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Analysis of Soil Nitrate Ion Selective Electrode (ISE) Sensor Using Arduino UNO

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Abstract: The ecological concern over soil and groundwater pollution caused by agricultural activities has led to the growing interest in precision agriculture. One of the most common types of fertilizer is the nitrogen fertilizer which needed in major amount for plant growth. Over fertilization will contaminate soil and groundwater which can have adverse effect on environment and human health. The main purpose of this research is to measure soil nitrate concentration using nitrate ion-selective electrode (ISE) sensor and Arduino programmable microcontroller. The optimum soil-to-water ratio and the effect of soil solution clarity will be investigated. Standard sampling procedures was conducted at oil palm plantation area, Felda Bukit Goh, Pahang, Malaysia. Validation of the results were carried out in the laboratory. The recorded data indicated accurate readings for Nitrate ISE Arduino was $R^2 = 0.84$. The soil-to-water ratio of 1:2.5 was observed as an optimal proportion ISE analysis. A clear soil solution was crucial for maintaining the accuracy of ISE sensor, to avoid declining of 46.2% accuracy. These results could assist researchers and farmers to accurately monitor the concentrations of soil nitrate on the field effectively as well as an insight to ISE sensor with Arduino technologies.

Keywords: Arduino UNO, ion-selective electrode (ISE), precision agriculture, soil nitrate, soil management

1. Introduction

The ecological concern over soil and groundwater pollution caused by agricultural activities has shifted the focus on precision agriculture to preserve the environment. The increasing demands for food supplies due to the increasing number of populations around the world put huge pressure on the agriculture sector to provide sufficient quality foods for consumers, which lead to the extensive use of fertilizers in the sector. It is expected that approximately 30-50% of crops production was credited to fertilizers [1]. In Malaysia, 90% of fertilizers used in farming were from the mineral category, which consisted of urea, ammonium sulphate, calcium nitrate, phosphate rock, super phosphates, ammonium phosphates, potassium chloride, potassium sulphate, and the compound fertilizers of nitrate, phosphorus, and potassium [2]. One of the general fertilizers used across all farms especially in Malaysia is the nitrogen-based fertilizer, which provide plant with sufficient nitrate as naturally produced nitrate in soil is sparse for plant's intake [3]. Nitrogen is crucial for

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chlorophyll production which promotes plant's growth [4]. It also boosts the production of chemical components that protect plant from plant diseases and various parasites [5]. Shortage of nitrogen and nitrate intake in plants would limit the growth of plant as well as reduction in growth of branches and leaves [6]. Therefore, the utilization of nitrate fertilizer is needed to improve crop yields and quality of plants.

However, over utilize of fertilizers brings a new problem to the agriculture sector in the form of contamination of soil and groundwater. The most common repercussions are unintended off-site nitrogen losses through farming runoff, soil leaching, and trace gas emissions [7, 8]. Leaching of nitrate is a one of the most concern phenomenon that occurs whenever rainfall or irrigation exceeded. Since nitrate is mobile in the soil, it is readily moves with any water passing beyond the rootzone. Hence, nitrate may eventually reach groundwater.

Uncontrolled use of nitrogen fertilizers could also lead to high mineral salt concentration in soil which induce "burning" of plants and damage leaves [9]. Another problem arises is the accumulation of nitrate in well water [10]. This would lead to methemoglobinemia in infants [11, 12, 13]. These apparent negative consequences call for an effective solution to avoid further pitfalls. Uncontrolled usage of fertilizers can be corrected by introducing *in-situ* method to monitor the amount of nitrate available on the field which subsequently give an insight to farmers on the amount of nitrate fertilizer needed to be utilized in specific time. The implementation of precision agriculture (PA) could provide the exact solution to this problem in order to improve outputs [14]. Importantly, PA may minimize the over-usage of fertilizer in agriculture [15]. Three fundamental specifications of development of proximal soil sensor in PA are analysis time, measurement precision, and cost. PA help farmers to break free from custom practice of relying on past experience and intuition in farming instead of utilizing technology and empirical data.

