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Fertiliser characterisation using optical and electrical impedance methods

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ABSTRACT

This paper addresses the problem of fertiliser characterisation using optical and electrical impedance methods. Comparative analysis was performed to estimate the methods effectiveness for quantitative and qualitative characterisation of water diluted fertiliser. Characterisation using optical method within the deep ultraviolet range indicates the variability of features that was not observed when using the impedance method. The combination of both methods showed potential for more accurate qualitative analysis than each method alone. Finally, both methods showed good sensitivity to fertiliser concentration variation that was possible to fit with a linear function for optical spectroscopy ($R^2 = 0.95$) and an exponential function for the impedance method ($R^2 = 0.99$).

1. Introduction

The electrical properties of soil have been widely used in agriculture (Albrektiene et al., 2012; Abdelgwad and Said, 2016; Rao et al., 2007; Kiti and Crnojevi-Bengin, 2013; Han et al., 2015; Wang et al., 2016; Li et al., 2017) to provide complex information about its physical and chemical properties (Rao et al., 2007; Kiti and Crnojevi-Bengin, 2013). The literature indicates a common use of optical and impedance methods for soil quantitative and qualitative analysis, that may be used to design low-cost portable sensors for real-time application, i.e. on-the-go soil sensors (Bah et al., 2012; Pajares, 2011). Pandey et al. (2013) used measurements averaging to reduce the distribution of acquisition factors. Environmental factors, such as the amount of water, ambient light, bulk density and temperature make it challenging to fully understand and provide a connection between measurements and soil properties. Therefore, a number of strategies with different degrees of success have been proposed for the environmental effects and soil properties prediction in a field (Pittaway et al., 2013; Abdelgwad and Said, 2016; Kulkarni et al., 2014). This study investigates the fertilisers characterisation as is an important quality measuring factor that influence the amount of macro and micronutrients in soil.

A certain technique that may be suitable for all soil components characterisation does not exist (Kim et al., 2016; Yokota et al., 2007; Yasrebi et al., 2004). Li et al. (2016) and Anggoro and Irman (2012) observed a larger impact of salt on the soil measurements than water, while carbon does not have significant influence when using impedance measurement technique. Nevertheless, carbon is easily observed using optical spectroscopy (Stevens et al., 2013; Mohamadi, 2016; Pittaway and Eberhard, 2014). Literature indicates that the optical transmission measurement in the UV range is sufficiently sensitive to the nutrients concentration change, organic matter and soil with large amount of water (Edwards et al., 2001; Albrektiene et al., 2012; Pittaway et al., 2013). Ji et al. (2015) proposes absorbance measurement of soil with volumetric water content between 40% and 50% to reduce the surface influence effect on measurements pointing out the importance of the water. Therefore, fertiliser in this study was diluted in deionized water for better observation of the relevant information when using optical and electrical impedance methods.

First of all, the methods sensitivity to the fertiliser concentration was investigated. Cavka et al. (2014) indicate various degree of curve-fit models effectiveness for water content prediction in a frequency domain. Wang et al. (2016) reported that the soil impedance changes a lot with the frequency below 100 kHz that confirms with other studies (Son et al., 2009; Sternberg and Levitskaya, 2001). The frequency range for impedance measurement in this study was selected between 100 Hz and 100 MHz. The correlation between fertiliser concentration under 1% and measurements have been obtained using a polynomial fitting procedure and then validated using a cross-validation function R^2 (James et al., 2014).

The second part of paper addresses the problem of the fertiliser identification. Edwards et al. (2001) observed strong absorption spectrum of nitrate between 200 nm and 210 nm, while Kim et al. (2016) monitored the absorbance of the total organic carbon in a water between 200 nm and 300 nm. The authors indicated a better agreement with measurements when the total organic carbon concentration is high. Albrektiene et al. (2012) indicate a high correlation between total

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Table 1
Fertilisers used for analysis.

Abbriviations	Fertiliser name	Components
$F(K_2O)$	Potassium sulphate	K_2O–50%
$F(P_2O_5)$	Triple super phosphate	P_2O_5–46%
$F(P_2O_5–CaO)$	calcium phosphate	P_2O_5–26%, CaO–40%
$F(MgO)$	Magnesium Sulphate	MgO–25%, SO_3–50%
$F(N)$	Urea	N–46%
orgCHKN	Organic fertiliser	Chicken manure (organic mass minimum 70%)
orgHRS	Organic fertiliser	Horse manure (organic mass minimum 70%)

organic carbon and light absorbance at 245 nm. The method published by the American Public Health Association (Association et al. (1992)) suggests a two-wave-length approach for water containing dissolved organic matter. The absorbance was measured at 220 nm and at 275 nm where nitrate does not absorb light. Thus, the UV range between 200 nm and 400 nm was of particular interest of this study.

The obtained measurements were normalized to enable better observation of their variation and used to identify the relevant features for fertiliser characterisation.

