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Research article

Mid-infrared spectroscopy for rapid assessment of soil properties after land use change from pastures to *Eucalyptus globulus* plantations

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ABSTRACT

There is an increasing demand for rapid and cost effective techniques to accurately measure the effects of land use change on soil properties. This study evaluated the ability of mid-infrared spectroscopy (MIRS) coupled with partial least squares regression (PLSR) to rapidly predict soil properties affected by land use change from agriculture (mainly pasture) to *Eucalyptus globulus* plantations in south-western Australia. We measured total organic carbon (TOC), total nitrogen (Total N), TOC/Total N (C/N ratio), microbial biomass carbon (MBC), microbial biomass nitrogen (MBN), and total phosphorus (Total P). The PLSR calibration models were developed using mid-infrared (MIR) spectra (4000 to 450 cm^{-1}) and square root transformed measured soil data (n = 180) from 23 paired pasture and *E. globulus* plantation sites representing the soils and climate of *E. globulus* plantation estates in south-western Australia. The calibration models for TOC, Total N, C/N ratio and Total P showed excellent correlations between measured and predicted data with coefficient of determination (R^2) exceeding 0.91 and minimum root-mean-square error (RMSE) of calibration [TOC (R^2 = 0.95, RMSE = 0.36), Total N (R^2 = 0.96, RMSE = 0.10), C/N ratio (R^2 = 0.92, RMSE = 0.14) and Total P (R^2 = 0.91, RMSE = 0.06)]. The calibration models had reasonable predictions for MBC (R^2 = 0.66, RMSE = 0.07) and MBN (R^2 = 0.63, RMSE = 0.06). The calibrated models were validated using soils from 8 independent paired pasture and *E. globulus* sites (n = 64). The validated predictions were excellent for TOC (R^2 = 0.92, RMSE = 0.40) and Total N (R^2 = 0.91, RMSE = 0.12), but less so for C/N ratio (R^2 = 0.80, RMSE = 0.35), MBC (R^2 = 0.70, RMSE = 0.08) and Total P (R^2 = 0.75, RMSE = 0.12). The results demonstrate the potential of MIRS-PLSR to rapidly, accurately and simultaneously determine several properties in land use change affected soils.

1. Introduction

South-western Australia is one of the major forest plantation growing regions in Australia, comprising one-third of Australian hardwood plantations of more than 301,000 ha (Gavran, 2013). Since the mid-1990s, there has been a significant expansion of hardwood plantations, predominantly *Eucalyptus globulus*, mostly on land with annual rainfall greater than about 600 mm and previously cleared for grazing agriculture (O'Connell et al., 2003). These plantations are generally managed for pulpwood under multiple rotations of short duration (approximately 10 years). Tree growth in the first rotation benefits from the enhanced soil nitrogen (N) and phosphorus (P) status resulting from agriculture including N inputs from leguminous pastures and P fertilisers. However there is a concern that soil fertility may decline under these hardwood plantations, affecting sustainable production (Mendham et al., 2002b).

Several studies have been conducted to understand the effects of converting pasture to plantation in south-western Australia, which included measurements of total soil carbon, permanganate oxidisable C, total soil N, potentially mineralisable N, total P and microbial biomass (e.g., Mendham et al., 2002b; Mendham et al.,

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2003, 2004; O'Connell et al., 2003). Measurement of these soil properties is time-consuming and expensive using conventional analyses. Moreover, the costs limit measurements for site-specific management or monitoring purposes. Thus there is a strong demand for easy-to-use and inexpensive methodologies to assess soil properties under land use change.

Infrared spectroscopic techniques offer a rapid, accurate, non-destructive and low-cost alternative for estimating many soil properties (Janik et al., 2007; Viscarra Rossel et al., 2006; Viscarra Rossel et al., 2008). In addition, these methods use simple sample pre-treatment, and do not require the use of potentially environmentally harmful chemicals (Viscarra Rossel et al., 2008). A further advantage is that infrared spectroscopic techniques have been shown to be sensitive to specific molecular vibrations of both organic and mineral components of the soil, making them potentially useful for assessing and monitoring soil quality and soil function (Janik and Skjemstad, 1995; Pirie et al., 2005; Viscarra Rossel et al., 2008). In the electromagnetic spectrum, the mid-infrared (MIR) wavelength region (4000–400 cm^{-1}) is characterised by fundamental molecular vibrations associated with particular chemical functional groups (e.g. aliphatic, amidic, aromatic and carboxylic) which can provide specific information on SOM (Viscarra Rossel et al., 2008; Calderón et al., 2011; Tatzber et al., 2011). Several soil physical, chemical and biological properties have been successfully predicted using mid-infrared spectroscopy (MIRS), including sand, clay, pH, EC, elemental compositions, organic C, inorganic C, total N and microbial biomass (e.g. Ludwig et al., 2008; Soriano-Disla et al., 2014; Vohland et al., 2014; Waruru et al., 2014).

