



Short communication

Near infrared spectroscopy—A tool for chemical properties and organic matter assessment of afforested mine soils

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ABSTRACT

The objectives of this study were to assess chemical and microbial properties of different post-mining barrens afforested with Scots pine (*Pinus sylvestris*) and to evaluate the potential of near infrared spectroscopy (NIRS) to predict these properties in the reclaimed soils. The samples were collected from four sites at different substrates and measured for the contents of organic C (C_{org}), total N (N_t), C_{org} -to- N_t ratio, dehydrogenase activity, contents of humified C, humic acid C and fulvic acid C, cation exchange capacity, exchangeable acidity, total exchangeable bases (TEB) and base saturation. The spectra of samples were recorded between 400 and 2500 nm. A modified partial least squares method with cross-validation was used to develop calibration models for prediction of chemical and microbial properties of the samples from their spectra. There were significant differences in the contents of C_{org} , N_t , dehydrogenase activity, pH and sorption properties between the mine soils. The samples from the studied sites had different spectral characteristics suggesting differences in chemical composition of their organic matter. “Excellent” predictive models (coefficient of correlation between the predicted and reference values ≥ 0.90) based on NIR spectra were developed for N_t , C_{org} -to- N_t ratio, humified C, humic acid C and exchangeable acidity. For other soil properties (except the TEB), “satisfactory” ($r = 0.80$ – 0.90) models useful for fast screening were developed. The results indicate the potential of NIRS to predict several properties of the reclaimed mine soils.

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1. Introduction

Extraction of minerals and fossil fuels may disturb soils over large areas, resulting in the need for restoration. Afforestation is a common strategy used to reclaim post-mining barrens. Post-mining barrens are often built of infertile materials extracted from large depths. They lack soil organic matter, exhibit extremely low microbial activity and have disadvantageous chemical and physical properties (Baldrian et al., 2008). Therefore, only some tree species are able to grow on these materials. In central Europe, Scots pine (*Pinus sylvestris*) is commonly used for afforestation of post-mining barrens (Baumann et al., 2006; Kuznetsova et al., 2010; Pietrzykowski, 2010; Pietrzykowski and Socha, 2011). This is because Scots pine has low nutritional requirements and is able to survive on acid and dry soils (Pietrzykowski, 2010).

The vegetation introduced onto the post-mining barrens should contribute to soil quality improvement. However, the effect of planted vegetation may differ depending on the properties of soil parent material. Assessment of the reclaimed mine soil quality requires a number of physical, chemical and biological soil properties to be measured. These include C and N contents, characteristics of the accumulated organic matter, soil acidity, cation exchange capacity and base saturation as well as soil microbial properties (Haering et al., 1993; Šourková et al., 2005; Pietrzykowski and Krzaklewski, 2007). The reclaimed mine soils may cover large areas and their spatial heterogeneity is often very high (Buczko et al., 2001; Hüttl and Weber, 2001). Therefore, although simple and inexpensive analytical methods may be required, the mine soil quality assessment must include a large number of samples.

Near infrared spectroscopy (NIRS, 750–2500 nm) is an analytical technique widely known for its rapidity, simplicity, cost-effectiveness and ability to predict numerous sample properties at one time (Foley et al., 1998). This method has been applied to measure several chemical and microbial natural soil properties (Ludwig and Khanna, 2001; Coûteaux et al., 2003; Viscarra Rossel et al., 2006). However, NIR spectroscopy has rarely been used for

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the determination of various properties of mine soils developed on different soil substrates. [Schimann et al. \(2007\)](#) used this method to evaluate recovery of microbial functions during restoration of soils destroyed by gold mining in French Guiana and reported promising results of the NIRS application. However, they studied only four soil properties (C and N contents, substrate induced respiration and denitrifying enzyme activity).

The objectives of our study were to compare several chemical and microbial properties of different mine soils afforested with Scots pine and to test the usefulness of NIRS to predict chemical and microbial properties in a diverse set of mine soils. The selected properties included C and N contents, characteristics of soil organic matter (content of humic C, humic acid C and fulvic acid C), characteristics of soil cation exchange properties (cation exchange capacity, exchangeable acidity, total exchangeable bases and base saturation) and dehydrogenase activity. The selected properties are of major interest for mine soil quality assessment, which is an important factor for the general success of reclamation efforts and ecosystem reconstruction.

2. Materials and methods

2.1. Experimental sites and sampling

The samples were collected from four post-mining sites at different substrates reclaimed for forestry and covered by 20–40 year-old Scots pine (*P. sylvestris* L.) stands in central and southern Poland. The sites were located at a maine waste dump at a lignite mine Bełchatów (BEL; 19°25' E; 51°13' N), a maine waste dump at the hard coal mine Smolnica (SMOL; 18°31' E; 50°15' N), a sand-quarry Szczakowa (SZCZ; 19°25' E; 50°14' N) and a maine waste dump at the open-cast sulphur mine Piaseczno (PIAS; 21°34' E; 50°33' N). General information and some characteristics of the sampled sites are given in [Table 1](#).

