium are depicted in (Figure 5-9), the results of the wet samples of calcium are shown in (Figure 5-10), the results of the dry samples are shown in (Figure 5-11), and the results of the post-treatment samples are shown in (Figure 5-12). These results show a small concentration of calcium in the O horizon (0-23 cm), A horizon (8-23 cm), AB horizon (23-48 cm), and Bt horizon (48-76 cm), followed by a slightly increased concentration in the BC horizon (76-87 cm) and a significant increase in the concentration of Ca in the Ck horizon (87-115 cm).

The PXRF can measure Ca, according to soil science. Weathered limestone and the deterioration of some primary minerals are the main sources of calcium in the soil. Available Ca and soil pH are typically directly correlated. For instance, the amount of exchangeable Ca increases as pH rises due to an increase in base saturation, which is consistent with (Figure 3-1) and Table 1 (Wood et al., 2005), (Arenberg & Arai, 2019) confirm that.

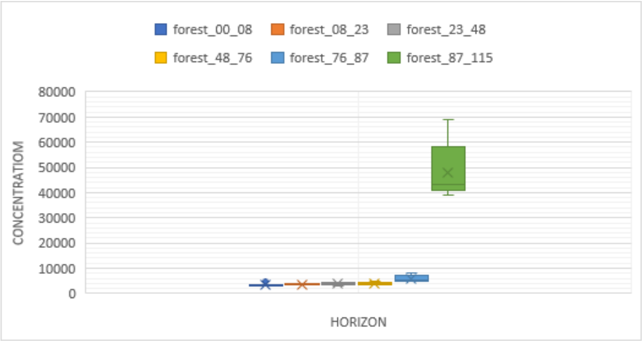

Figure 5-9: Calcium (ppm): pre-treatment

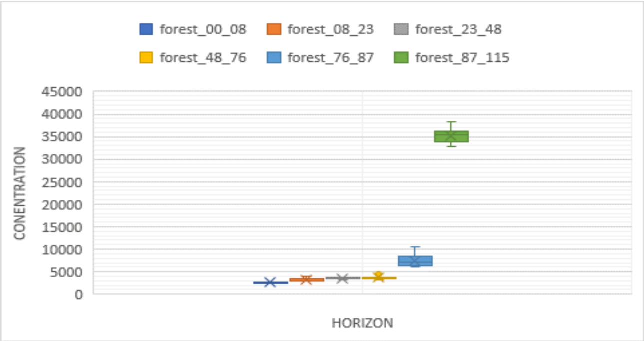

Figure 5-10: Calcium (ppm): wet

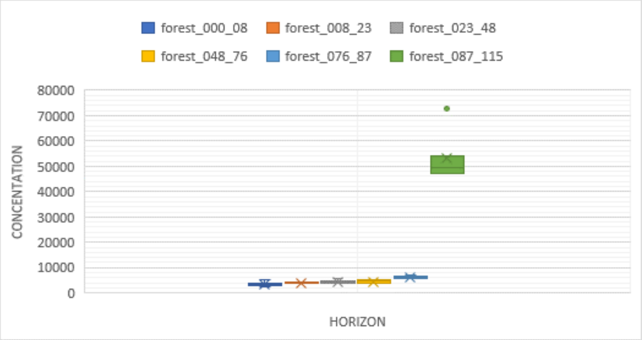

Figure 5-11: Calcium (ppm): dry

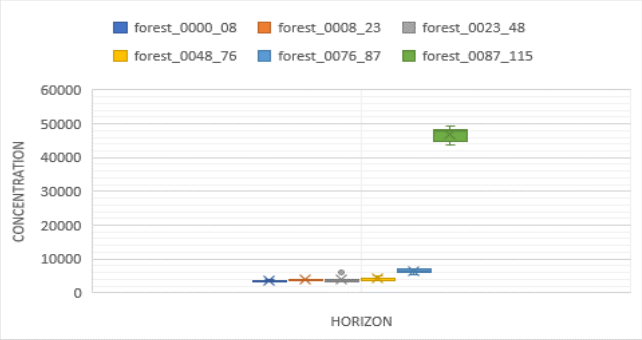

Figure 5-12: Calcium (ppm): post-treatment

(Calcium element analysis, own work)

