Performance of mid infrared spectroscopy to predict nutrients for agricultural soils in selected areas of Ethiopia

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ABSTRACT

Knowledge of soil nutrient status is a basic requirement in sustainable agriculture. However, assessment of soil properties has long been done through conventional laboratory analysis, which is costly and time-consuming. Therefore, developing alternative, cheaper and faster techniques for soil analysis is highly required. Mid-infrared spectroscopy (MIRS) techniques are rapid, convenient, environmentally friendly, and nondestructive techniques for quantifying several soil properties. This study aimed to evaluate the prediction performance of MIR for pH, organic carbon (O.C.), available phosphorus and sulfur, total nitrogen, exchangeable cations, and micronutrient. Soil samples were collected from southern Ethiopia. In this study, properties of 3882 soil samples were used as references from different parts of Ethiopia. Partial least squares regression (PLSR) was used for calibration. The correlation of measured and predicted properties of soil samples collected was analyzed using the Pearson correlation coefficient. Better prediction was obtained for Ca ($R^2 = 0.95$ and RPD = 3.9), CEC ($R^2 = 0.92$ and RPD = 3.5), TN ($R^2 = 0.92$ and RPD = 3.4), OC ($R^2 = 0.91$ and RPD = 3.4), Mg ($R^2 = 0.84$ and RPD = 2.6), pH ($R^2 = 0.85$ and RPD = 2.4) and Fe ($R^2 = 0.65$ and RPD = 1.7). In general, soil properties could be predicted using MIRS methods. On the other hand, soil nutrients that showed poor prediction require further studies.

1. Introduction

Soil fertility is a basic requirement for sustainable agriculture in a country like Ethiopia, whose economy is based on agriculture. However, various recent studies have shown that soil fertility is declining in many farmlands mainly due to traditional farming practices [1], insufficient and unbalanced fertilization, and severe soil nutrient depletion.

The optimum and balanced fertilization is the main objective of ensuring increased production and productivity of agriculture. It is widely used by identifying and evaluating the fertility status of soils through field crop fertilizer response and laboratory analysis [1]. Most of the required analyses are time-consuming. In addition to this, using reagents for laboratory analyses may release environmental pollutant molecules [2]. Thus, the need for fast and cheap methods that would enable the analyses of a large number of samples and which are environmentally friendly. Recently, infrared spectroscopy (I.R.) has been a promising technique for predicting soil properties [3].

Technological achievements in the mid-infrared (MIR) range have made this spectral range much more attractive during the last decade. Although there is no doubt that near-infrared spectroscopy can be very useful for some analyses, several comparative studies have shown the superiority of the MIR techniques in most cases [4]. And therefore, the current study has focused solely on MIR techniques. The main difference between the two ranges is that absorption in mid-infrared spectroscopy corresponds to fundamental bands of molecular vibrations, whereas near-infrared absorptions are complex due to overtones [2].

Infrared spectroscopy operates on the principle that molecules absorb specific electromagnetic frequencies representing their structural characteristics [5]. Infrared spectroscopy estimates C and N concentrations in bulk soil [4]. Compared with conventional wet-chemistry methods, I.R., including near-IR (NIR) and mid-IR (MIR) spectroscopic techniques are time and cost-efficient, nondestructive, require less sample preparation, and often result in similar accuracy without the use of chemical reagents [2].

In laboratory analyses, Melich-III extractant is used to determine soil nutrients using an inductively coupled plasma (ICP) atomic emission spectrometer, which provides fairly accurate results because of good linear sensitivity [3]. However, such analytical methods cannot easily be adapted for field use because these instruments are quite expensive and they require complex sample pretreatment [5].
Currently, Ethiopia is using MIR to analyze some soil properties. Still, there is a scarcity of studies on the performance of MIR to predict different agricultural soil properties in the study area. Therefore, this study aimed to evaluate the potential of MIRS for predicting soil nutrients.

2. Materials and methods

A laboratory analysis was conducted to predict nutrients of 463 soil samples collected from the study area using MIR.

2.1. Description of the study area

The study was conducted in some selected woredas of Kambata Tembaro zones, including Kedida Gamela, Kecha Bira, and Damboya woredas of SNNPR (Figure 1).

