SOIL MACRONUTRIENT DETECTION BASED ON VISIBLE AND NEAR-INFRARED ABSORPTION SPECTROSCOPY

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Dedicated to my beloved father and mother

MOHD YUSOF BIN HASHIM

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and

my brothers and sisters

for their support and encouragement.

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ABSTRACT

Precision agriculture using cost-effective soil fertility measurement is important to obtain adequate quality and quantity of crops. Modern agriculture uses soil spectroscopy, which is a fast, cost-effective, environmentally friendly and reusable method. Soil fertility is used in modern agriculture to sustain plant growth and optimize crop yield. However, most existing light sources and computerized photodetection modules in soil spectroscopy are bulky in size, consume high power and expensive such as tungsten-halogen lamps, deuterium lamps and commercial spectrometer. This thesis proposes an improved experimental module based on absorbance spectroscopy to determine the nitrogen (N), phosphorus (P) and potassium (K) in various soil samples which are extracted using colour-developing reagent. The experimental module consisting light-emitting diode (LED) in visible and near-infrared range, and integrated passively quenched silicon photodiode. The optical absorption of various soil samples, including agricultural and non-agricultural soils are experimentally investigated in absorbance mode using an optimal wavelength range of 467 nm until 741 nm. Beer-Lambert Law (BLL) is applied to identify the relationship between the nutrient concentration and the amount of absorbed light. At a wavelength (λ) of 467 nm, N gives a coefficient of determination (R^2) between 0.49 and 0.63 for agriculture soil samples. Meanwhile, R^2 of agricultural soils for K gives a value from 0.54 to 0.73. At $\lambda = 741$nm, P produces R^2 in the range of 0.47 to 0.82. Furthermore, research findings using LED and photodiode follow the BLL. BLL states that high concentration has many chemical absorbing species which will lower the transmitted light intensity and give low output voltage. This study has shown that absorbance spectroscopy with proposed LED and photodiode modules are able to distinguish the nutrient concentration in the soil.

ABSTRAK

Pertanian teliti menggunakan kaedah mengukur tahap kesuburan tanah dengan kos yang berkesan adalah penting bagi mendapatkan kualiti dan kuantiti tanaman yang mencukupi. Pertanian moden menggunakan spektroskopi tanah merupakan kaedah yang pantas, kos berkesan, mesra alam dan boleh diguna pakai semula. Kesuburan tanah digunakan dalam pertanian moden untuk mengekalkan kadar pertumbuhan dan mengoptimumkan hasil tanaman. Walau bagaimanapun, sebahagian besar sumber cahaya sedia ada dan modul pengesanan foto berkomputer dalam spektroskopi tanah mempunyai saiz yang besar, menggunakan tenaga yang tinggi dan mahal seperti lampu tungsten-halogen, lampu deuterium dan spektrometer komersial. Tesis ini mencadangkan satu modul eksperimen yang ditambah baik dengan menggunakan spektroskopi serapan untuk menentukan nitrogen (N), fosforus (P) dan kalium (K) dalam pelbagai sampel tanah yang diekstrak dengan menggunakan reagen warna. Modul eksperimen ini terdiri daripada diod pemancar cahaya (LED) dalam julat cahaya tampak dan inframerah dekat dan pelindap pasif silikon fotodiod bersepadu. Serapan optik bagi pelbagai sampel tanah, termasuk tanah pertanian dan tanah bukan pertanian diselidik secara eksperimen dalam mod serapan menggunakan jarak gelombang optimum di antara 467 nm dan 741 nm. Hukum *Beer-Lambert* (BLL) digunakan untuk mengenal pasti hubungan antara kepekatan nutrien dan jumlah cahaya yang diserap. Pada panjang gelombang (λ) 467 nm, N memberikan pekali penentuan (R^2) diantara 0.49 dan 0.63 bagi sampel tanah pertanian. Sementara itu, R^2 untuk tanah pertanian untuk K pula memberikan nilai dari 0.54 hingga 0.73 . Pada λ = 741 nm, P menghasilkan R^2 dalam julat dari 0.47 hingga 0.82. Tambahan lagi, dapatan kajian dengan menggunakan LED dan fotodiod mengikuti BLL. BLL menyatakan bahawa kepekatan yang tinggi mempunyai spesies penyerap kimia yang banyak dimana ia akan menurunkan tahap keamatan cahaya dan memberikan voltan keluaran yang rendah. Kajian ini menunjukkan bahawa spektroskopi serapan dengan modul LED dan fotodiod yang dicadangkan dapat membezakan kepekatan nutrien di dalam tanah.