5.4 Sulphur element

The results of the pre-treated samples of S are shown in (Figure 5-13); in the AB horizon (23-48 cm), Bt horizon (48-76 cm), BC horizon (76-87 cm), and Ck horizon (87-115 cm) no values for S were observed. whereas the O horizon (0-23 cm) and A horizon (8-23 cm) have values. The results of the wet samples of S recorded in all horizons O horizon (0-23 cm), A horizon (8-23 cm), AB horizon (23-48 cm), Bt horizon (48-76 cm), BC horizon (76-87 cm), and Ck horizon (87-115 cm) can be seen in Figure (5-14). The results of the dry samples of S are shown in (Figure 5-15); in the A horizon (8-23 cm), AB horizon (23-48 cm), Bt horizon (48-76 cm), and BC horizon (76-87 cm) no values of S were recorded. In contrast, values can be found in the O horizon (0-23 cm) and Ck horizon (87-115 cm). The results of the post-treatment samples of S recorded in the O horizon (0-23 cm), A horizon (8-23 cm), AB horizon (23-48 cm), Bt horizon (48-76 cm), and BC horizon (76-87 cm) appear in (Figure 5-16). However, there isn't a value record in the Ck horizon (87-115 cm).

These findings indicate that PXRF is not able to detect Sulphur and that it has limitations when it comes to light major element identification.

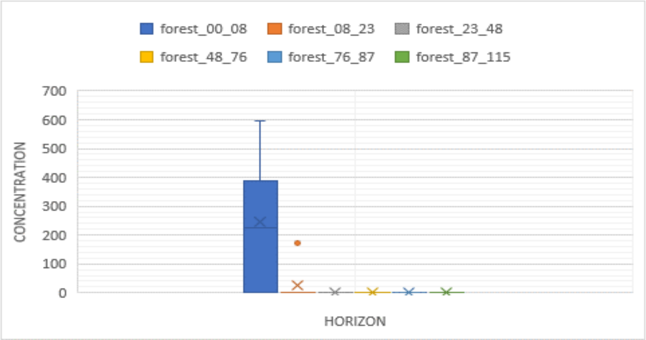

Figure 5-13: Sulphur (ppm): pre-treatment

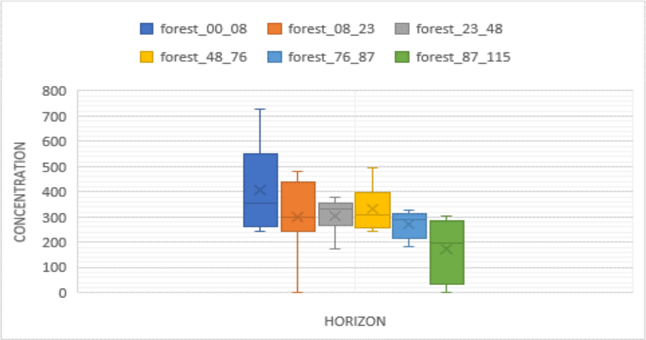

Figure 5-14: Sulphur (ppm): wet

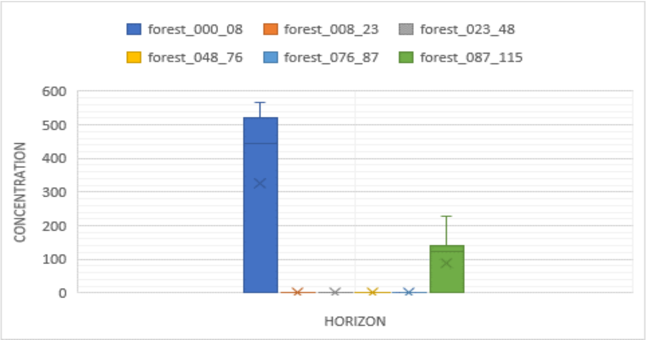

Figure 5-15: Sulphur (ppm) dry

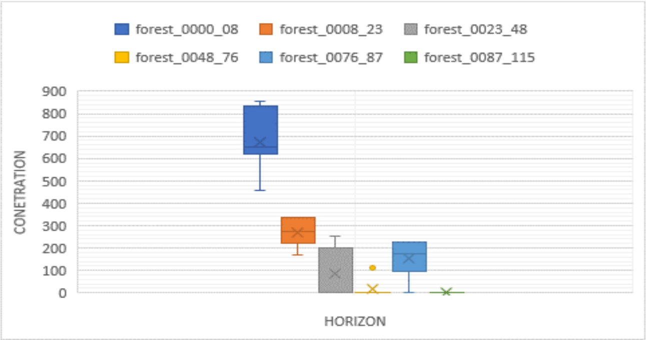

Figure 5-16: Sulphur (ppm): post-treatment

(Sulphur element analysis, own work)