Eight soils were sampled at each experimental post-mining site of a 10 m × 10 m square ($n=8$). Five subsamples were taken at the corners and in the middle of an experimental site and mixed representative samples were prepared. The subsamples were taken from the initial AC horizon (0–8 cm depth, where organic-mineral horizons exhibited some features of parent material). To compare properties of reclaimed mine soils with the natural forest soils, a sample was taken from the A horizon of natural pine forest soils near the sampled mine soil. These samples ($n=$) were used as natural reference soils (NAT). In total there were 36 samples.

The soil samples were divided into two parts. One part was immediately sieved (2 mm mesh size) and stored field-moist at 4 °C for microbial measurements; the other part was air-dried (105 °C), sieved (2 mm mesh size), finely ground and used for chemical analyses.

2.2. Physical and chemical analysis

The soil texture of the samples was determined hydrometrically ([Ostrowska et al., 1991](#)). The contents of organic C (C_{org}) and total N (N_t) were measured by dry combustion using the Leco CNS 2000 analyser. Samples containing $CaCO_3$ were washed in 10% HCl to remove carbonates prior to the determination of C_{org} . To measure exchangeable acidity the samples (40 g) were treated with 1 M $Ca(CH_3COO)_2$ using 1:2.5 soil/solution ratio. Suspensions were shaken for 1 h, filtered and titrated with 0.1 M NaOH to pH = 8.2. The exchangeable acidity was calculated from the amount of base used and was expressed in $cmol_{(+)} kg^{-1}$ ([Ostrowska et al., 1991](#)).

Total exchangeable bases (TEB) were calculated as a sum of base cations (Na^+ , K^+ , Ca^{2+} , Mg^{2+}) extracted in 1 M CH_3COONH_4 (pH = 7).

The concentrations of Na^+ , K^+ , Ca^{2+} , Mg^{2+} in the extracts were measured using atomic absorption spectrometry. Cation exchange capacity (CEC) was defined as the equivalent sum of TEB and exchangeable acidity; base saturation (BS) was defined as the sum of base cations as a percentage of CEC.

2.3. Fractionation of soil organic matter

Soil organic matter fractionation was done using a modified method of [Kononova and Belchikova \(1960\)](#). Briefly, 5 g of soil was shaken in 100 ml of 0.1 M NaOH + 0.1 M $Na_4P_2O_7$ for 4 h. The dark-colored supernatant solution was separated from the residual soil by centrifugation at $3000 \times g$ for 30 min. The solution contained a humified C comprised of humic acid C and fulvic acid C. A subsample of the solution was acidified to pH 1.0–1.5 with concentrated H_2SO_4 . Precipitated humic acids were separated by centrifugation. The C content in the alkaline solution (humified C), the acidified solution (fulvic acid C) and in the precipitated humic acids (humic acid C) was determined by wet combustion using $K_2Cr_2O_7$ ([Tiurin, 1965](#)).

2.4. Microbial analysis

Dehydrogenase activity (DHG) in the soils was determined following the method of [Casida et al. \(1964\)](#) by reduction of 2,3,5-triphenyltetrazolium chloride (TTC). The soil samples (5 g) were amended with 3 ml of 0.2 M $CaCO_3$ and 1 ml of 3% TTC and incubated for 24 h at 37 °C. The triphenyl formazan (TPF) formed was extracted from the reaction mixture with methanol and assayed at 485 nm.

2.5. Sample treatment and near infrared spectral reflectance measurements

The NIR-reflectance was recorded at 2 nm intervals between 400 and 2500 nm (Vis – NIR region) in the dried and ground samples using a Foss NIRSystems spectrometer (Silver Spring, USA). Each sample was scanned once, thoroughly mixed and scanned a second time. The final spectrum was a mean of both scans. Absorbance values ($A = \log 1/\text{reflectance}$) were used for the data transformation and statistical analysis.

2.6. Statistical analysis

One-way ANOVA was used to compare individual chemical and microbial properties in the studied mine and natural soils. The Tukey's honestly significant differences (HSD) test for multiple comparisons was used if significant differences were found ($p < 0.05$). The data were log-transformed to fulfill the assumption of normality.

Principal components analysis (PCA) based on the second derivatives of the spectra was used to compare spectral characteristics of the samples from different sites (WinISI II – version 1.02 software). The derivatives were calculated at gaps of four data points and smoothing was done over length segments of four data points. Derivation of spectra was done in order to enhance their features ([Tillmann, 2000](#)). To remove scatter, standard normal variate and detrend (SNVD) procedures were applied to the spectra prior to the PCA (WinISI II – version 1.02 software). The standard normal variate procedure scales each spectrum to have a standard deviation of 1.0 and the detrending removes the linear or curvilinear trend of increase in absorbance data. The PCA reduced the number of variables describing each spectrum to a smaller number of new variables (PCs).

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