

NPK DETECTION SPECTROSCOPY ON NON-AGRICULTURE SOIL

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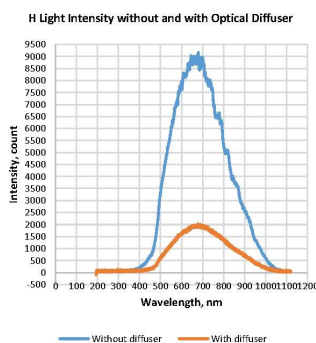
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Graphical abstract



Abstract

Soil is a medium for plant roots to grow, absorb water and necessary solutes for growth. Soil macronutrient testing is helpful for determining the nutrients content in soil before applying fertilizer for quality and process controls of agricultural produce and soil fertility. Spectroscopy is an emerging technology which is rapid and simple has been widely used in agricultural and food analysis processes. The capability of spectroscopy to characterize material from the transmission or absorbance has been used in this paper to measure nitrogen (N), phosphorus (P) and potassium (K) content in non-agriculture soil. The paper details preliminary characterization of soil spectroscopy with a Deuterium-Halogen light source and Ocean Optic spectrometer to measure the absorbance level of the macronutrients. The extracted nutrients were mixed with the colour reagent and specific colored solution was developed. Two soil samples have been employed for the experimental characterization, which are mud flood and kaolin. The result shows that high absorbance level of N at 450 nm in wavelength, P at 750 nm for both samples. The absorbance level of K was measured high at 500nm for mud flood and 450nm for kaolin. In addition, the tested macronutrients give similar wavelength of peak absorbance level at 970 nm for both samples. For future works, the optical measurements will be implemented using visible and near infrared LED and the photodetector in order to replace the spectrometer usage for soil spectroscopy. This would lead to achieve the primary objective of this research in developing a simple and low cost spectroscopy uses light-emitting diode (LED).

Keywords: Spectroscopy, Non-agriculture soil, NPK, Absorbance, LED

Abstrak

Tanah adalah medium untuk pertumbuhan akar, menyerap air dan bahan larut yang diperlukan. Ujian makronutrien tanah membantu dalam menentukan kandungan nutrien di dalam tanah sebelum menggunakan baja untuk mengawal kualiti dan proses keluaran pertanian dan kesuburan tanah. Spektroskopi merupakan teknologi baru yang cepat dan mudah telah digunakan secara meluas dalam proses pertanian dan analisis makanan. Keupayaan spektroskopi untuk menyifatkan sesuatu bahan dari transmisi atau penyerapan telah digunakan dalam kertas kerja ini untuk mengukur kandungan nitrogen (N), fosforus (P) dan kalium (K) di dalam tanah bukan pertanian. Kertas itu memperincikan penyiasatan awal spektroskopi tanah dengan menggunakan sumber cahaya Deuterium-Halogen dan spektrometer Ocean Optics untuk mengukur tahap keserapan makronutrien. Nutrien yang diekstrak telah dicampur dengan reagen dan larutan berwarna khusus telah dihasilkan. Dua sampel tanah telah digunakan untuk eksperimen iaitu tanah banjir lumpur dan kaolin. Hasil kajian menunjukkan bahawa tahap penyerapan yang tinggi oleh N pada 450 nm, P pada 750 nm untuk kedua-dua sampel. Tahap penyerapan K diukur tinggi pada 500nm untuk banjir lumpur dan 450nm untuk kaolin. Di samping itu, makronutrien yang diuji memberi tahap puncak penyerapan yang sama pada gelombang 970 nm untuk kedua-dua sampel. Bagi kerja-kerja masa depan, ukuran optik akan dilaksanakan menggunakan cahaya boleh dilihat dan berhampiran inframerah LED dan pengesan foto dalam usaha untuk menggantikan penggunaan spektrometer untuk spektroskopi tanah. Ini akan membawa kepada mencapai objektif utama kajian ini dalam membangunkan spektroskopi yang mudah dan kos rendah menggunakan diod pemancar cahaya (LED).