2.2. Data collection and sample preparation

Three thousand eight hundred eighty-two soil samples were collected from different regions of Ethiopia and used as references. For all soil types, ten subsamples were collected within 15 m distance between and among each sub-sampling point in a circle method and composited. For each main sampling point, about 1 kg of representative composite soil sample was collected and logged in to properly labeled plastic sample bag and transported to the laboratory. Four hundred sixty-three soil samples were collected from Kedida Gamela, Kecha Bira, and Damboya woredas of SNNPR and analyzed and predicted by Mehlich 3 and MIR methods, respectively, to correlate their contents. Sample preparation was conducted at the National Soil Testing Center (NSTC), Addis Ababa. The samples were air-dried and crushed using standard procedure and sieved through a 2 mm mesh sieve.

2.3. Analysis of soil chemical properties

The pH of the soils was determined in H₂O using 1:2 soil to solution ratio using an ELMETRON pH-meter, and electrical conductivity was measured using HANNA EC 215 conductivity meter as outlined by [6]. Sulfate-sulfur (SO₄²⁻⁻S), available P, exchangeable basic cations (K, Na, Ca, and Mg), and extractable micronutrients (B, Cu, Fe, Mn, and Zn) of the soils were determined using Mehlich 3 soil test extraction procedure [7]. An inductively coupled plasma (ICP) spectrometer was used to determine the concentrations of exchangeable basic cations and extractable nutrients.

Figure 1. Map of the sampling locations.
2.4. Mid infrared (MIR) diffused reflectance analysis

The soils, including reference (standard) samples, were sub-sampled and grounded using Retsch mortar grinder RM 200 to powdery samples with appropriate size. Soil samples were analyzed using standard laboratory procedures.

2.5. Data analysis

Quantitative analysis of the spectra was done using the Quant 2 evaluation function of the OPUS version 7 software. The spectra were preprocessed using the first derivative + vector normalization (SNV) method in the spectral frequency region of 4001.6–599.8 cm⁻¹. The statistical parameters were computed using the following formula.

\[ R^2 = \left( 1 - \frac{\text{SSE}}{\sum (y_i - \bar{y})^2} \right) \times 100 \]  

(1)

where SSE is the sum of squared error, \( y_i \) is the actual value, and \( \bar{y} \) is the mean component value.

\[ \text{RPD} = \frac{\text{SD}}{\text{SEP}} \]  

(2)

where S.D. is standard deviation and SEP is the standard error of prediction and.

\[ \text{RMSEP} = \sqrt{\frac{1}{M} \sum_{i=1}^{M} \text{Differ}_i^2} \]  

(3)

where M is the number of spectra, and Differi is the difference between the actual concentration of sample i (as determined by another method) and the predicted concentration.

3. Results and discussions

3.1. Descriptive statistics of reference samples

Descriptive statistics analysis was summarized in Table 1. The concentration of P, K, Na, S, Zn, and Cu for the studied samples was highly skewed. This variation in the sample suggested the need for transformation. The log transformation was robust and improved their normality.

3.2. Calibration and validation of the models

A preliminary calibration model was generated using MIR absorbance spectra of soil samples. The coefficient of determination (R²), Eq. (1), residual prediction deviation (RPD), Eq. (2), root mean square error (RMSE), Eq. (3), were used to evaluate the prediction ability of the MIR spectroscopy technique are presented in Table 2. The models of calibration and validation showed the prediction potential of MIR [8].

The calibration sample of reference soil data and their respective spectra at an intensity of 10% was not evaluated since it might be too small to represent the total dataset as indicated by [9]. Varying calibration sample intensity between 20 to 80% showed a change in the stability of the model. When the calibration sample intensity increased from 20 to 40% and from 50 to 80%, increases in R² and RPD values of O.C., CEC, and exchangeable Ca were observed; whereas a similar trend was found as the intensity increased from 30 to 40% and 50–80% for total N Change in the intensity from 40 to 50% resulted in a dramatic drop in validation R² and RPD values for O.C., total N, CEC and exchangeable Ca. These statistical values were relatively less variable and stable when calibration intensity increased from 30 and 40% for O.C., CEC, and exchangeable Ca, and between 20 to 30% for total N validation. These results indicated that the model did not improve much by intensifying the calibration sample in the dataset; this might be due to a significant variation of the soil properties that represented a more heterogeneous sample set. According to [10], stable validation results were observed with 20% calibration samples from a diverse sample set of 1,100 for O.C. prediction. Similarly, in their investigation for optimum calibration sample intensity [9], reported that a stable validation result was achieved using 20% of 2,086 topsoil sample sets used for inorganic and organic carbon predictions. Since the intent of modeling is to develop a robust model with the lowest possible representative samples, in this study, 20% calibration sample