TABLE OF CONTENT

LIST OF TABLE

LIST OF FIGURES

LIST OF SYMBOLS AND ABBREVIATION

N   -   Nitrogen

P   -   Phosphorus

K   -   Potassium

PA   -   Precision Agriculture

NIR   -   Near-Infrared

OM   -   Organic Matter

BLL   -   Beer-Lambert Law

T   -   Transmittance of light

I_i -   Intensity of light before it strikes the substance

I_t -   Intensity of light after passed through the substance

P_i -   Power of light before it strikes the substance

P_t -   Power of light after passed through the substance

A   -   Absorbance

ε -   Molar absorptivity of the sample

L   -   Path length of the cuvette

C   -   Concentration of the sample

NA	-	Numerical aperture
UTM	-	Universiti Teknologi Malaysia
R^2	-	Correlation of determination
EDX	-	Energy Dispersive X-Ray
SEM	-	Scanning Electron Microscope
R_L	-	Load resistance
Wt %	-	Weight percentage
cps/eV	-	Count per second per electrode volt
$\Re_\lambda$	-	Responsivity of photodiode
V_o	-	Output voltage

LIST OF EQUATIONS

CHAPTER 1

INTRODUCTION

1.1 Introduction

This chapter discusses the background of the study, including soil features for agricultural plant growth, soil macronutrient, soil nutrient testing, the nutrients needed by the plants and the impact of the fertilizer amount to the plant growth and the environment. In addition, this chapter has also discussed on several types of soil sensor for measuring various soil properties. Apart from that, the identified problem that leads to this research and the objectives of this study are highlighted.

1.2 Background of Study

The soil is a natural resource which covers the uppermost of the earth's surface and act as a medium for plant roots to grow. The soil consists of minerals, organic matter (OM), living organisms, air and water for life support to the plants. Furthermore, the plant roots absorb the required amount of water and necessary minerals for growth from the soil. Soil fertility measurement is important to determine the availability of nutrients and necessary solute in the soil to ensure better quality and quantity of the crops [1-3]. The fertility level of the soil can be obtained by measuring the soil nutrients such as nitrogen (N), phosphorus (P), potassium (K), soil moisture, soil minerals and soil OM which are the important elements needed for plant growth [3-8].

Soil nutrients are divided into three categories: primary macronutrients, secondary macronutrients and micronutrients. The primary macronutrients, the essential nutrients that highly required in large quantities for plant fertilizing are nitrogen (N), phosphorus (P) and potassium (K) [2, 4, 9-11]. Soils secondary macronutrients that are composed of calcium (Ca), magnesium (Mg) and sulphur (S) are required in small quantities for the plants. Micronutrients that are needed in trivial amounts for the plant to grow in a healthy way are iron (Fe), boron (B), molybdenum (Mo), copper (Cu), chlorine (Cl), nickel (Ni), zinc (Zn) and manganese (Mn) [2, 12]. Soil nutrients are important to specify the fertility of the soil and commonly used for nutritional deficiency detection, which can affect the production and/or quality of plants [13, 14].

The nutrient content must be monitored regularly by the farmers for stabilization of the soil fertility and to control the amount of the fertilizer applications [6, 15, 16]. Excessive use of fertilizer may lead to the contamination of land surface and groundwater, reduce the harvest rate, poor quality in fruit production, and vegetable underdeveloped in colour, size and quantity [1, 4, 6, 10]. Therefore, soil nutrient measurement is needed to accurately determine the presence of nutrients in the soil and used for fertilizer applications at various soil types [4]. This test can result in efficient fertilizer usage, increase crop production at low expenditure, environment protection and product quality enhancement [11, 16].