Kata kunci: Spektrometer, Tanah bukan pertanian, NPK, Penyerapan, LED

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1.0 INTRODUCTION

Soil macronutrients, nitrogen (N), phosphorus (P) and potassium (K) are essential for plant growth and needed in large quantity [1, 2, 3]. N, P and K are the most important nutrients in agriculture and NPK fertilizer is the most common fertilizers available in the market [3]. Excessive use of fertilizer can lead to surface and ground water pollution [1] but under application can cause drop in quality of the products [2]. The quantity of NPK varies depending on type of crops and plant growth level [2]. Therefore, the quantity of fertilizer have to be estimated based on the requirements for optimum production at each location in the field [1].

Nowadays, soil nutrient testing or precision agriculture (PA) is required to determine the nutrients availability in soil before applying any fertilizer for quality and process control of agriculture produce and soil fertility [4]. One of PA is conventional soil testing which require the farmers to collect soil samples from their crop field and send to specialized soil analysis laboratory and wait for one to two weeks for the result. This method is time consuming, expensive [1, 4, 5], require highly skill operators and variables that affect the crop yield cannot be optimized at real-time [4]. Due to the cost, the number of samples analyse per field may be limited and cause the soil nutrient concentrations within the field cannot be characterized effectively [1].

There are four types of soil sensor have been used to measure various soil parameters which are mechanical sensor, optical sensor, electrochemical sensor and electrical and electromagnetic sensor. Mechanical sensor is capable to measure soil physical composition, optical sensor use visible and near-infrared wavelength to measure reflectance, absorbance and transmittance of soil [5, 6], electrochemical sensor use ion-selective electrode or ion-selective field effect transistor to detect the nutrients by interact with nutrient ion in soil solution [1, 5, 6] and electrical and electromagnetic sensor use electrical circuits to determine the soil texture, salinity, organic matter, moisture content and other parameters [5, 6]. Table 1 is the listed advantages and disadvantages of the available sensors.

Table 1 Advantages and disadvantages of available sensor

Type of sensor	Advantages	Disadvantages
Mechanical sensor	Maps the specific location of different types of soil within the field [6]	No obvious potential utilization data collected for crop production [6]
Optical sensor	Non-destructive and rapid technique to evaluates its properties in visible and near-infrared (NIR) wavelength ranges [6]	Affected by combination of soil types [1, 6]
Electrochemical	Portable, rapid	Time consuming

Type of sensor	Advantages	Disadvantages
sensor	response, able to measure analyte directly [1]	and requires complex laboratory assessment and analysis [1]
Electrical and electromagnetic sensor	Rapid response, low cost and high durability [5]	Limited by operation speed, contact height, fluctuation in soil moisture and temperature and topsoil depth [5]

Spectroscopy is widely use in optical method to detect the nutrients content in soil such as laser-induced breakdown spectroscopy (LIBS). This technique give real-time analysis, simple sample preparation and small amount requirement but due to soil heterogeneity and matrix effect, this technique is difficult to analyze [4]. Further explanation is discussed in section 1.2.

To overcome LIBS technique limitation, LED and colour reagent has been implemented because this technique is less expensive and low power consumption [7]. This technique is adopted for this research not limited to its efficient and low power consumption, but it does not required acids or other dangerous chemicals.

1.1 Transmittance and Reflectance of Light

Each molecule has its own unique absorption and reflection spectrum. For soil nutrients detection, Beer-Lambert Law is utilized. Beer-Lambert law stated that there is relationship between absorbance and concentration of an absorbing species in a solution [8].

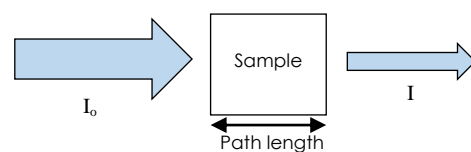


Figure 1 Absorption of light by a sample

Figure 1 shows the absorption of light by a sample when light passes through it. The measurements are usually made in terms of transmittance (T), reflectance (R) and absorbance (A) which can define as

$$T = \frac{I}{I_0} \quad (1)$$

$$R = \frac{1}{T} \quad (2)$$

$$A = -\log T = -\log \frac{I}{I_0} = \epsilon LC \quad (3)$$

Where I_0 = initial light intensity

I = light intensity after passing through the sample.