| Soil properties | Calibration set (n = 776) | Validation set (n = 3106) |
|-----------------|--------------------------|--------------------------|
|                 | R² | RMSE | RPD | R² | RMSE | RPD |
| pH              | 0.86 | 0.3 | 2.7 | 0.83 | 0.40 | 2.4 |
| Available P (mg kg⁻¹) | 0.31 | 28.2 | 1.2 | 0.25 | 36.1 | 1.2 |
| Exchangeable K (cmol kg⁻¹) | 0.46 | 151 | 1.4 | 0.32 | 159 | 1.2 |
| Exchangeable Ca (cmol kg⁻¹) | 0.95 | 650 | 4.4 | 0.95 | 738 | 3.9 |
| Exchangeable Mg (cmol kg⁻¹) | 0.85 | 235 | 2.6 | 0.84 | 256 | 2.6 |
| Exchangeable Na (cmol kg⁻¹) | 0.25 | 38.2 | 1.2 | 0.52 | 17.8 | 1.5 |
| SO₄⁻² (mg kg⁻¹) | 0.43 | 3.2 | 1.3 | 0.37 | 4 | 1.3 |
| OC (%)          | 0.92 | 0.3 | 3.6 | 0.91 | 0.4 | 3.4 |
| TN (%)          | 0.96 | 0.03 | 4.8 | 0.92 | 0.04 | 3.4 |
| Extractable Fe (mg kg⁻¹) | 0.76 | 38.3 | 2.1 | 0.65 | 39.3 | 1.6 |
| Extractable Zn (mg kg⁻¹) | 0.66 | 1.8 | 1.7 | 0.50 | 2.7 | 1.4 |
| Extractable B (mg kg⁻¹) | 0.59 | 0.2 | 1.6 | 0.6 | 0.2 | 1.6 |
| Extractable Mn (mg kg⁻¹) | 0.48 | 36.17 | 1.4 | 0.42 | 48.3 | 1.3 |
| Extractable Cu (mg kg⁻¹) | 0.65 | 1.1 | 1.7 | 0.55 | 1.2 | 1.5 |
| Extractable Mo (mg kg⁻¹) | 0.27 | 0.6 | 1.1 | 0.35 | 0.55 | 1.2 |
| CEC (cmol kg⁻¹) | 0.94 | 4.6 | 4.0 | 0.92 | 5.4 | 3.5 |

Table 2. Calibration and validation results to predict some soil properties.
intensity was used for total N and 30% for developing a model to predict the rest of the soil's chemical properties.

3.3. Organic carbon and total nitrogen

Soil organic carbon (SOC) and T.N. were predicted well for the validation set (R² = 0.91 and RPD = 3.4) and (R² = 0.92 and RPD = 3.4), respectively, which showed that MIRS had the potential to accurately predict these attributes for agricultural soils of Ethiopia (Table 2 and Figure 2A and B).

According to [11], for agricultural applications, values of RPD greater than 2 indicate that the models provide precise predictions. According to [10] and [12], predictions of R² ranging from 0.65 to 0.75 are satisfactory. The result of this study indicates that the forecast for SOC and T.N. is excellent interns of both R² and RPD. The adequate prediction of SOC and T.N. (R² > 0.75) helps to monitor the soil status for sustainable soil management [10]. The result of the current study confirmed that MIR is the best method to obtain rapid, cost-effective, and accurate information about soil quality since SOC is a crucial attribute of soil quality that influences a variety of biological, chemical, and physical properties of soils [13]. Soil organic carbon acts as both source and sink for nutrients, determines soil chemical, physical and biological status, and strongly correlates with soil nitrogen supply. The result of this study lines with the findings of [14], who reported a good prediction performance of MIR for SOC and T.N. Also [15], reported similar findings for Ethiopian soils.

3.4. Exchangeable calcium and magnesium

The prediction of exchangeable Ca (R² = 0.95 and RPD = 3.9) and Mg (R² = 0.84 and RPD = 2.6) showed good performance (Table 2 and Figure 3). High R² values of 0.95 and 0.84 for Ca and Mg indicate that the model could explain 94% and 84% of the variation between the measured and predicted contents of exchangeable Ca and Mg, respectively. This variation indicates that MIR associated with the PLSR method is the better alternative and cost-effective method to analyze soil exchangeably Ca and Mg in the agricultural soils of the country. This good prediction of Ca and Mg from the current study is useful for assessing the two soil nutrients and allows recommending the use of the MIR scanning approach. In line with the result of this study [19], reported a good prediction performance of MIR for exchangeable Ca and Mg.