One of the tests is known as conventional soil test which required the farmers to send the soil samples to the soil analysis laboratory and usually it will takes about four to five weeks for the result of the soil test to be released. This test requires skilled operators in the chemical processes [11, 16]. Furthermore, the sample preparation and the sample treatment process are time-consuming [9, 11-13, 16-18]. Another limitation with this conventional soil test is the cost which limits the number of soil samples analyzed per field [4, 11, 13] and the variable affecting crop yield cannot be altered and optimized in real-time [12, 13]. Thus, a rapid, cost-effective and reliable analysis of soil nutrient test should be developed to provide quality information on soil fertility in real time [19].

One of the technologies used nowadays is called precision agriculture (PA). PA is a farming management approach used in many developed countries to increase the agricultural profit by applying the farm input at the right location and in the right quantity. PA is a useful technique to monitor the soil moisture, surface temperature, photosynthetic activity, and weed and pest infestations [20]. This technique helps to improve the quality and quantity of the products, increase the efficiency of resources used and give better crop management. In addition, PA also could improve the environmental quality and reduced the soil compaction, which limits the roots grow and leads to the insufficient amount of water and nutrient uptake by the plants [1, 12, 13, 17, 20-24]. However, this technique only applicable for large farms [23], time-consuming and costly [13], and the satellite imagery is limited to cloud-free days [21]. Furthermore, this approach is complex and requires expertise to analyze the informations [25-27]. Generally, the device of PA is mounted on the tractor or pulled by the agricultural vehicle. The required instruments and technologies typically for PA are satellite positioning system, control data and transfer system, sensing system and precision application systems [28, 29]. The focus of this research is the sensing part.

Currently, there are four types of sensors have been used in PA such as the mechanical sensor, the optical sensor, the electrochemical sensor, and the electrical and electromagnetic sensor. These soil sensors are utilized to measure various soil properties including soil moisture, soil pH, soil compaction, OM detection, irrigation detection, soil conductivity, soil macronutrient detection and soil mapping.

Figure 1.1 (a) shows the mechanical sensor which usually mounted on a tractor and only capable to measure the soil physical composition and soil compaction. Secondly, an optical sensor as shown in Figure 1.1 (b) uses visible and near-infrared (NIR) light wavelength range to measure the reflectance of soil, which is assembled to the motorcycle for collection of data in real-time. In general, the optical sensor is widely used for soil spectroscopy where the spectra of absorption, reflected or transmitted light (after interacting with the sample) is captured and analyzed by a spectrometer [10, 13]. Thirdly, the electrochemical sensor uses ion-selective electrode or ion-selective field effect transistor to detect the nutrients by