ϵ = molar absorptivity ($L \text{ mol}^{-1} \text{ cm}^{-1}$),

L = path length (cm) and

c = concentration of the absorbing chemical species (mol/L) [8, 9].

The significance of Beer-Lambert law is to measure the absorbance of a particular sample and to infer the concentration of the solution. There are two situations need to consider. First, if light beam of appropriate wavelength passes through a diluted solution, the photons will encounter a small number of absorbing chemical species (chemical species will absorb light at particular wavelength) and the result might give high T and low A. [9]. Second, if the beam of light encounter the solution for a long period of time, the result might be low T and high A. Thus, Beer-Lambert law stated that absorbance is proportional to the concentration of the sample and proportional to the path length of beam through the sample [9, 10].

1.2 Laser Based Spectroscopy

Laser-induced breakdown spectroscopy (LIBS) is based on atomic emission spectroscopy that has been used widely for soil analysis. This method use laser as light source. The light source is focused on a solid sample and form microplasma that emit light. The emitted light is collected by fiber and detected to monitor the concentrations of nutrients via their unique spectral signature [4, 11]. During calibration phase, LIBS provides quantitative measurements and suitable for low cost field portable instrumentation [11]. This method is widely used to detect the total carbon content and contaminations in polluted soil. The advantages of this method is real-time analysis, require minor sample preparation and small requirement but the drawback is due to soil heterogeneity and matrix effect [4].

1.3 LED Based Spectroscopy

LED based spectroscopy uses LED as light source and spectrometer to measure the light spectrum after passing through a sample. The soil nutrients nitrogen (N), phosphorus (P) and potassium (K) are verified using standard soil test kit which available in the market along with standard colour chart [2]. Using standard colour chart, the result is not reliable because the result always fluctuating due to tester's judgement, so, spectrometer is used to investigate the colour-develop solution. Solution of tested soil is illuminate by visible and near-infrared wavelength range to measure the absorption peak to choose the suitable LED [7]. The selected LED is then illuminate the solution and lights will be reflected depending on absorbance coefficient of the soil. The reflected light is received by another optical fiber and send to the spectrometer to determine the intensity of transmitted light [2, 7]. When light travels through the solution, intensity of light will decrease with increasing number of absorbing species in the solution [7].

LEDs are highly suitable as a light source due to wide wavelength range from ultraviolet to infrared, less expensive and has long durability. Therefore, it is possible to find matching LED wavelength to the absorption band of sample nutrients. The emission spectra of the LEDs are measured by an optical

spectrum analyzer. The quantitative analysis of three soil nutrients is performed using colour reagent solution [7].

In this paper, the characterization of macronutrients content of soil samples (mud flood and kaolin) is reported for visible light wavelength range light source with DH-2000. The result from this paper would help to develop a simple and low cost spectroscopy using LED in determining the macronutrients content in various type of soil samples including agriculture soil.

2.0 METHODOLOGY

2.1 Calibration System

In this experiment, DH-2000 Deuterium Tungsten Halogen Light Source with a wavelength range from 210nm to 1200nm has been employed as light source. The sample was placed in the cuvette has been transmitted with a light from DH-2000. One end of the cuvette holder is connected to the light detector via optical fiber cable. For calibration system, the sample was replaced with optical diffuser to identify the light source spectrum. The light detector is an Ocean Optics spectrometer which has a range from 200 nm to 1100 nm and a spectral resolution to 0.65 nm.

The spectrometer was interfaced to the computer using Ocean View software. Ocean View is a specifically designed program provided by Ocean Optics in order to acquire the data from the spectrometer in real time. In this experiment, optical attenuator was used to reduce the light source intensity. Thus, by having the optical attenuator, the power dissipation can be controlled.