5.5 Manganese element

The manganese element in pre-treatment, wet, dry, and post-treatment statuses is displayed in (Figure 5-17), (Figure 5-18), (Figure 5-19), and (Figure 5-20). Figures show that the manganese concentrations in the four statuses do not significantly differ from one another.

According to the data, the PXRF can measure manganese concentration (Foecke et al., 2022) (Zhu et al., 2011a). This is confirmed.

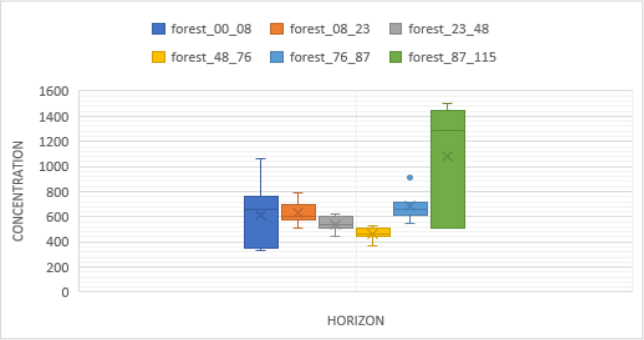

Figure 5-17: Manganese (ppm): pre-treatment

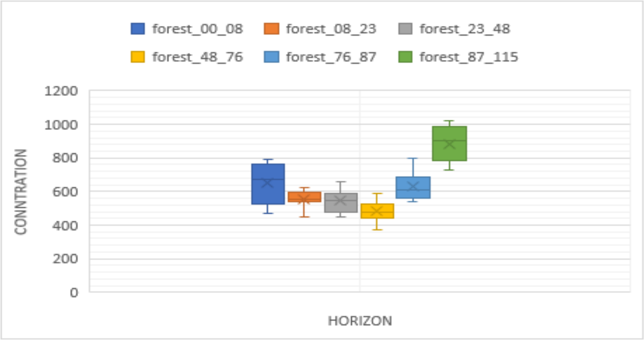

Figure 5-18: Manganese (ppm): wet

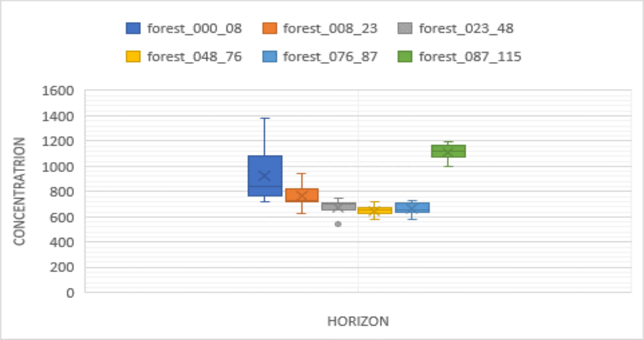

Figure 5-19: Manganese (ppm): dry

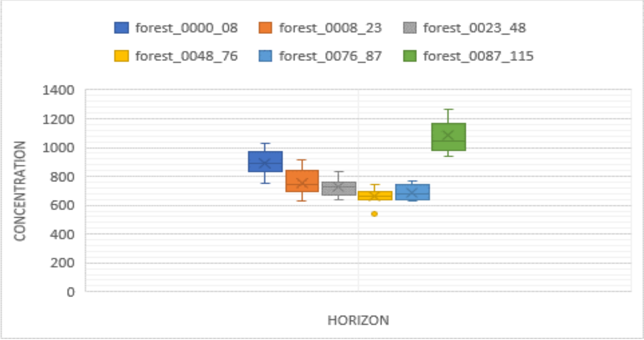

Figure 5-20: Manganese (ppm): post-treatment

(Manganese element analysis, own work)

5.6 Iron element

(Figure 5-21), (Figure 5-22), (Figure 5-23), and (Figure 5-24) show the iron element in the wet, dry, post-treatment, and pre-treatment statuses. As shown in Figures, no obvious change in concentration was observed across all statuses. The dry status in the BC horizon (76-87 cm) has slightly changed, but it has not changed significantly.