The laboratory determination of soil nitrate utilizes the automated ion-analyser [16]. Kjedahl method is also the go-to method for nitrate measurement in soil, however it is time consuming and not suitable for real-time assessment. Therefore, numerous studies had been done to figure the most efficient technique to measure soil nitrate. Several researchers chose optical and near infrared-based sensor to measure soil nitrate [17, 18, 19]. While some others chose the electrochemical-sensors which are consisted of Ion sensitive field effect transistor (ISFET) and ion selective electrode (ISE) [14, 19, 20, 21, 22]. ISE are potentiometric sensors that are capable of giving direct measurement in unfiltered soil extracts. A nitrate ISE consisted a solid polymer membrane with a porous plastic disk at the tip of the electrode to detect nitrate ion in aqueous soil sample. A voltage that dependent on the level of nitrate ion available in soil sample solution develops once the ions passing through the membrane. The voltage produced between the sensing and reference electrodes is the reflection of the concentration of nitrate ion in the sample solution [23]. According to Nernst equation, ISE response is linear equation:

$$E = E^{\circ} + m(\ln a) \qquad (1)$$

Where E, measured voltage, E_o, standard potential for the combination of the two half cells m, slope ln, natural logarithm and a, activity of measured ion species. The Nernst equation may be rewritten to express the electrode response to the concentration, C, of the measured ion species, assuming the ionic strength is fairly constant:

$$E = E^{\circ} + m(\ln C) \qquad (2)$$

However, most of the studies on the development of ISE were carried in the period year of 1994–2010. Therefore, this project focused onto development a sensor system which paired ISE sensor with the Arduino microcontroller and software system. This project has a merit as the pairing of Nitrate ISE sensor with Arduino UNO microcontroller was not possible in the period year of 1994-2010 as both technologies were either not maximized or not available yet.

1.1 Effect of Soil-to-Water Ratio and Soil Clarity to the Accuracy of ISE

Following the same trend as previous section, some researches showed contradicting results. A study showed that there were no significant differences in the readings of nitrate from soil sample ratios of 1:15, 1:5, and 1:3 [22]. Another study tested the ratios of 2:1, 1:1, 1:2.6, and 1:5 and observed that the change in electrode voltage were similar in each ratio. By maintaining a constant ratio throughout the experiment was essential in maintaining the accuracy of ISE [24]. Nonetheless, another group of researchers claimed that the soil-to-water ratio could affect the accuracy of ISE. Pioneer study on this matter claimed that the effective ratio of 1:5 was effective [25]. Another study used the ratio of 1:10 in their research [26]. The difference in ratio used in previous studies showed that there was no specific ratio chosen for ISE sensor. Therefore, an in-depth study would be conducted in this project in order to determine the optimal soil-to-water ratio for nitrate measurement using ISE sensor.

2. Materials and Methods

2.1 Sensor System Development

The main appeals of Arduino UNO microcontroller and Vernier Nitrate ISE sensor are the versatility and the adaptability of both equipment to be utilized in various conditions (Fig. 1). These instruments enabled the authors to develop a sensor system that can be operated in the laboratory or on the field as *in-situ* handheld device. For the laboratory set-up, Vernier Nitrate ISE (Fig. 1B) sensor was connected to the Arduino UNO via port 1 on the Arduino Interface Shield (Fig. 1A). A connection to computer from the sensor system was made via USB cable. The computer is the source of power for the sensor system as well as used for displaying data.