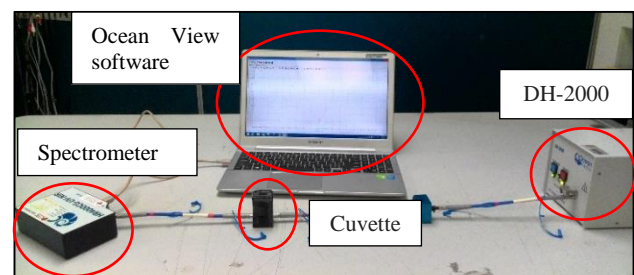


Figure 2 Experimental setup to measure soil absorbance level

2.2 Soil Absorbance Level System

The absorbance level in soil was measured using the experimental setup as shown in Figure 2. The experimental setup consist of three parts, light source, sample and spectrometer. Light source used was DH-2000 and couple to the samples in a cuvette and the data was send to Ocean Optic spectrometer. Two different types of soils were used in this experiment which were mud flood and kaolin. These soil samples had been treated into fine powdered samples.

The soil nutrients extraction is using LaMotte NPK soil test kit which is available in the market. NPK soil test kit senses the amount of N, P or K in soil samples and depending on the amount of the elements in the soil, the colour of solution changes [12]. Experimentally, the extraction of the nutrients on the tested soil was done. Then, the extracted solution was divided into three test tube and mix with provided extractants for N, P and K. The developed colour solution of the mixture was compared with colour chart provided. The colour-develop solution was dripped into quartz cuvette to measure the absorbance level of the nutrients.

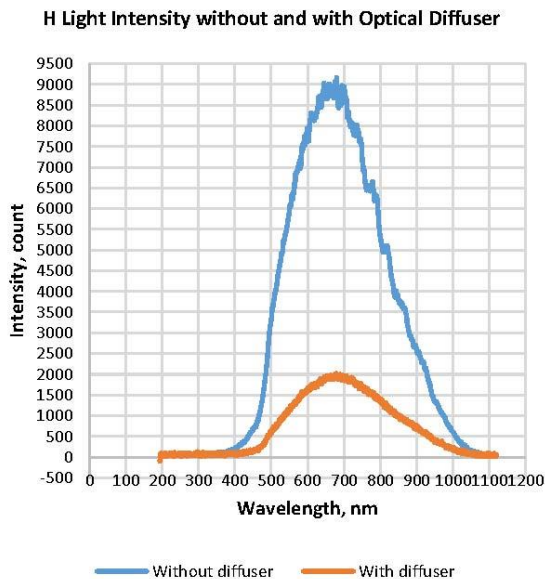


Figure 3 Plotted graph of H light without and with optical diffuser

3.0 RESULT AND DISCUSSION

3.1 Calibration Measurement

DH-2000 light source can emit deuterium (D) and halogen (H) light which D cover ultraviolet light and H cover visible light. For this experiment, only H light is study. Figure 3 shows the emission spectrum of H light. The plotted graph depicts that H produces a continuous spectrum of light, from near ultraviolet until deep into the infrared which covers wavelengths from 400 nm to 1000 nm. Besides that, the plotted graph shows the highest intensity of light obtained at 646 nm. In addition, the intensity of light reduced about 77% of its original value when the optical diffuser was connected to the experimental setup.

3.2 Soil Absorbance Level Measurement

By using extractants from LaMotte, the colour-develop solution produce low red colour for N, medium blue colour for P and low white colour for K on mud flood sample. For kaolin, N and P were measured with very low red colour and very low blue colour respectively. The reagent was seemed failed in extracting K level in kaolin. From colour chart

provided, all tested nutrients for two types of soil samples give low concentration values. Further analysis using spectrometer was conducted.

Figure 4 and 5 indicate the plotted graph of absorbance level of mud flood and kaolin respectively. From the plotted graph, N and P for mud flood and kaolin produced absorption peak at same wavelength which are 450nm and 750nm respectively however for K, the wavelength for absorption peak for mud flood is at 500nm while for kaolin at 450nm. At 970nm, all nutrients for mud flood and kaolin yield absorption peak at same wavelength.