2. Materials and methods

To eliminate the external factors influence on the measurements (Abdelgwad and Said, 2016; Watson and Zielinski, 2013; Li et al., 2016), the ambient temperature (near 22° C) and relative humidity (under 50%) were kept constant.

2.1. Sample preparation

Fertilisers selected for analysis are presented in the Table 1. To investigate their influence on soil, the fertilisers were diluted in deionized (DI) water and then mixed with air-dried soil in various concentrations. Chemical analysis was then performed by a certified laboratory at the Agriculture Institute of Slovenia (Agrecalture Institute of Slovenia, 2018). The results of analysis are shown in Table 2 indicating entirely different fertiliser influence on the soil chemical properties. The correlation between added fertiliser concentration and nutrients change may be observed.

Brief nutrients level classification based on literature review (Čop et al., 2010; Sirsat et al., 2017) and results in Table 2 indicate that 0.1% fertiliser concentration in the soil sample results in a high level of nutrients. Medium level of nutrients in soil would correspond to roughly 0.05% fertiliser concentration. Using these observations, 0.05% and 0.1% fertiliser concentrations were used for analysis.

Solution with 0.1% fertiliser concentration was prepared by mixing 20 g of DI water with 20 mg of fertiliser until a uniform texture is reached. When the fertiliser was fully diluted, the mixture was filtered using LLG-Plain disc filter paper (Lab Logistics Group GmbH, Cat. No.9.045 840) to remove remaining solids. Fig. 1 shows an example of the prepared solutions placed in plastic cuvettes. It can be seen that there is no visual difference between them.

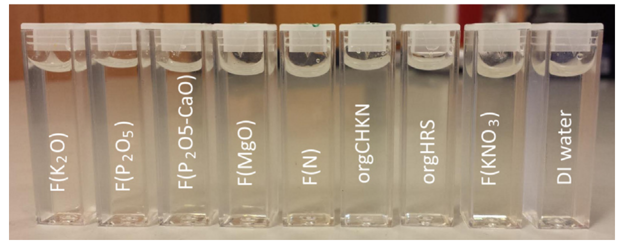

Fig. 1. Photography of the deionized (DI) water with various fertilisers prepared for laboratory tests.

2.2. Electrical impedance method

The impedance measurement set-up is shown in Fig. 2 that includes a Network Analyzer HP3377A connected to the personal computer (PC) for data storage and processing using a USB-GPIB 82357B converter. The measuring process was controlled using a graphical user interface developed in Matlab software (The Mathworks Inc., 2015). The photograph shows the impedance sensor designed for water impedance measurement placed in cylindrical sample holder. Each measurement was performed using 40 frequency steps between 100 Hz and 100 MHz to provide good fit of the impedance signal over whole frequency range.

Fig. 3 shows impedance magnitudes and phases that correspond 200 g of air-dried sieved soil samples with different water content. The plots demonstrate similar behaviour of the impedance magnitudes and phases over the frequency range having various degree of deviation. Therefore, impedance behaviour may be characterised using impedance magnitude or impedance phase alone. For simplicity, only the impedance magnitude was used in this analysis.

Additional measurements were performed to investigate the correlation between measurements of fertiliser in soil and in water. The representative examples of the impedance measurement are shown in Fig. 4, where the same fertilisers were added into 200 g of soil and into 200 g of DI water, respectively. It indicates similar behaviour between measurements that may be used to divide soil analysis into two subproblems: fertiliser behaviour characterisation in water and correlation estimation between measurements of fertilised soil and fertilised water.

The measurement results shown in Figs. 3 and 4 indicate that fertiliser in soil as well as water in soil results in an impedance magnitude deviation compared to the air-dry soil without fertiliser. As expected, the largest deviation is obtained for the soil with the highest moisture and fertiliser content. The fertiliser showed a significant influence on the soil impedance measurements without any specific curvature change and, therefore may reduce the accuracy of the soil moisture prediction using the impedance method alone.

The impedance magnitude normalization was performed by dividing it with the impedance magnitude of DI water as a known

Table 2
Chemical analysis of the soil samples after fertiliser addition.

Soil samples	pH in KCl mg/100 g	P_2O_5 mg/100 g	K_2O mg/100 g	Mg mg/100 g	OM %	N %
Soil	7.2	3.4	6.4	22	4.2	0.25
Soil + 0.05%$F(P_2O_5)$	7.2	14	6.4	22	4.2	0.25
Soil + 0.05%$F(K_2O)$	7.2	4	18	25	4	0.25
Soil + 0.1%$F(P_2O_5)$	7.1	35	8.2	24	4.4	0.25
Soil + 0.1%$F(K_2O)$	7.1	4.2	44	23	4.2	0.25
Soil + 0.1%$F(P_2O_5–CaO)$	7.5	19	7.1	22	4.2	0.25
Soil + 0.1%$F(MgO)$	7.2	4.0	6.4	26	4.2	0.25
Soil + 0.1%*orgCHKN*	7.2	3.5	6.4	23	4.3	0.25