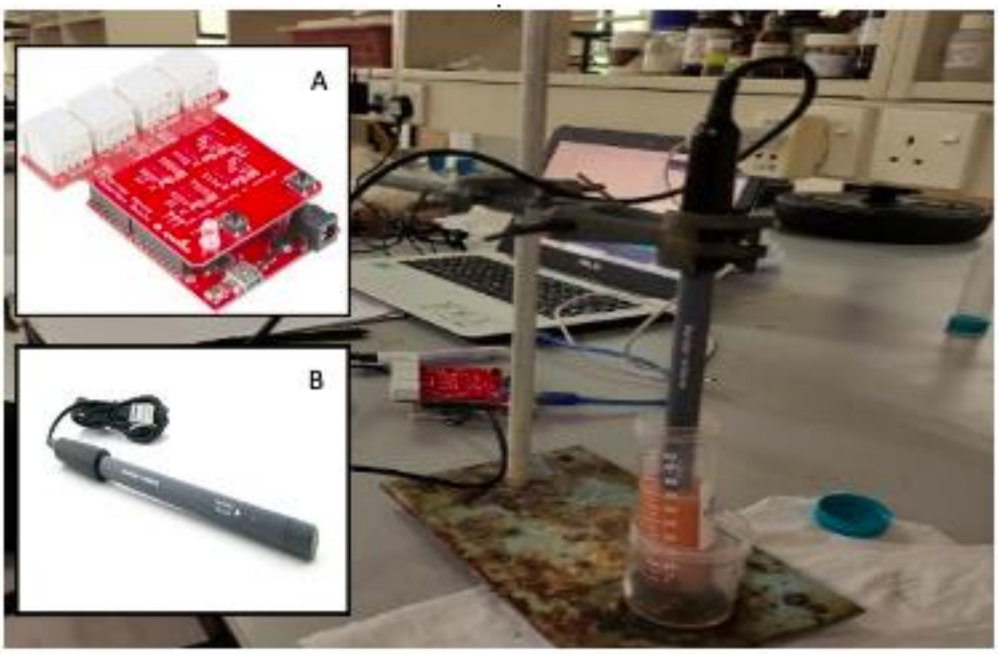


Fig. 1 - Experimental setup consisting of (a) Arduino® UNO programmable microcontroller and; (b) Vernier Nitrate ISE sensor

For on-site set-up, the connection from Vernier Nitrate ISE sensor and Arduino® Interface Shield was the same as before. A connection between the shield was made with a Digital Control Unit (DCU) via the digital sensor port (BTD socket). An LCD was connected to the DCU to display data. A 9V battery was connected to the board through the power port which replaced computer as the power supply for the sensor system (Fig. 2). The on-site system design was as follows

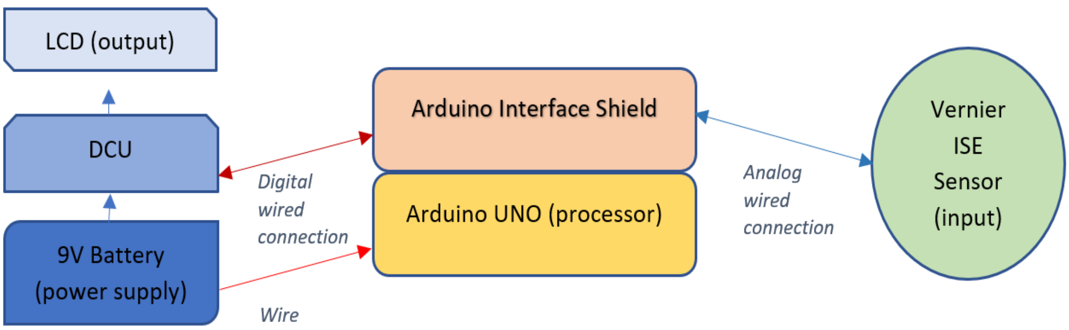


Fig. 2 - In-situ block diagram system for Vernier Nitrate ISE Arduino UNO sensor

The software development was made using Arduino IDE software. One of the advantages of using this software is the vast library available that contain package sections of code that grant user to add functionality to a program. The VernierLib library makes software development more efficient and less time-consuming.

2.2 Sensor Calibration

Sensor calibration was made by standard method suggested by [27], by preparing nitrate calibration standards (0, 25, 50, 75, 100, 250, 500, 750, 1000 ppm) and measuring the standards with Vernier ISE sensor.

2.3 Field Sampling Procedure

Sampling was carried out on-site at the oil palm plantation of Felda Bukit Goh in Kuantan, Pahang, Malaysia (3°54'51.8"N 103°15'50.1"E) to test the capability of the Nitrate ISE sensor to measure nitrate in soil at an agricultural area where the applications of nitrate fertilizers were regular (Fig. 3). A total of 25 samples were collected by using grid soil sampling technique with the distance of each soil samples was 10 m in X and Y directions [28].