The approximate concentration of plant elements for healthy plant growth have obtained high N concentration, medium K concentration and low P concentration [13] but for agriculture sample in reference [2], the result yield low N, medium P and high K. In terms of wavelength of absorbance level, reference [7] reported wavelength for nitrate is at 540nm and 650nm and phosphate at 700nm and 900nm. Reference [11] reported that potassium has absorbance at 404nm. From the conducted experiment, the concentration of NPK does not give same agreement with reference [2, 13] and the wavelength of absorbance give same agreement for P and K but not for N. In future, improvement will be implemented by allowing the colour solution to absorb more quantity of chemical reagent. Hence that, the non-agriculture soil which were tested for three macronutrients content in this paper show similar behavior as agriculture soil which reported in [2, 11, 13].

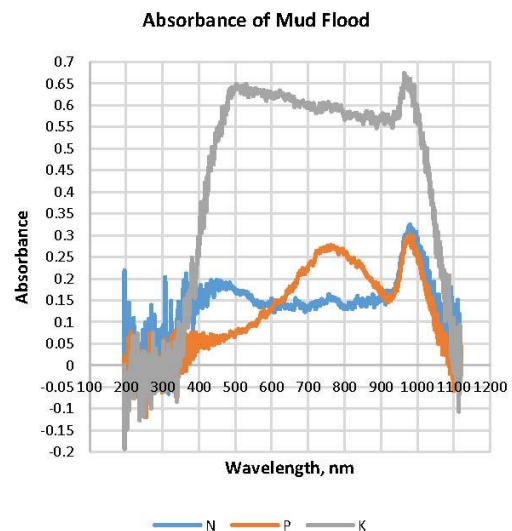


Figure 4 Plotted graph of absorbance level of mud flood

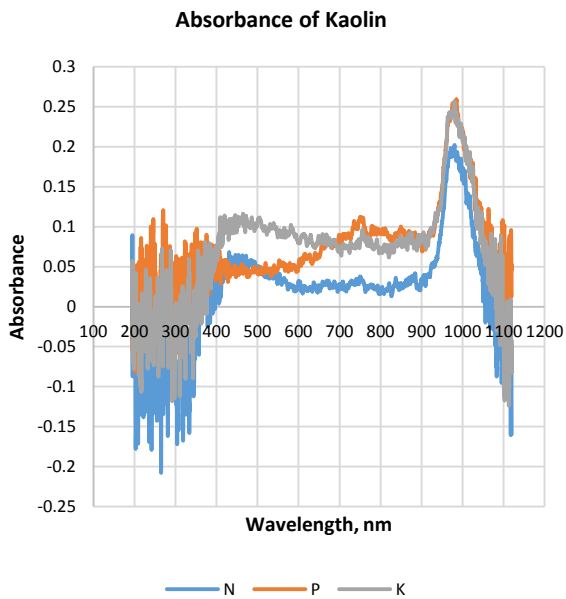


Figure 5 Plotted graph of absorbance level of kaolin

4.0 CONCLUSION

This paper highlights the preliminary experimental analysis using a spectrometer for measuring absorbance of soil nutrients level. As for calibration system using optical diffuser, the light intensity with diffuser decrease due to scattering of light by diffuser. Then, the research is conducted by replacing optical diffuser with sample to measure the absorbance level of the soils. The samples were extracted using LaMotte NPK soil test kit which is available in the market and developed specific colour solution. The colour solution of N, P and K give different wavelength for absorbance value which at 450nm for N and 750nm for P for both sample and K at 500nm and 450nm for mud flood and kaolin respectively. These wavelength give same agreement for P and K but not for N. In future, the visible and near infrared LED and photodetector will be implemented to replace the spectrometer for soil spectroscopy. This would help in developing a simple and low cost using LED based soil spectroscopy to achieve the primary objective of this research.

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