3.5. Exchangeable potassium

Exchangeable potassium was not well predicted, with the PLSR model having R² = 0.32 and RPD = 1.2 (Table 2). According to [20], when R² < 0.5 and RPD < 2, the prediction is unreliable. This unreliable result is because K is affected by its high mobility in the soil solution, quickly changing its content, providing less specific prediction results. The result of this study is in line with the findings of [2], who reported that MIR spectroscopy was not suitable to estimate K concentration. Similar MIR spectra of some K containing minerals such as smectites and illites with that of water may be the other reason for the poor prediction performance of the PLSR model for K [20].

3.6. Soil reaction (pH)

The prediction of soil pH showed precise prediction performance having R² = 0.83 and RPD = 2.4, respectively (Table 2). This indicates MIR-PLSR method is an alternative method to analyze soil reaction for agricultural soils of Ethiopia. However, soil pH or proton activity is not expected to have a direct spectral response; the present study and many other studies still could be more or less well predicted in several cases. In general, MIR techniques can promote rapid measurement of soil properties.
pH to determine the degree of soil acidity or rate of lime required to achieve an acceptable pH.

### 3.7. Available P, SO₄⁻²–S, and exchangeable cation capacity

Available P was predicted with $R^2 = 0.25$ and RPD 1.2, while SO₄⁻²–S was predicted with $R^2 = 0.37$ and RPD 1.3 in this study (Table 2). This implies validation of the model in terms of P and S determination was poor. Similar results were reported by [11, 21, 22, 23], and [8] reported similar findings. This insufficient validation for P and S is due to their low concentration in the soil. Disagreeing with the result of this study [24], and [14] reported the excellent prediction performance for P and S values of $R^2 = 0.85$ and 0.93, respectively, from the Canadian landscape. In contrast [25], from India reported an $R^2$ value of 0.90.

An excellent prediction was obtained for CEC ($R^2 = 0.92$ and RPD = 3.5). Both high $R^2$ and RPD values for the validation of this method indicate that CEC was predicted well (Table 2 and Figure 4). This result is in agreement with that of [25, 26], and [27], who reported that MIR provides good predictions for CEC of soils collected from India, Canada, and the U.S., respectively. Similarly [28], showed good prediction accuracy for CEC of soil samples collected from southeastern Australia with $R^2 = 0.84$. The result of the current study showed improvement in $R^2$ value than the previous studies. Therefore, this good modeling of CEC indicates that the MIR method can avoid many constraints associated with traditional laboratory analysis.

### 3.8. Prediction of micronutrients

The MIR-PLS model showed different performance levels on micronutrient prediction. Relatively better performance ($R^2 = 0.65$ and RPD = 1.7) was found for extractable Fe among the rest of extractable micronutrients (Table 2). This may be associated with the ability of Fe to form a complex with SOM and clay content, which are spectrally active in MIR. However, the model is unreliable for predictions of most soil micronutrients. $R^2$ values for Zn, B, Mn, Cu and Mo were found to be 0.5, 0.6, 0.42, 0.55, and 0.35, respectively, for their validation set, indicating poor prediction performance. Generally, the result of this study revealed that the use of MIRS for predicting micronutrients requires further investigation and may require the development of another model.

### 3.9. Comparison of MIR spectra predicted and measured soil properties of study areas

The means and ranges of the soil properties measured and predicted are shown in Table 3.

Soil pH values predicted from MIR spectra varied from 5.32 to 8.80 with a mean value of 5.98, but water measured soil pH ranged from 4.6 to 8.5 with a mean value of 6.16 (Table 3). Both predicted and measured pH values showed very strongly acidic to moderately alkaline soil reactions. The means of both measured and indicated pH categorized to moderately acidic(5.6–6.6) as per rating of [30] for Ethiopian agricultural soils. MIR spectra predicted soil pH was significantly and positively correlated with pH measured by the wet chemistry method ($r = 0.56$, $P < 0.001$). Based on this statistical analysis, there was good agreement between MIR spectra prediction and wet chemistry analysis of pH. This finding is in agreement with reports of [21].

Available P predicted from MIR spectra ranged between 1.60 and 17.02 mg kg⁻¹ with a mean value of 4.49 mg kg⁻¹, while Mehlich-III extracted available P that was measured by ICP ranged between 0.00 and 267 mg kg⁻¹ with a mean value of 14.56 mg kg⁻¹ (Table 3). Based on MIR spectra predicted available P data, the P status of all soils was categorized as deficient level, but ICP measured available P data showed very low to very high status based on rating suggested by [29]. This justified that MIR spectra prediction of available P had no good agreement with wet chemistry analysis; this finding agrees with the results of [21]. However, MIR spectra predicted available P was significantly and positively correlated with measured available P ($r = 0.39$). Therefore, calibration between two methods could be necessary for future studies in order to efficiently develop predictive models available.