Fig. 3 - Oil palm plantation of Felda Bukit Goh, Kuantan (a) on-site view and; (b) satellite view from Google maps and grid line for sampling purposes

First, designated soil slot was drilled using an auger, 20 cm deep hole was dig in the ground and 100g of soil were collected for laboratory analysis. Three centrifuge tubes were filled with 25 ml, 50 ml, and 75 ml of deionized (DI) water to represent the ratios of 1:2.5, 1:5, and 1:7.5 respectively. Afterwards, soil sample were added to the tubes. The tubes were capped and shaken for 30 seconds. During this time, the soil was in slurry condition due to the vigorous shakes. The measurements of nitrate were taken instantly to represent the slurry soil test. To obtain a clear soil sample solution, the solution was left to sit for approximately 60 seconds until soil particles settled at the bottom of the tubes (two layers of soil and water were formed) before being measured.

In order to validate on-site measurement data, samples were analyzed using spectrophotometer (Merck Millipore, Spectroquant Pharo 300) with nitrospectral subsequent to extraction with calcium chloride solution by means of Spectroquant Nitrate-Test.

3. Results

3.1 Calibration

Recorded data obtained from the sensor calibration procedure were plotted (Fig. 4). The calibration curve reflected accurate operation of Arduino Nitrate ISE sensor in both low-range nitrate concentrations and high-range nitrate concentrations, with high value of coefficient of determination, R^2 recorded ($R^2 = 1$) for both tests. The R^2 value is a measure of how well a set of sensor data fits a calibration curve, i.e., the relationship between sensor readings and reference instrument readings. The closer the value of R^2 to 1, the higher the accuracy of the sensor reading. This metric is widely used in the agricultural and environmental works, as being done by [14, 15, 19, 24] in their studies.