interacting with the nutrient ion in the soil solution. The commercial of electrochemical sensor is given in Figure 1.1 (c) that measure the soil pH level [4]. Lastly, the electrical and electromagnetic sensor as in Figure 1.1 (d) is using the electrical circuits to detect the soil texture, salinity, OM, moisture content and other parameters [4, 11, 13, 24]. Prioritizing the implementation of the sensors for the PA, the laboratory analysis should be conducted to determine the correlation between the proposed systems with the tested soil properties.
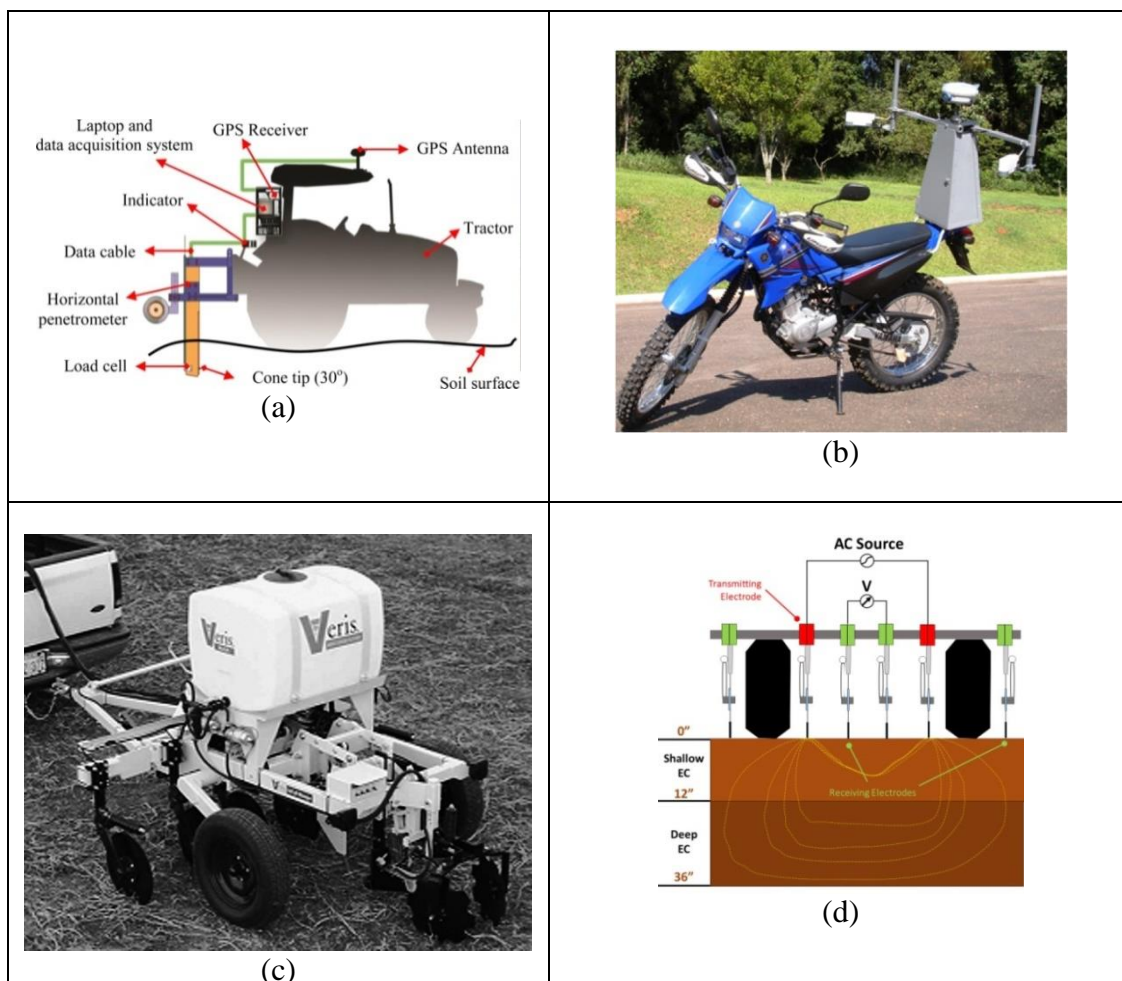
Figure 1.1: The configurations of soil sensors; (a) mechanical sensor [30], (b) optical sensor [31], (c) electrochemical sensor [4], (d) electrical and electromagnetic sensor [32]

Among the four soil sensors, the optical and electrochemical sensors are the most suitable methods to measure the soil macronutrients content [16, 22]. However, the electrochemical method is time-consuming, requires complex laboratory analysis and not suitable for in-situ evaluation due to the sensitivity towards the pressure and

temperature, whereas the optical sensor is efficient, low power consumption and does not require dangerous chemical [3, 10, 13, 24]. Therefore, for this research, the optical sensor method has been chosen for soil macronutrients analysis.