A combination of spectroscopic measurements and partial least squares regression (PLSR) has been well demonstrated to qualitatively and quantitatively analyse a range of chemical compounds in soils (e.g. Wold et al., 1984; Stark et al., 1986; Holmgren and Nordén, 1988). Studies using MIRS-PLSR techniques have reported good predictive capability for total organic C (TOC) with $R^2 > 0.93$ and RMSE of 0.10–1.73 g kg^{-1} in 1.00–104 g C kg^{-1} of soil (e.g. Madari et al., 2006; Janik et al., 2007), for total N with $R^2 > 0.80$ and RMSE of 0.14–0.18 g kg^{-1} in 0.04–5.60 g N kg^{-1} of soil (Madari et al., 2006; Ludwig et al., 2008), C/N ratio with $R^2 > 0.84$ (e.g. Ludwig et al., 2008; Soriano-Disla et al., 2014) and reasonable predictions for microbial biomass with $R^2 > 0.60$ (e.g. Ludwig et al., 2008) in various agricultural, forest and native vegetation soils. Viscarra Rossel et al. (2008) reported robust predictions while validating soil organic C, total N and other soil properties, however they found a poor prediction for total P in both calibration and test validation. Although many studies have reported on MIRS predictions of soil chemical properties such as C, N and P (e.g. Madari et al., 2006; Janik et al., 2007), few have attempted prediction of soil biological properties (Vohland et al., 2014).

This study focussed on the feasibility of MIRS techniques for rapidly and inexpensively predicting a suite of soil chemical and biological parameters to reliably inform wise management of land use changed sites from pasture to *Eucalyptus globulus* plantations, e.g. nutrient management, harvest residue conservation. The key soil properties explored in the study were total organic C, total N, C/N ratio, microbial biomass C, microbial biomass N, and total P, and we hypothesised that mid-infrared spectroscopic techniques can rapidly predict these soil properties. The objectives of the study were a) to develop MIRS-PLSR calibration models for the studied soil properties; b) determine the contribution of specific functional groups and molecular vibrations to calibration models, and c) validate the calibration models using an independent soil set.

2. Materials and methods

2.1. Study sites and soils

The study used 31 paired sites of *E. globulus* plantations and adjacent pastures in south-western Australia. These sites were paired based on soil morphology and total N and P concentrations as an indicator of site fertiliser history. Detailed description of the sites, soils and climate characteristics were reported by O'Connell et al., 2003 and Mendham et al., 2003. The sites were selected as representative of the 600 mm–1300 mm yr^{-1} range in rainfall and in soil properties of the *E. globulus* plantation growing areas in south-western Australia. Pastures were established between 35 and 80 years prior to the study, after clearing of the natural forest (*E. marginata, E. calophylla, E. diversicolor*). The pastures contained a mix of sown annual species including subterranean clover (*Trifolium subterraneum*), capeweed (*Arctotheca calendula*), and grasses (*Lolium rigidum, Hordeum leporium, Bromus diandrus*). Soil fertility was enriched through N inputs from leguminous species and annual applications of P fertilisers (typically applied as superphosphate) and trace elements to increase agricultural productivity. The *E. globulus* plantations were established on pastures, and soils were sampled in the first rotation at ages 7–11 years (Mendham et al., 2004).

In each of the pasture and plantation land uses, soils (0–10 cm depth) were randomly sampled within four contiguous replicate plots (each 4 × 10 m) along a 40 m transect located at least 20 m from the boundary with the other land use. Six soil cores per plot were randomly collected and aggregated, resulting in 4 samples per land use per site. The samples were transported in cooled-insulated containers to the laboratory and stored at 4 °C until processing. Subsamples were prepared, (i) field moist, further moistened to −10 kPa matric potential for microbial biomass analysis; and (ii) air dried (<40 °C) and sieved (<2 mm). Subsamples of the <2 mm soils were finely ground for 3 min in a 100 mm diameter steel ring and puck mill (Rocklabs, Auckland, New Zealand) for chemical analysis and acquisition of infrared spectra.

2.2. Soil analyses

Total organic C (TOC, as total C in the acidic, non-calcareous soils) and total N (Total N) were determined by Dumas combustion of finely ground soil samples using a Euro EA Elemental Analyser (HEKAtech GmbH, Germany), and C/N ratio was calculated as TOC/Total N. Total P was determined on sulfuric acid/hydrogen peroxide digested samples at 360 °C using colourimetry in a flow injection auto analyser (Rayment and Higginson, 1992). Microbial biomass was measured by chloroform fumigation-extraction method (Sparling and West, 1988). Soil moisture content was increased to −10 kPa matric potential to ensure effective fumigation. Fumigated (48 h in evacuation chamber) and control (unfumigated) samples were extracted in 0.5 M K_2SO_4 solution and aliquots analysed for C and N. Microbial biomass carbon (MBC) was calculated as the difference in Walkley-Black C (Walkley, 1947) between fumigated and control samples divided by kEC factor (0.3), and the microbial biomass nitrogen (MBN) was calculated as the difference between total Kjeldahl N in fumigated and control samples divided by kEN factor of 0.37 (Mendham et al., 2002a, 2002b). The kEC and kEN factors accounted for the efficiency of extraction of microbial biomass C and N respectively in Western Australian soils (Sparling and Zhu, 1993).

2.3. Infrared spectroscopy and analysis

Diffuse reflectance infrared spectra (7800–450 cm^{-1} at 4 cm^{-1}