According to (Weindorf et al., 2012) (Zhu et al., 2011a), the data indicates that the PXRF can measure iron levels.

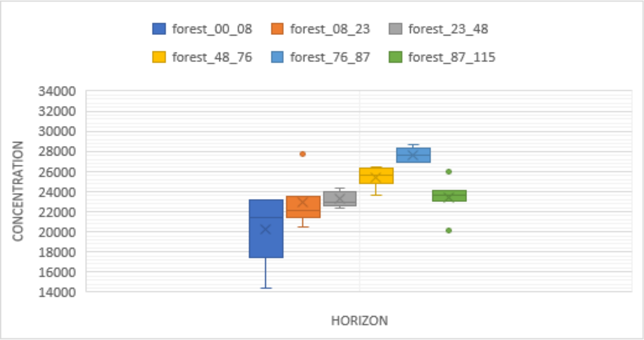

Figure 5-21: Iron (ppm): pre-treatment

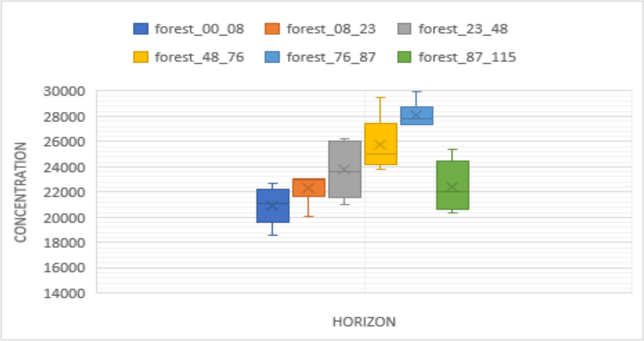

Figure 5-22: Iron (ppm): wet

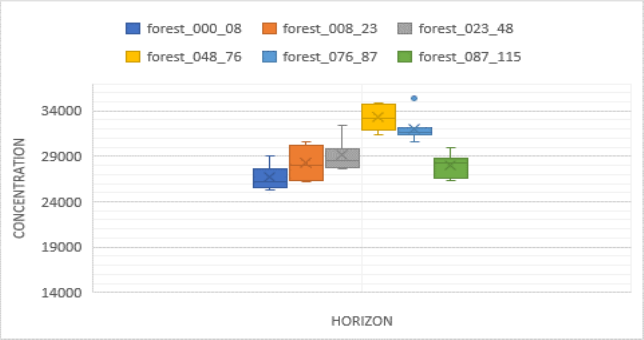

Figure 5-23: Iron (ppm): dry

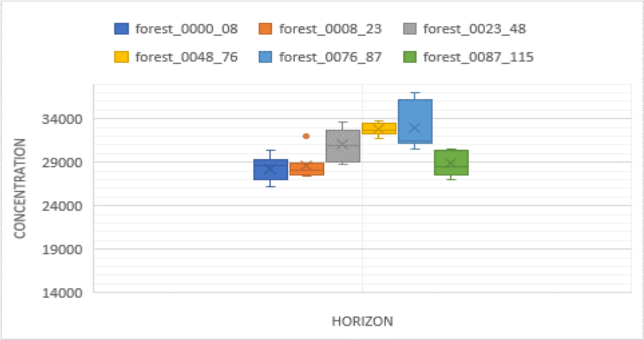

Figure 5-24: Iron (ppm): post-treatment

(Iron element analysis, own work)

5.7 Rubidium element

The rubidium element is shown in (Figure 5-25), (Figure 5-26), (Figure 5-26), and (Figure 5-27) in its wet, dry, post-treatment, and pre-treatment statuses. The four statuses have changed slightly, as seen in Figures, but not significantly.

Data from (Zhu et al., 2011a) support the suggestion that the PXRF can measure rubidium levels.

