



Visible near infrared reflectance spectroscopy to predict soil phosphorus pools in chernozems of Saskatchewan, Canada



Dalel Abdi^a, Barbara J. Cade-Menun^{b,*}, Noura Ziadi^a, Gaëtan F. Tremblay^a, Léon-Étienne Parent^c

^a Agriculture and Agri-Food Canada, Quebec Research and Development Centre, 2560 Hochelaga Boulevard, Québec, QC G1V 2J3, Canada

^b Agriculture and Agri-Food Canada, Swift Current Research and Development Centre, P.O. Box 1030, Swift Current, SK S9H 3X2, Canada

^c Department of Soils and Agri-Food Engineering, Université Laval, Québec, QC G1K 7P4, Canada

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ABSTRACT

To date, there are few direct or rapid methods to quantify the concentration of many soil phosphorus (P) pools, including organic P (OP). Visible near infrared reflectance spectroscopy (VNIRS) is a rapid, inexpensive, and accurate technique for analyzing a wide variety of organic materials and is increasingly used in soil science. The aim of this study was to examine the potential of VNIRS to predict the concentrations of a number of soil P pools, including OP, total P analyzed by digestion (TP), available P using the Olsen (P_{Ols}) and Mehlich-3 (P_{M3}) methods, and other related soil chemical properties [organic matter (SOM) and Mehlich-3 extractable Al, Fe, Ca, Mg, and Mn] that could influence the soil P cycle. Soil samples ($n = 360$) were taken from experimental sites near Indian Head, SK, Canada, from short-term (8 yr) and long-term (31 yr) no-tillage plots of a field pea (*Pisum sativum* L.)–spring wheat (*Triticum aestivum* L.) rotation receiving five P fertilizer application rates annually. We randomly selected 80% of each sample set for calibration, while the remaining 20% was used for validation. Prediction models were developed using modified partial least squares regression with cross-validation. The VNIRS models were evaluated using the coefficient of determination of validation (R_v^2) and the ratio of standard error of prediction to standard deviation (RPD). The VNIRS predictions for OP were moderately successful for the total soil sample set and for the short-term set ($0.80 \leq R_v^2 < 0.90$ and $2.25 \leq RPD < 3.00$), and were moderately useful for the long-term set ($0.70 \leq R_v^2 < 0.80$ and $1.75 \leq RPD < 2.25$). The VNIRS predictions were successful for soil Ca_{M3} and SOM, moderately successful for Mg_{M3} , moderately useful for P_{Ols} , P_{M3} , and Al_{M3} , and less reliable for TP, Fe_{M3} , and Mn_{M3} . This study demonstrated that VNIRS is a promising analysis technique for soil OP, P_{Ols} , P_{M3} , Ca_{M3} , Al_{M3} , Mg_{M3} , and SOM. Success in predictions of OP, P_{Ols} , and P_{M3} may be attributed to their relationship to SOM and/or to Ca_{M3} or Mg_{M3} .

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1. Introduction

Phosphorus (P) is one of the most important nutrients and occurs in inorganic and organic pools in soils. Inorganic P as orthophosphate (HPO_4^{2-} or $H_2PO_4^-$) is the directly available form for plant or microbial uptake. Within plants and microbes, orthophosphate is converted to a number of organic P (OP) compounds for metabolism and storage. This OP is ultimately returned to the soil, where it acts as a resource of long-term bio-available P (Oehl et al., 2001). To date, total OP cannot be estimated directly, and instead is determined indirectly as the difference between total P (TP) and inorganic P (IP) by ignition (Saunders and Williams, 1955) or extraction (Hedley et al., 1982; Bowman and Moir, 1993; Turner et al., 2005). These procedures involve treating soil with several chemical reagents and are time consuming. Furthermore, there is potential for error in the steps for TP and IP determination, increasing the potential for errors in OP measurement. Advances in

understanding the soil OP cycle at various time and spatial scales require the development of an appropriate method that can rapidly and accurately measure OP.