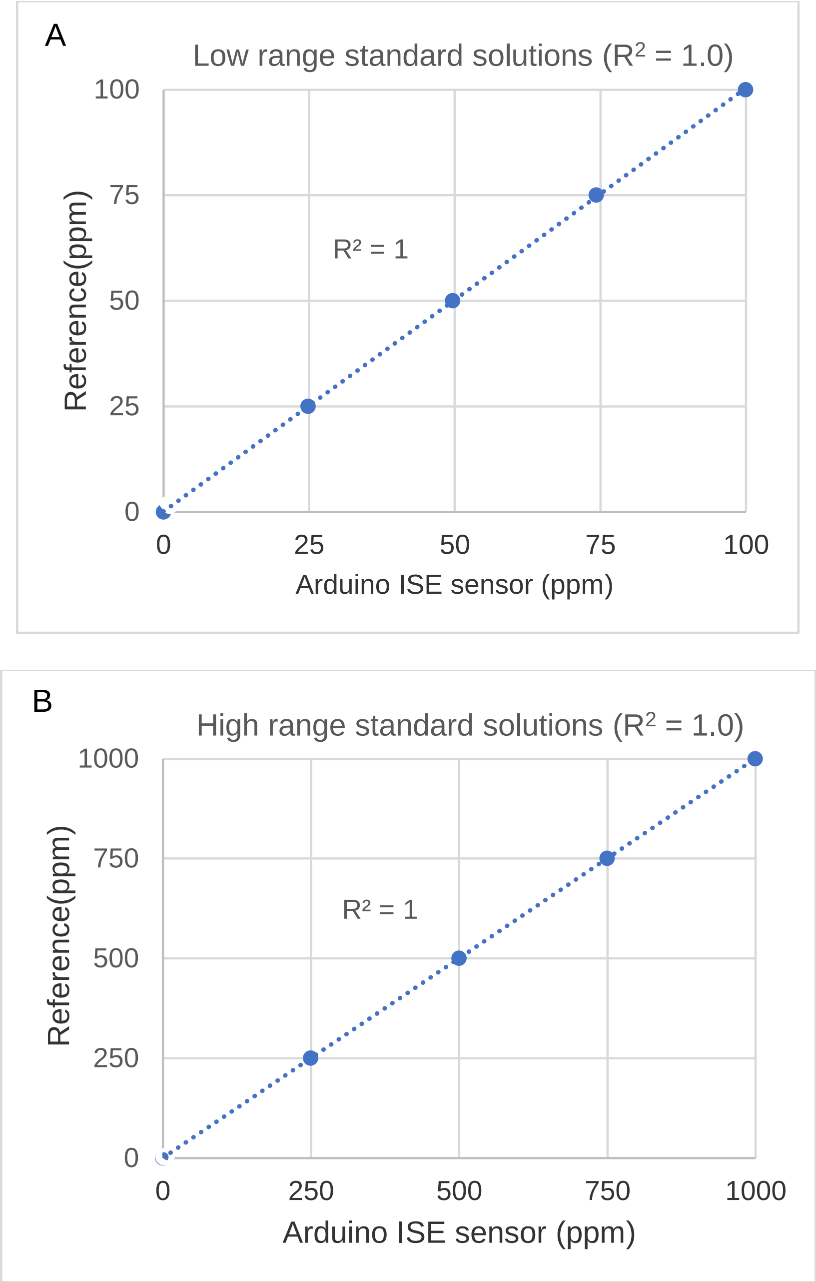


Fig. 4 - Plotted graph of calibration procedure using (A) low-range and (B) high-range nitrate standards solutions, where $R^2 = 1.0$

3.2 Soil-to Water Ratio

With the aim to evaluate the accuracy of the sensor, nitrate measurements were made in various soil-to-water ratio. All collected data were analysed and calculated R^2 values were 0.84, 0.78 and 0.66 respectively (Fig. 5).

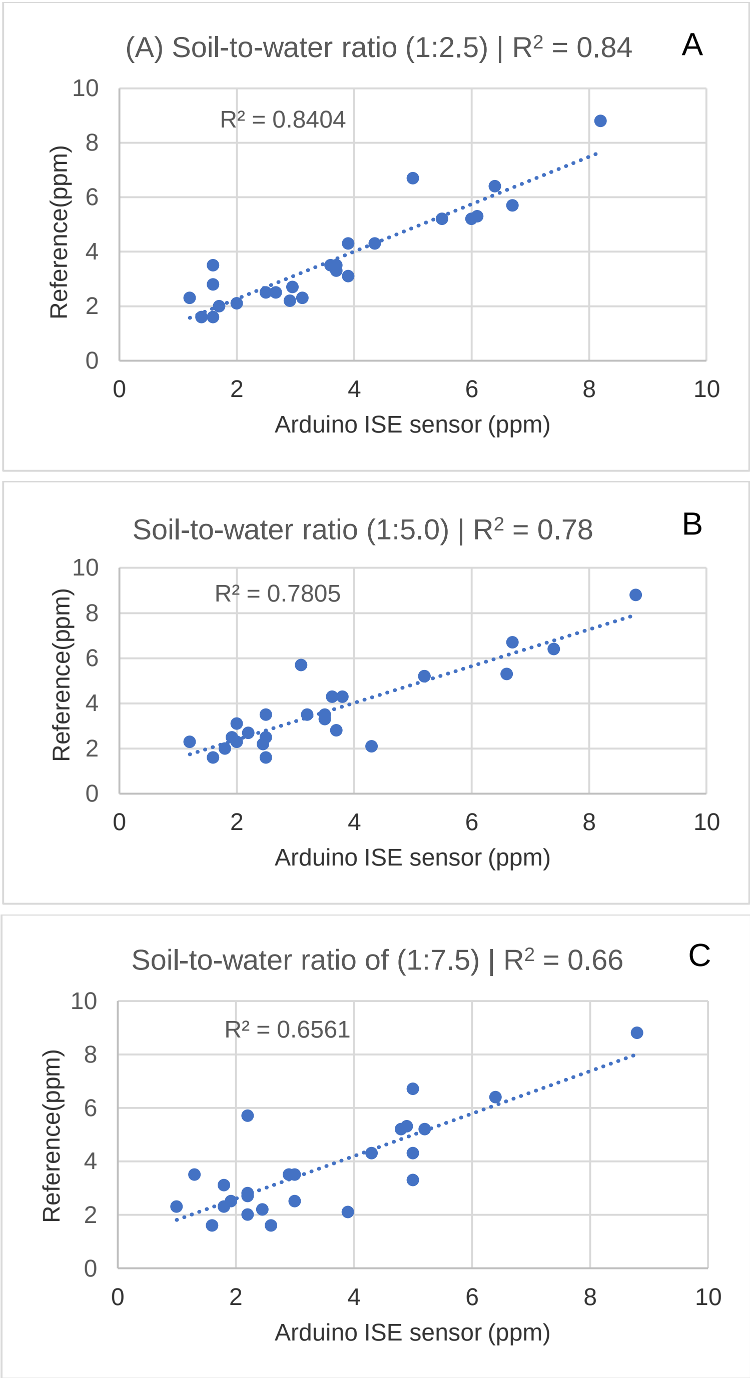
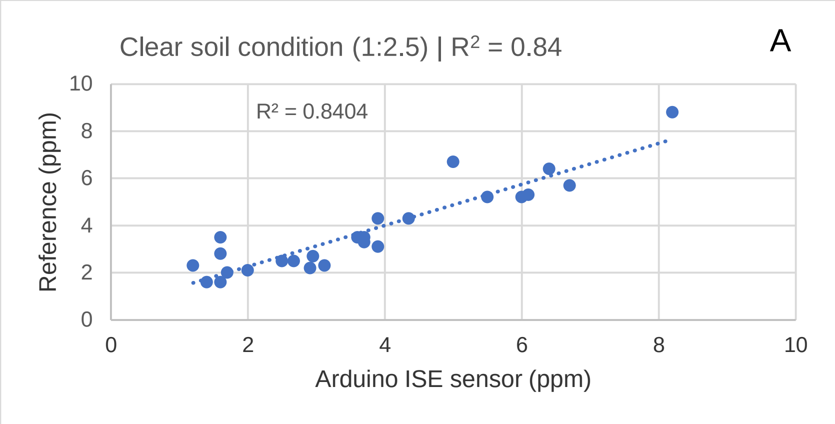