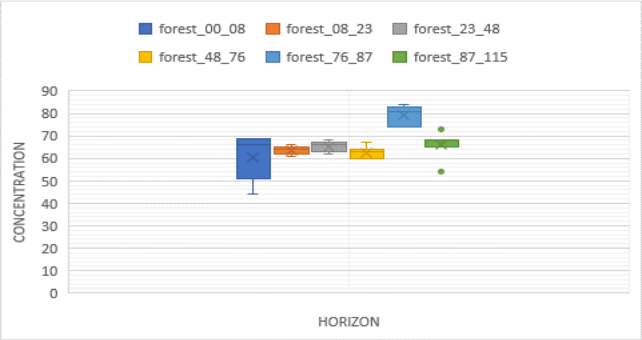

Figure 5-25: Rubidium (ppm): pre-treatment

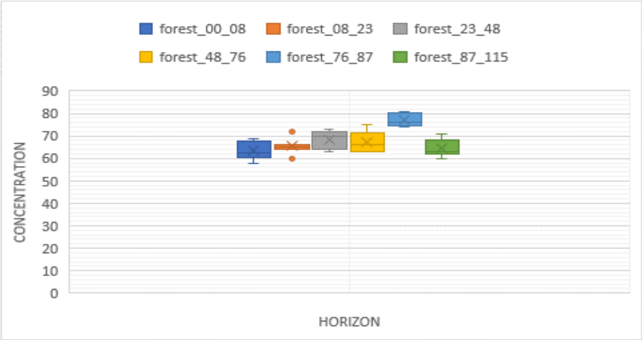

Figure 5-26: Rubidium (ppm): wet

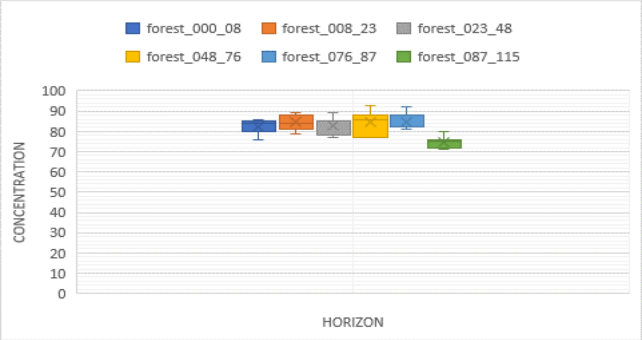

Figure 5-27: Rubidium (ppm): dry

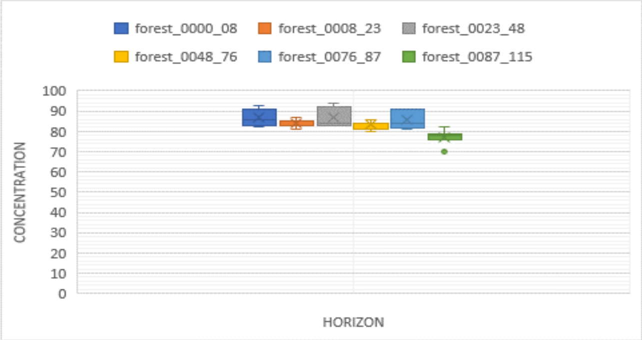

Figure 5-28: Rubidium (ppm): post-treatment

(Rubidium element analysis, own work)

5.8 Titanium element

(Figure 5-29), (Figure 5-30), (Figure 5-31), and (Figure 5-32) show the titanium element in wet, dry, post-treatment, and pre-treatment states. There is no change in concentration in the dry or post-treatment states, but there is a slight change in the BC horizon (76-87 cm) and Ck horizon (87-115 cm) in the pre-treatment state, and a change in the BC horizon (76-87 cm) in the wet state.

The data indicated that the PXRF could be used to measure titanium concentration (Zhu et al., 2011a) confirm that.