Visible near infrared reflectance spectroscopy (VNIRS) is a rapid, inexpensive, accurate, and environmentally friendly technique that can predict the concentration of soil attributes from an empirical model calibrated using soil spectra (Chang et al., 2001; Malley et al., 2004). Many soil attributes are predictable using a single reflectance spectrum (Viscarra Rossel et al., 2006). Hence, it could be a promising alternative technique to routine soil analysis methods. The VNIRS spectra result from the radiation absorbed by various chemical bonds (e.g. C—H, N—H, O—H, C—O, C—N, and N—O) found in soil organic constituents, as well as Ca—O, Fe—O and Al—O in minerals (Miller, 2001; Soriano-Disla et al., 2014; Nocita et al., 2015). Thus, soil properties such as soil organic matter (SOM), carbon (C), texture, moisture, calcium (Ca), magnesium (Mg), aluminum (Al)/iron (Fe) oxyhydroxides and iron oxides, termed primary properties, can be directly estimated by VNIRS (Dalal and Henry, 1986; Reeves et al., 1999; Chang et al., 2001; Cozzolino and Morón, 2006; Brunet et al., 2007). Other soil parameters that are

* Corresponding author.

E-mail address: barbara.cade-menum@agr.gc.ca (B.J. Cade-Menun).

spectrally inactive such as pH, P, potassium (K), manganese (Mn), zinc (Zn), and copper (Cu) may be predicted by VNIRS (Chang et al., 2001) owing to their relationship to primary properties (Sarathjith et al., 2014).

The ability of VNIRS to predict soil constituents, more or less accurately, has been shown in several studies (Chang et al., 2001; Brunet et al., 2007; Nduwamungu et al., 2009a; Abdi et al., 2012; St. Luce et al., 2012, 2014). Abdi et al. (2012) found a moderately useful prediction for total P ($0.70 \leq R^2 < 0.80$) by VNIRS for 192 gravely sandy loam soil samples from an experiment site in eastern Canada, due in part to its relationship to spectrally active SOM. The potential of VNIRS to predict soil TP has been investigated in a few studies and ranged from moderately successful in fine sandy soil (Bogrekci and Lee, 2005) to moderately useful in clay soil (Malley et al., 2004).

Soil available P prediction using VNIRS has been shown to depend on the type of extracting solution used for the analysis. For example, Chang et al. (2001) and Nduwamungu et al. (2009a) confirmed that soil P extracted with Mehlich-3 solution (P_{M3}) was poorly predicted by VNIRS for soils collected from 448 sites in the U.S.A, and 150 soil samples collected from five soil series near Montreal (Quebec, Canada), respectively. A similar result was obtained by Abdi et al. (2012), probably due to the poor correlation of P_{M3} with organic C. However, VNIRS results were considered useful when P was extracted using the Olsen method (P_{Ois}) (van Groenigen et al., 2003). In a recent study conducted in Germany, results showed acceptable near infrared reflectance spectroscopy (NIRS) prediction for rapidly and slowly available organic P fractions extracted using the Hedley procedure from one soil type sampled from one small region (Niederberger et al., 2015). The authors found that model prediction quality was higher for organic than for inorganic P fractions, because organic compounds can be more easily excited by the NIR irradiation than inorganic ones. To assess whether the prediction of P by NIRS depended on direct detection of soil P or rather indirectly on interaction with SOM, the authors used regression analysis between the predictive quality of NIRS models, and the relationships between P and soil total C and total nitrogen (N). To our knowledge, the VNIRS prediction potential of soil total OP has not been previously assessed.

The Mehlich-3 extraction is used in many Canadian provinces as the official agro-chemical method for soil testing (Ziadi et al., 2013), because it extracts available P and exchangeable cations. Nduwamungu et al. (2009a) found that extractable Ca, Cu, and Mg could be reliably predicted by NIRS, whereas Al, Fe, K, Mn, and Zn have not been successfully predicted. Abdi et al. (2012) reported excellent prediction potential of VNIRS for Mehlich-3 exchangeable Ca and Mn ($R^2 > 0.95$), successful for Mg ($0.90 \leq R^2 < 0.95$), and moderately useful for Fe and Zn ($0.70 \leq R^2 < 0.80$), but less reliable prediction for K and Cu ($R^2 < 0.70$). They concluded that these results should be validated on soils of diverse textures that could influence spectral absorbance.

The objectives of this study were to assess (i) the potential of VNIRS to predict soil P pools, including total OP, TP, P_{Ois} , and P_{M3} , and other chemical attributes, such as SOM, and Mehlich-3 extractable Al, Fe, Ca, Mg, and Mn, in loam and clayey loam soils collected from adjacent farms under different long-term management near Indian Head, SK, Canada; and (ii) whether the VNIRS predictions of P pools depended on their relationship to SOM and/or to soil minerals.