Fig. 5 - Comparison of R^2 measurements of soil nitrate concentration at three different soil-to-water configuration which are (a) R^2 = 0.84 for ratio 1:2.5; (b) R^2 = 0.78 for ratio 1:5.0 and; (c) R^2 = 0.66 for ratio 1:7.5

3.3 The Effect of Clear and Slurry Soil Condition

In order to verify the comparison between clear and slurry soil condition, the specific experiments have been conducted. The results obtained from nitrate measurement in different soil clarity with a constant ratio of 1:2.5. It can be observed that the measurement (Fig. 6) from clear soil condition was significantly improved compared to measurement in slurry soil condition as demonstrated by the R^2 = 0.84 for clear soil and R^2 = 0.45 for slurry soil condition.



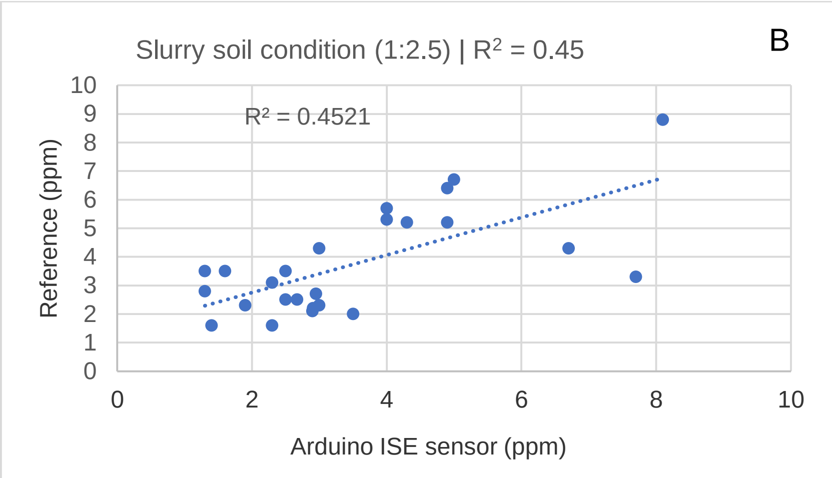


Fig. 6 - The R^2 value of nitrate concentration measurement results from (a) R^2 = 0.84 for clear and; (b) R^2 = 0.45 for slurry soil sample condition.

4. Discussions

4.1 Calibration

Before on-site detection system can be utilized, Nitrate ISE sensor was soaked with the standard solution and calibration steps were strictly followed as provided by the manufacturer. Low and high range standard solution were used. The calibration standards were free from interfering ions, in contrast to the soil condition on the field which contained numerous interfering ions that could interrupted the readings of ISE sensor. After the sensor was properly calibrated, the tip of Nitrate ISE can be inserted into the aqueous samples for measurements. Both low and high range standard were perfectly calibrated with the reading of R^2=1.00 respectively, and ready to utilize (Fig. 4).