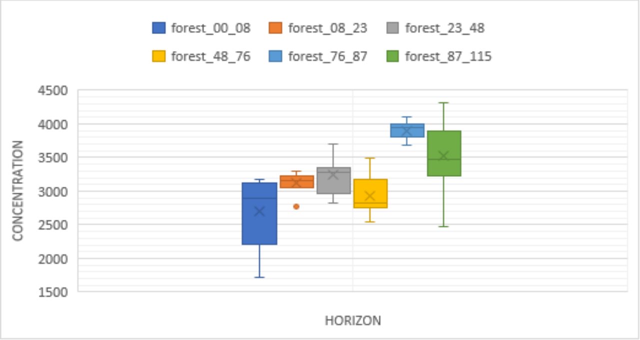

Figure 5-29: Titanium (ppm): pre-treatment

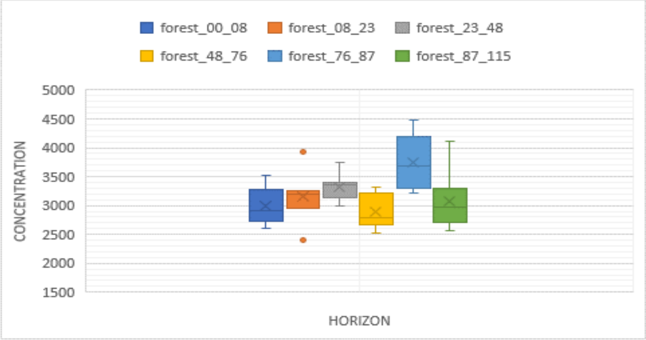

Figure 5-30: Titanium (ppm): wet

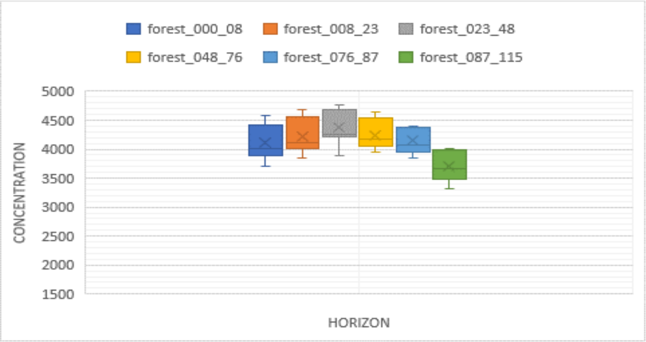

Figure 5-31: Titanium (ppm): dry

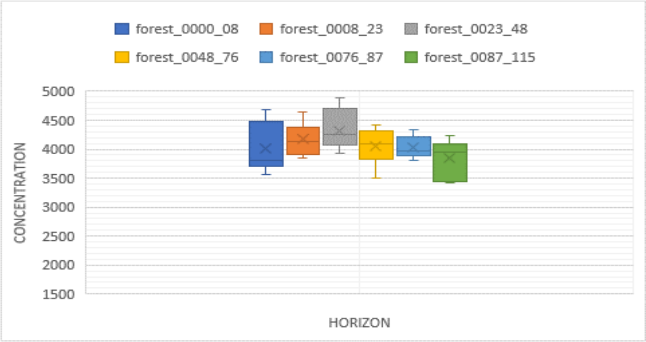

Figure 5-32: Titanium (ppm): post-treatment

(Titanium element analysis, own work)

6 Summary and Recommendation

Summary

In summary, the feasibility of using portable X-ray fluorescence to determine element concentrations in situ and in the lab was investigated in this study. A recent high-resolution, portable XRF analyzer not only brings excellent performance to the field site, often matching that of the laboratory instrument but also unrivaled time and cost savings. In the study area (Gödöllő University forest) seven PXRF measurements were taken on each genetic soil horizon of a soil profile. Elements were measured in the field and in the lab when the sample was wet, dry, and post-treatment. Sample treatment methods included air drying, homogenization, and fine grinding. The results showed that there was no significant difference in element concentrations (calcium (Ca), manganese (Mn), iron (Fe), rubidium (Rb), and titanium (Ti)) in all four states, whereas (magnesium (Mg), aluminum (Al), and sulfur (S)) measurements show a large difference in element concentrations, with no values in some states or very low in others due to their lightness. As for the other factors, temperature, time, soil moisture, and treatment method, they do not affect the results

This study underscores the potential of PXRF as a valuable technique for the real-time assessment of soil concentration profiles on the site. By offering consistent results for specific elements and identifying limitations for others, PXRF emerges as a promising tool for a wide range of soil-related applications. These findings contribute to the ongoing development of more efficient and accurate methods for understanding soil properties and supporting various environmental and agricultural endeavors.