Typically, the optical method usually involves the use of the spectrometer for soil spectra measurement. A spectrometer is a photometer which measures light intensity in a function of wavelength [33] and the spectroscopic technique uses an optical sensor to characterize the elements of materials according to its absorbance or reflectance at a specific wavelength. The characterization of the elements in the sample is achieved from the soil spectrum by illuminating the sample using a wide wavelength range light source and the reflected or transmitted light is sent to the spectrometer. The advantages of spectroscopic technique are rapid, convenient and non-destructive analytical technique. However, the size of commercial spectrometer (Ocean Optics spectrometer) has an approximate weight of 570 g with a dimension of 148.6 mm x 104.8 mm x 45.1 mm and costly (> RM10,000) [34]. Meanwhile, the commercial halogen light source (Ocean Optics DH-2000) is expensive (> RM5,000) with approximately 6 kg in weight and dimension of 150 mm x 135 mm x 319 mm [35]. These costs and sizes become the main limitations for in-situ measurement [36, 37].

Currently, LED is utilized extensively in instrumentations as a light source due to its compact size, low power consumption and much longer lifetime. As reported by [15], three LEDs are used which consists of green, red and infrared LEDs with wavelengths of 524 nm, 632 nm and 849 nm respectively of soil nutrient determination. Six soil nutrients that tested with various concentrations are conducted with ammonium-nitrate, nitrate-nitrogen, phosphorus, iron, manganese and calcium oxide. The nutrient extraction involves commercial soil extractions which produce coloured solution. Moreover, [1] also used three LEDs (green, red and infrared) for ammonium-nitrate, nitrate-nitrogen and phosphorus determination in ten soil samples. These nutrients are extracted chemically according to Olsen method. However, the concentration determination from colour-developed solution is completely human dependent and can lead to confusion. Besides that, the extraction process required complex chemical procedure. Apart from that, one research

conducted by [6] utilized reflective method by placing a mirror at the back of the sample's cell for total internal reflection. The interest nutrients are N, P and K which have been extracted using a commercial soil kit and developed coloured-solution. Six high bright LEDs and one photodiode are constructed in concentric configuration and dipped into the chemical cell containing the extracted samples. The proposed sensor involves longer path length and using reflective mode, which causing more interaction of light with the samples and a portion of the reflected light is loss to the surrounding. In this thesis, an absorbance spectroscopy is utilized using LEDs and photodiode to develop a convenience, miniaturize and low-cost soil macronutrients sensor by employing commercial colour reagent which required simple extraction preparations.

Absorption spectroscopy is a technique to measure the existing elements in the samples by measuring the absorbed radiation of the elements. Each atom absorbs UV, visible or NIR light and release the energy. The amount of energy is measured in the form of photons absorbed by the sample. Each element emits a specific spectral line and has a distinct pattern of the wavelength of the absorbed energy. This happens because of the unique configuration of electrons in its outer shell. The absorption spectroscopic technique is a reliable method to analyze any type of materials. However, the measurement of solid sample must be in liquid form for data analysis. Therefore, the soil samples must be liquefied by the extraction method [38].

1.3   Problem Statement

Nowadays, the soil fertility measurement is important before any cultivation or plantation takes place. One of the fertility measurements is known as soil nutrient analysis. The nutrients availability in the soil is identified at various locations. Precision agriculture (PA) is the technology used to determine various soil properties and only suited to be used in large farm areas. In the undeveloped country, the application of PA is costly and complex. Besides, the conventional method which required the farmers to send their soil samples across the field to the soil analysis laboratory can be expensive and time-consuming depending on the number of

samples and nutrients [9, 13]. In order to overcome the drawbacks, the optical method which is rapid, simple and cost-effective soil analysis utilizing LEDs as excitation light and silicon (Si) photodiode as the detector is proposed.

Moreover, the soil is a complex and natural resource that consists of various organisms and elements. To analyze the nutrient contained in the soil, the nutrient extraction should be conducted prior to soil nutrients measurement. In this research, the extraction of nutrients is carried out using commercial colour-developing reagent where the reaction between the reagent and the sample will form a specific coloured solution according to the concentration of the nutrients in the samples. The evaluation of coloured solution or concentration level will fluctuate according to the human eyes [1, 15]. Therefore, an absorbance spectroscopy is proposed to overcome the problems in improving the accuracy of concentration level evaluation.