4.2 Effect of Soil-to-Water to the Accuracy

Looking back at previous studies and projects on this topic, researchers have divided opinions on the most optimum soil-to-water ratio for nitrate ISE measurement. A study by [29] compared the soil-to-water ratios of 1:1.15, 1:5, and 1:3 and revealed that there were no significance differences recorded. [24] also supported this claim when compared the ratios of 2:1, 1:1, 1:2.6, and 1:5. Interestingly, the pioneer research on this subject in 1970 incorporated the ratio of 1:5 as the best [30]. Another study assumed the ratio of 1:10 throughout their study [26]. These findings suggested that although nitrate ISE is a suitable device for nitrate measurement in soil, there is however a gap of knowledge existed in the optimum way to operate the device, specifically in term of soil-to-water ratio.

The R^2 values were obtained from three different ratios of 1:2.5, 1:5, and 1:7.5 were recorded. The highest R^2 was calculated to be 0.84 from ratio of 1:2.5 (Fig. 5A), followed by 0.7805 from ratio of 1:5 (Fig. 5B), and the lowest recorded R^2 was 0.66 from ratio of 1:7.5 (Fig. 5C). The results showed that the ratio of 1:2.5 was the optimum ratio for nitrate measurement using Nitrate ISE device, as the ratio recorded the highest R^2 value of 0.84. It can clearly reveal that, by adding too much water into the soil solution might affected the accuracy of the ISE by interrupting the ions concentration in the mixture. It was advisable that interfering ions such as ClO_4^-, I^-, ClO_3^-, CN^-, BF_4^- might interrupted the measurements [23]. [27] also highlighted the issues of ISE responds to several interfering ions such as iodide, chloride, and bromide. As reported by a study, maintaining a constant soil-to-water ratio was essential to preserve the accuracy of an ISE sensor [24]. Therefore, the ratio of 1:2.5 was selected and used for the rest of field sampling.

4.3 The Effect of Soil Clarity Condition to The Accuracy

A previous study mentioned that soil solution clarity and the soil-to-water ratio during measurement play an integral part in the accuracy of the sensor [21]. Interestingly, there were contradicting reports from a handful of previous researches on the effect of soil solution clarity to the accuracy of ISE sensor. A pioneer study on this matter claimed that there was no significance difference between the measurement of unfiltered and filtered soil samples, they also reiterated that clear soil sample solution could have assisted in prolonged the lifespan of ISE electrode. [22, 31] supported this claim by noting that ISE sensor is capable of measuring nitrate in slurry soil sample. However, several researchers rebuffed this claim by asserted that ISE was not suitable for nitrate measurement in slurry soil sample solution. The results obtained were poor in slurry soils [32].

Recent researches also mentioned that ISE measurement in slurry soil would reduce the accuracy of the sensor [33, 34]. In order to validate the finding, we conducted the experiment, and the Figure 6 shows the different soil clarity at a constant ratio of 1:2.5. It can be observed that the measurement from clear soil condition was significantly higher compared to measurement in slurry soil as proven by the $R^2 = 0.84$ for clear solution and $R^2 = 0.45$ for slurry solution. Measurement that taken in slurry soil condition was 46.2% declining in accuracy compared to measurement in clear soil condition. These results proved that slurry soil mixture could significantly disrupted the working of an ISE sensor and in-line with the results from some of the previous studies [32,33,34].

5. Conclusion

An *in-situ* Arduino Nitrate ISE sensor that can measure the nitrate concentration in soil in less than 60 seconds has been successfully developed which can enhance productivity and at the same time reduce wastage and preserve the environment. The soil-to-water ratio of 1:2.5 was established as the optimum soil-to-water ratio for nitrate measurement using Nitrate ISE. The R^2 of 0.84 shows a good result in term of the accuracy of the Arduino ISE sensor after testing of 25 sets of samples. In addition, the measurement accuracy declined approximately 46.2% was observed with slurry sample solution. It was very crucial to make sure that measurement was done in clear sample solution in order to maintain the accuracy of ISE sensor and preserve the condition of the ISE membrane.

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