Recommendation

I recommend using this PXRF to determine the concentrations of elements on the site, such as concentrations (calcium (Ca), manganese (Mn), iron (Fe), rubidium (Rb), and titanium (Ti)) in all four states, whereas I do not recommend determining element concentrations for light elements such as, (magnesium (Mg), aluminum (Al), and sulfur (S)).

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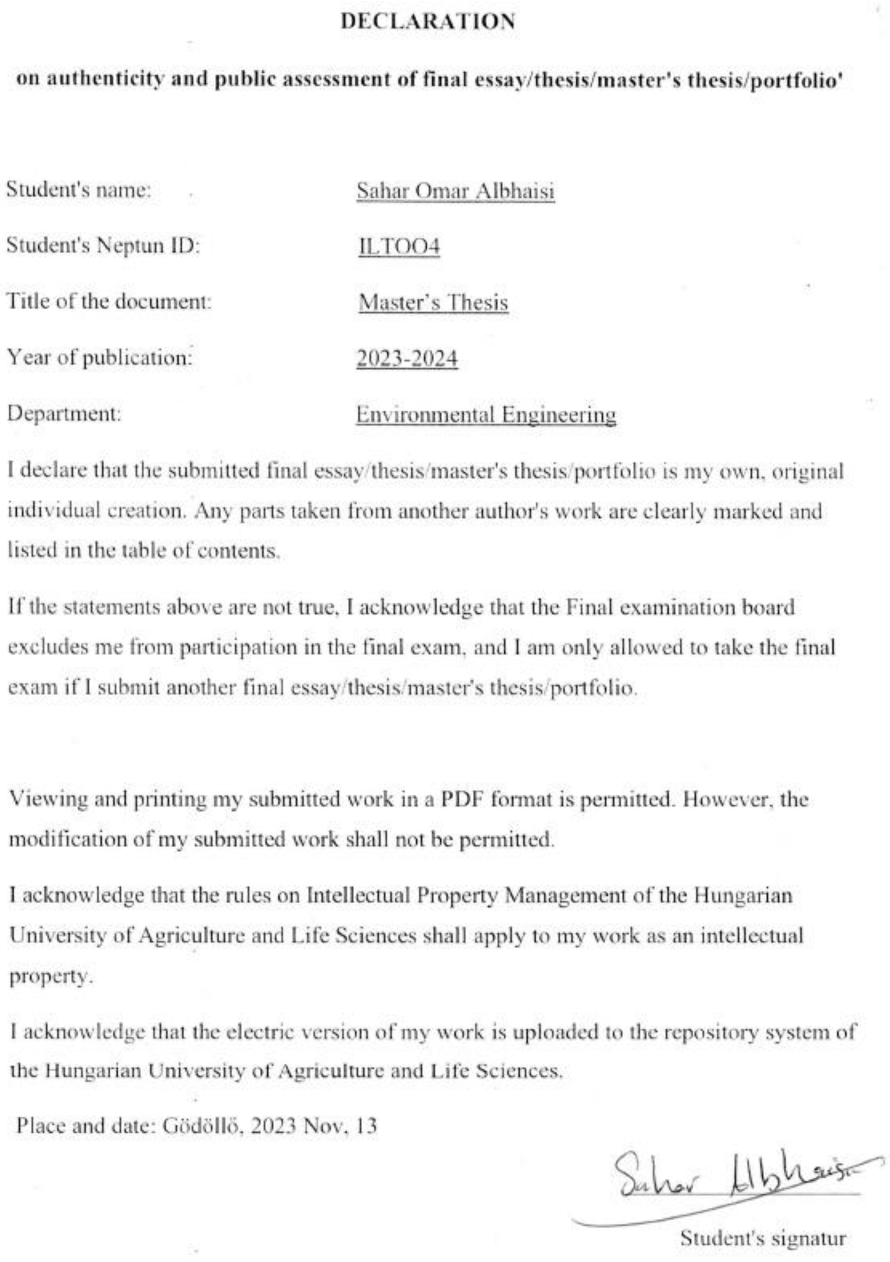
8 Declaration

1. Student



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on authenticity and public assessment of final essay/thesis/master's thesis/portfolio'

