



Soil analytical quality control by traditional and spectroscopy techniques: Constructing the future of a hybrid laboratory for low environmental impact

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ABSTRACT

Soil analysis is an important information in agriculture and environmental monitoring. It is usually performed by wet chemical analysis with high cost and chemical products consumption. In the world, it is estimated that 1.5 billion ha is used as agricultural area. If every 5 ha 2 samples (2 depths) were collected, we would have 600 million soil samples for chemical and granulometric analysis. Considering just the analysis of organic matter (OM) by wet combustion method in the laboratory as an example, we would be utilizing about 840 thousand kg of dichromate and ammonium ferrous sulfate and 3 million L of sulfuric acid. The use of these reagents can have a huge ecological consequence if they do not have an adequate final disposal. An alternative methodology such as proximal sensing can be utilized with low environmental impact. Therefore, the objective of this study was to: i) evaluate the analytical quality of soil attributes via different traditional laboratories and sensors, ii) evaluate the prediction of the models using sensors, iii) assess the uncertainties of lime recommendation analyzed by the laboratories. We applied 96 soil samples at two depths collected in São Paulo State, Brazil. The determination of 15 soil attributes was performed by four different routine laboratories, and they were predicted by 4 sensors (400–2500 nm). Results indicate that the determination of attributes via chemical analysis with low quality led to high error in spectral models. The great predictive performances of clay, OM, cation exchange capacity (CEC), and pH enable the use of sensors in the evaluation of these attributes. Overall, the criteria for classification of analytical results showed that sand, silt, clay, pH, OM, CEC, and base saturation were the attributes that can be determined by the spectroscopy technique with high-quality outcome. The lime recommendation derived from proximal sensor analysis can be used as an efficient method, since it presented a high correlation with the laboratory result. In this sense, a hybrid laboratory analysis can be developed to optimize analysis with better quality control, which is indicated as a great opportunity in the near future.

1. Introduction

Soil is one of the most important resources for humanity. The chemical and granulometric characteristics of soils are responsible for water dynamics, climate, organisms, forests, carbon and others. In addition to their environmental importance, they are the basis for food production. FAO indicates that in the next 30 years we will increase by 35% in population, going from 7.6 to 10.26 billion people in the world. How to feed so many people? This concern was also discussed in [McBratney et al. \(2014\)](#), in which the authors introduced the term “soil security”, indicating that it is imperative to take care of soils or we will likely have environmental problems in several areas.

The study of soil attributes (i.e., clay, carbon, nutrients) implies on the determination of its analytical value. Soil attributes mapping is recommended prior to soil management, as soil analyses are essential for assessment and monitoring of its chemical and physical conditions, indicating the need for fertilization, liming and conservation techniques. The most applied chemical methods in laboratories are those called traditional wet analysis.

Considering Brazil, the agriculture area is around 60 million ha with a potential to expand to 70 million in the coming years. Nowadays, Brazil uses precision agriculture (PA) techniques in approximately 10% of its farmland, which involves between 100,000 and 200,000 soil analyses per year. The number of soil analyses is even more impressive

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when considering sampling at two depths (0–20 and 20–40 cm, in general), which can reach 400,000 soil analyses. Considering the world demand, there is an estimate that > 600 million soil samples can be analyzed every year.

However, the numbers of analyses are not exactly the main issue. One analysis takes about 3 to 15 days for delivering results, which is not adequate considering the speed required in PA. Despite the longtime, the traditional laboratory analysis uses several types of chemical substances. For instance, to determine the soil organic matter (OM), the wet combustion method is predominantly applied, which uses dichromate ($\text{Cr}_2\text{O}_7^{2-}$) (0.196 g), ammonium ferrous sulfate hexahydrate ($\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$) (1.20 g), and sulfuric acid (H_2SO_4) (5 mL) for only one sample. Considering just the analysis of OM, 1.396 g and 5 mL of these reagents are used, reaching a disturbing number of 698 kg and 2500 L of these toxic chemical compounds per year. Considering the potential of 600 million soil samples to be analyzed globally, it will consume about 840 thousand kg of dichromate and ammonium ferrous sulfate and 3 million L of sulfuric acid. Besides, for OM, it is estimated a cost of US\$ 5.00 per sample, with an annual expenditure of US\$ 2.5 million. We have to emphasize that this is not a local problem. Countries with established agriculture, especially the developing ones (from Latin America and Africa) that have a lot of land for agricultural expansion, are the most affected.

Soil analyses are subject to several errors due to sampling, products, methods, humans, and others (Olsen and Sommers, 1982). In fact, O'Rourke and Holden (2011) stated that routine analyses have some limitations, i.e., reagents quality, brands of equipment and extensive sequence of steps. Consequently, repeated analysis of the same sample may show variations. These variations are only acceptable if they are within a certain interval. This was ratified by Cantarella et al. (2006) that identified differences for a same sample in the same or between laboratories. These issues took the community of soil scientists on the investigation of new techniques for soil analysis. The main focus was how to reach soil results in a fast, simple, more stable and no-pollutant method.