Exchangeable Ca and Mg predicted from MIR spectra varied from 2.28 to 51.5 and 1.24 to 6.55 Cmol(+) kg⁻¹ with mean values of 9.05 and 2.32 Cmol(+) kg⁻¹, respectively. In comparison, Mehlich 3 extracted Ca and Mg, which ICP measured varied from 2.32 to 33.24 and 0.68 to 6.33 Cmol(+) kg⁻¹ with mean values of 11.05 and 2.32 Cmol(+) kg⁻¹, respectively. In this study, predicted and measured values of Ca and Mg were significantly and positively correlated ($r = 0.56$ and 0.46), respectively. This indicates that MIR spectra prediction of exchangeable Ca and Mg had good agreement with wet chemistry analysis.

Exchangeable K and Na predicted from MIR spectra varied from 0.05 to 1.09 and 0.06 to 0.88 Cmol(+) kg⁻¹ with mean value of 0.62 and 0.39 Cmol(+) kg⁻¹, respectively whereas Mehlich-3 extracted K and Na

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**Table 3. Descriptive statistics of measured and MIR spectra predicted soil attributes.**

| Parameters | Range | Predicted | Mean | Predicted |
|------------|-------|-----------|------|-----------|
|            | Measured | Predicted | Measured | Predicted |
| pH         | 4.64–8.50 | 5.32–8.80 | 6.16 | 5.98 |
| P (mg kg⁻¹) | 0.00–267 | 1.60–17.02 | 14.56 | 4.49 |
| S (mg kg⁻¹) | 3.00–27.00 | 1.86–13.54 | 7.54 | 9.92 |
| Ca (Cmol(+)) kg⁻¹ | 2.32–33.24 | 2.28–51.50 | 11.05 | 9.05 |
| Mg (Cmol(+)) kg⁻¹ | 0.68–6.33 | 1.24–6.55 | 2.32 | 2.49 |
| K (Cmol(+) kg⁻¹) | 0.29–6.05 | 0.05–1.09 | 1.34 | 0.62 |
| Na (Cmol(+) kg⁻¹) | 0.06–3.40 | 0.06–0.08 | 0.32 | 0.39 |
| Fe (mg kg⁻¹) | 46.84–212.23 | 37.80–231.35 | 141.06 | 138.65 |
| Zn (mg kg⁻¹) | 0.90–47.72 | 0.31–4.80 | 9.11 | 3.04 |
| B (mg kg⁻¹) | 0.02–2.03 | 0.05–0.77 | 0.40 | 0.33 |
| Mn (mg kg⁻¹) | 26–330 | 37.02–159.45 | 143.24 | 102.6 |
| Cu (mg kg⁻¹) | 0.50–3.40 | 0.21–9.11 | 1.20 | 1.13 |
| Mo (mg kg⁻¹) | 2.21–18.71 | 0.005–4.12 | 9.15 | 1.05 |
measured by ICP varied from 0.29 to 6.05 and 0.06 to 3.40 Cmol (+)kg⁻¹ with mean value of 1.34 and 0.32 Cmol (+)kg⁻¹, respectively. The correlation result showed that MIR spectra predicted exchangeable K and Na was non-significant but positively correlated. This result implies that MIR spectra prediction did not accurately predict exchangeable K measured by the wet chemistry method. This finding is in agreement with that of [30].

Except for Mn, all extractable micronutrients were correlated positively and significantly with their contents extracted by the Mehlich-3 method. On the other hand, a very strong correlation was observed for extractable B with an r-value of 0.99** followed by Zn, Mo, Cu, and Fe with the r-value of 0.95**, 0.74**, 0.59** 0.48**, respectively.

4. Conclusions

This study showed the possibility of MIR spectra combined with the PLSR method to determine soil properties such as O.C., TN, exchangeable Ca and Mg, CEC, pH, and extractable Fe. The predicted contents were significantly and positively correlated for soil samples collected from Kedida Gamela, Kechabira, and Damboya woredas. However, the poor prediction was observed for available P, SO4-2-S, and all extractable micronutrients except Fe.

Declarations

Author contribution statement

Alemu Lelago: Performed the experiments; Wrote the paper.
Mesfin Bibiso: Analyzed and interpreted the data; Wrote the paper.

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Data availability statement

Data will be made available on request.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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