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Student's Neptun ID:	ILTOO4
Title of the document:	Master's Thesis
Year of publication:	2023-2024
Department:	Environmental Engineering

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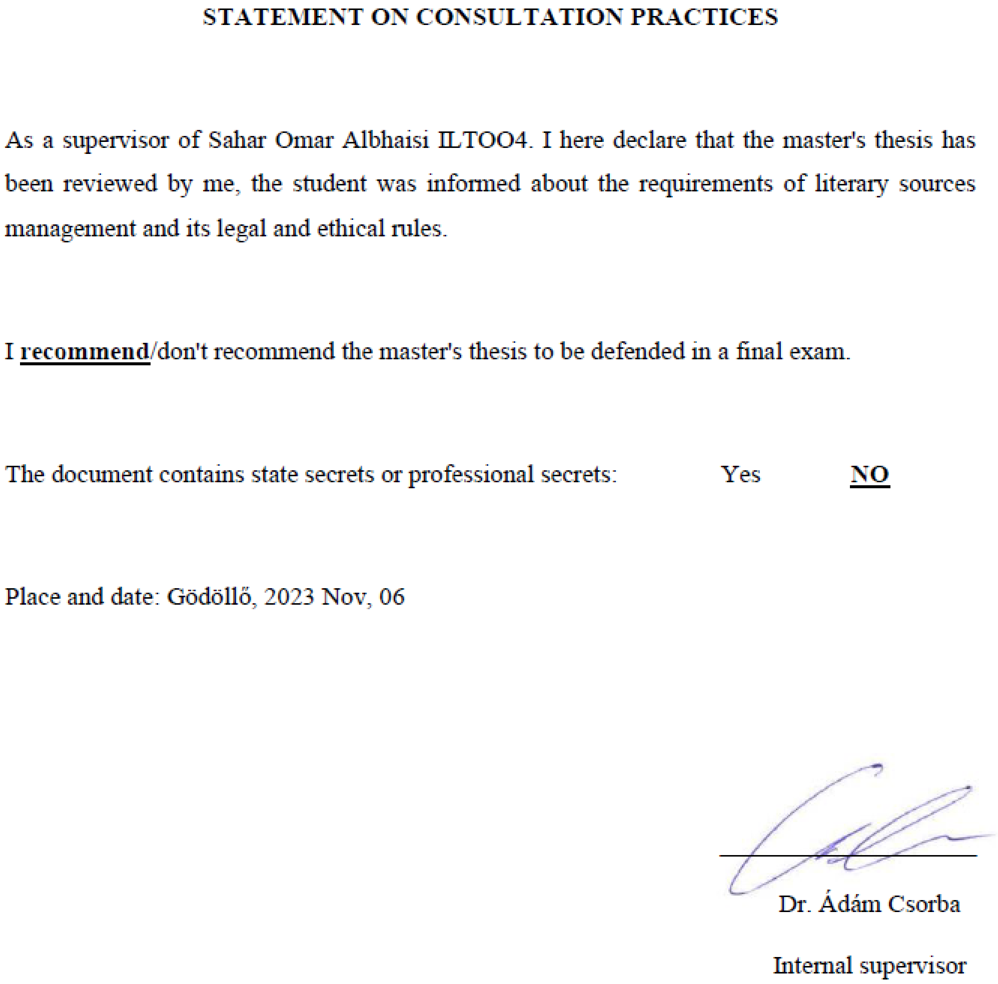
Sahar Albhaisi

Student's signatur

2. Supervisor



STATEMENT ON CONSULTATION PRACTICES

As a supervisor of Sahar Omar Albhaisi ILTOO4. I here declare that the master's thesis has been reviewed by me, the student was informed about the requirements of literary sources management and its legal and ethical rules.

I **recommend**/don't recommend the master's thesis to be defended in a final exam.

The document contains state secrets or professional secrets: Yes **NO**

Place and date: Gödöllő, 2023 Nov, 06

Dr. Ádám Csorba

Internal supervisor

Attachments

Annex A: 01-forest_XRF_data_field
Annex B: 02_forest_XRF_data_wet
Annex C: 03_forest_XRF_data_dry
Annex D: 04_forest_XRF_data_treatment