In this sense, years of experiments took many authors to observe proximal sensing as a promising technique that can bring light to the issue. Indeed, first findings of Zheng and Schreier (1988) quantified soil patterns and field fertility using spectral reflection. Ben-Dor and Banin (1995) created the near infrared analysis (NIRA), which is an approach to examine the capability for predicting soil properties from the reflectance curves in the near infrared region of arid and semiarid soils of Israel.

Spectroscopy is a proximal sensing technique based on the detection of the electromagnetic radiation reflected by the soil. In the same way, spectroscopy in the visible (Vis: 400–700 nm), near infrared (NIR: 701–1100 nm) and short-wave infrared (SWIR: 1101–2500 nm) regions of the electromagnetic spectrum associated with chemometric methods has allowed the quantification of physical, chemical and mineralogical attributes (Viscarra Rossel and Behrens, 2010). Vis-NIR-SWIR spectroscopy technique emerges as a promising new option for soil analysis, with advantages such as: the possibility of predicting several attributes in just one spectral reading, facility of data acquisition from large amounts of samples, it is a rapid analysis and without the use of environmentally hazardous chemicals (Minasny and McBratney, 2008; Viscarra Rossel and Behrens, 2010). Nanni and Demattê (2006) reached important correlation between traditional and spectroscopic methods, but the authors emphasized the necessity of more research in this regard. Although spectroscopy is promising, results are variable as reviewed by Soriano-Disla et al. (2014) and Nocita et al. (2015). O'Rourke and Holden (2011) highlighted that with the spectroscopic methods it is possible to have costs reduction for OM of about 90%, with the potential to read 720 samples per day. In addition, the soil attributes quantification via reflectance spectroscopy technique is based on multivariate statistical methods, generating calibration models that correlate the spectral with the analytical values obtained from laboratories

with standardized methodologies. Thus, the predictive quality of the models depends on the precision and accuracy of the reference laboratory determinations (Rayment et al., 2012; Reeves III, 2010).

The types and the range of spectral sensors also influence the quantification of soil characteristics. This method of determining soil attributes by sensors has gained prominence due to two fundamental factors, namely fast information and the environmental appeal regarding the use of a safe methodology. Hence, the quantification of soil attributes must be performed to generate necessary information about it and ensure proper management of this natural resource.

Techniques using spectral data have been applied and shown promising success in the quantification of soil attributes worldwide (Viscarra Rossel et al., 2016). However, few scientific studies compared the analytical results between different laboratories to evaluate how their data impact on the spectral models. If we assure that the variation on the results are real, can sensors detect this variation? How much of the sensor prediction performance is affected by the reference value of laboratory analysis? Can a sensor perform better applying the most accurate reference value or applying an average reference value? The answers for these questions are still unknown.

Regarding the variations of soil spectral features considering different sensors, Romero et al. (2018) observed that there is low difference between sensor measurements, which leads to a more stable technique. Taking into consideration these low differences, caused by geometry and equipment variation, Ben-Dor et al. (2015) determined a protocol to standardize measurements between sensors. This takes spectroscopy methodology a step ahead of traditional ones.

Seeking the increase of food production for the human needs, optimization of fertilizers in agricultural production, and the decreasing of chemical products in the soil analyses, it is essential to search for alternatives and thereby reduce the environmental impact. In this sense, understanding the variations between laboratories is a fundamental part of this study. We intend to demonstrate the advantages and limitations of soil analysis methodologies and the impact of different laboratories and sensors in the prediction of attributes, thus allowing better decisions for soil management. We understand that it is not possible to substitute traditional soil analysis completely, because reflectance is a dependent variable. The objective of the present study was to: i) evaluate the analytical quality of soil attributes via different laboratories and sensors, ii) evaluate the prediction of the models using sensors, and iii) assess the uncertainties of lime recommendation analyzed by the laboratories. Therefore, the focus is to bring light on the development of a hybrid laboratory analysis approach, where one would have in simultaneous both measurements: 80% of soil samples analyzed by spectroscopy and only 20% by traditional analysis.

2. Material and methods

2.1. Characterization of the study area, collection and preparation of soil samples

We collected the soil samples in an area that covers 29 municipalities located at São Paulo State, Brazil. The study areas present tropical climate, with hot and humid summer, cold and dry winter, with average temperatures of 20 °C, with annual thermal amplitude of up to 7 °C. Rainfall varies from 1000 to 1500 mm/year. 48 profiles were collected at two depths of 0–20 cm and 80–100 cm (A and B horizons, respectively) totalizing 96 soil samples. The collected material was dried in 45 °C for 48 h, ground and sieved in a 2 mm mesh.

2.2. Laboratory and spectral analysis

For the reference soil analysis, four laboratories were selected in São Paulo State, which are regularly evaluated by the proficiency test of the Agronomic Institute of Campinas (IAC), Brazil. The 96 soil samples were divided in three replicates (in a total of 288 soil samples) and sent

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