1.4   Research Objectives

The objectives of this research are:

1.   To analyze the absorbance of various soil samples in visible and near-infrared (NIR) light range using colour-developing reagent.
2.   To characterize the performance of Si photodiode in absorption spectroscopy (between 200 nm and 1100 nm) using light-emitting diode (LED) light source.

1.5   Scope of Research

This research is conducted to measure the concentration level of soil macronutrients on two types of samples consists of agricultural soils and non-agricultural soils. The interest soil macronutrients are nitrogen (N), phosphorus (P) and potassium (K) have been extracted using commercial colour-developing reagent. The reagent will react with the nutrients and produce specific coloured solution depending on the nutrient content in the samples.

Initially, the extracted nutrients are analyzed using commercial halogen light sources and commercial spectrometer for absorbance measurement. This system is measured in visible and NIR wavelength range between 400 nm and 1100 nm. Apart from that, the utilization of LED and Si photodiode in replacing the usage of commercial halogen light source and spectrometer is carried out. Four wavelengths of LEDs are used, including blue, green, red and NIR LEDs with a center wavelength of 467 nm, 530 nm, 634 nm and 741 nm respectively. The Si photodiode works in the wavelength range of 200 nm until 1100 nm. LED and the photodiode is chosen for soil macronutrients detection due to the compact size, easy to implement for a portable device, inexpensive and mechanically robust [15, 36, 39]. Furthermore, photodiode is one of the components in the spectrometer module.

Generally, the principle of this research followed Beer-Lambert law by illuminating the samples using halogen light or LED in a cuvette and the transmitted light (after interact with the samples) is detected by the photodiode. Simultaneously, the transmitted power is measured using an optical power meter. Besides that, the performance of current-voltage (IV) characteristics of the photodiode is analyzed for soil spectroscopy analysis.

1.6   Significance of the Study

In this study, two types of soil samples consist of agricultural soils and non-agricultural soils are used. The non-agriculture soil samples are chosen to identify the potential commercial soil for agricultural cultivation. To conduct the soil nutrient analysis, soil nutrient extraction should be executed, importantly because the soil is complex and with the natural resources that consist of many living organisms and minerals. The extraction of soil nutrients is conducted using colour-developing reagent to identify the concentration of nutrients in the soil samples. From the reagent, a specific colour solution is formed according to the nutrient contents. The wavelength of each nutrient in the Vis-NIR wavelength range (from 400 nm until 1100 nm) is identified for further data measurement. Moreover, the proposed method

for soil macronutrients content measurements using LEDs and Si photodiode based on absorption spectroscopy is executed.

1.7 Overview of the Thesis

This thesis has five main chapters, each of them dealing with a different aspect. Chapter one discusses the background of the research study, the research objectives and the motivation to achieve the research objective. In this chapter, the organization of the thesis is also given.

Chapter two is the literature review of the theory used for this research. It focuses the information about the soil physical characteristics and the importance of soil macronutrients for plant growth. In addition, the soil sensors for various soil parameters are explained. Besides that, the spectroscopic technique used for soil spectroscopy is discussed.

Chapter three describes the methodology to carry out the research. This chapter is divided into three parts. Part one is the preliminary test using the commercial Vis-NIR light sources to measure and identify the absorbance level and wavelength of nutrients across the wide wavelength range. Part two is the absorbance measurement of soil samples using LEDs and a spectrometer. Finally, part three consists of measurements with LEDs and Si photodiode.

The fourth chapter is discussing regarding the results and discussion obtained from the conducted experiments. All relevant figures, graphs and tabulated data are highlighted in this chapter.

Chapter five is briefly summarized about the research achievement. In addition, several recommendations are provided for further improvement and continuation for future development.

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