2. Materials and methods

2.1. Experimental site description

The experimental site was described in detail by Lafond et al. (2011). Briefly, the experiment was located approximately 19 km south-east of Indian Head, Saskatchewan, Canada (50.42° N, 103.58° W), on two adjacent fields (Fig. 1), one under short-term no-till (ST, 8 years) and the other under long-term no-till (LT, 31 years). The soil of the LT field was loamy with a pH of 6.8, while the soil of the ST field was clayey

loam with a pH of 7.3. The soil type for both fields was Orthic Black Chernozem (Canadian System of Soil Classification, 1998). A continuous cropping no-till system was established on the LT field in 1978 and on the ST field in 2001. Prior to this, the ST field was managed using a fallow-crop system involving tillage to 10 cm soil depth. Within these farms, a number of plots were established, with a variety of treatments (Fig. 1).

The soil samples for our study were collected from plots on each farm in which a P fertilizer trial was established in 2003. The experimental design was a split-plot with crop rotation (pea and spring wheat) as main plots in each 24 m by 10.7 m block and five duplicated P treatments (0, 11, 22, 33, and 45 kg P_2O_5 ha⁻¹) as sub-plots (10 plots per block, each 2.4 m by 10.7 m). The fertilizer was applied each year, with the same application rate for each plot each year. Both crops were present at each farm each year, alternating between blocks and seeding directly into the stubble of the previous crop. There were two replicates for each crop, two tillage treatments and five duplicated fertilizer treatment combinations ($2 \times 2 \times 2 \times 5 = 40$ plots in total).

2.2. Soil sampling and analysis

Soil samples were collected in autumn 2008 ($n = 120$) and in the spring ($n = 120$) and autumn ($n = 120$) of 2009 at three depths (0–7.5, 7.5–15.0, and 15.0–30.0 cm), air-dried, sieved, and ground (<2 mm). Total P was extracted using the wet acid digestion method of Parkinson and Allen (1975). Briefly, 0.5 g of soil was mixed with 3.75 mL of H_2SO_4 and 3 mL of digestion solution (175 mL $H_2O_2 + 0.21$ g Se + 7 g $LiSO_4 \cdot H_2O$), and digested at 360 °C for 2.5 h. Total OP was determined by the difference between 0.5 M H_2SO_4 extractable P in a 0.5 g soil sample ignited at 550 °C and an unignited sample according to Saunders and Williams (1955). Concentrations of TP and OP were measured in digests or extracts by the colorimetric molybdate blue method (Murphy and Riley, 1962). Soil organic matter was determined by loss on ignition by weighing the samples that were ashed (550 °C, 3 h) during analysis for OP. Mehlich-3 extraction, followed by inductively coupled plasma optical emission spectroscopy (ICP-OES), was used to determine soil available P and exchangeable Al, Fe, Ca, Mg, and Mn (Mehlich, 1984). Available P was also extracted using the Olsen method (Olsen et al., 1954) and determined by the colorimetric molybdate blue method.

2.3. Visible near infrared reflectance spectroscopy analysis

The absorbance of soil samples [$\log(1/R)$ where R is the measured reflectance] was determined in the visible and near infrared regions between 400 and 2498 nm at 0.5 nm intervals using a NIRSTM DS 2500 monochromator Instrument (Foss NIRSystems Inc., Silver Spring, MD), with a cup containing approximately 25 mL of soil sample. Each spectrum was the average of 32 co-added scans.

Different treatments were selected to improve calibration models, including the critical T-statistics values of 2.0 and 2.5 for T-outliers detection, and the following math treatments: 1,16,16,1; 2,32,24,1; 1,20,20,1; 1,40,40,1; and 2,40,40,1. The T-outliers are determined using the Student's t-test and the standard error (Infrasoft International LLC, 2002). The four numbers (e.g. 1,16,16,1) are derivative treatments; the first indicates the order of the derivative, the second, the gap over which the derivative was calculated, the third, the number of the smoothing points, and the fourth, the second smooth. Scatter correction with standard normal variate and detrending (SNVD) was applied to all spectra using the WinISI IV software (ver. 4.6.8, Infrasoft International, LLC, State College, PA, USA) to reduce scatter and particle size effect, and to remove the linear or curvilinear trends of each spectrum (Barnes et al., 1989; Nduwamungu et al., 2009a; Abdi et al., 2012; St. Luce et al., 2012). In order to develop the most accurate VNIRS prediction models, calibration equations were developed by soil

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