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Development of Nitrate-Nitrogen Calibration Curve using Laboratory Resistivity Method at Neutral pH Condition

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Abstract: The abundance amount of nutrients in a waterbody will lead to the occurrence of algae blooms where some of the algae produce toxins that are harmful especially to higher forms of life. Nitrate which is one of nutrient category has several advantages towards the environment especially around the chemistry in soil and plant metabolism. Thus, excessive amount of nitrate can contribute to the pollution of water due the formation of eutrophication. Hence, this conducted study are very important to prevent the water solution from getting to intensify. The purpose of this study was to obtain the electrical resistivity value on nitrate-nitrogen concentration at neutral pH range by using soil box resistivity meter to generate calibration curve nitrate-nitrogen concentration and resistivity value. A series of experiments were conducted at high (200 mg/L to 1000 mg/L), medium (20 mg/L to 100 mg/L), and low (2 mg/L to 10 mg/L) concentration of nitrate-nitrogen. The range of neutral condition covers from 6.4 to 7.4. Therefore, calibration curve of relationship between nitrate-nitrogen concentration and resistivity value was generated. The results of the graph show a decreasing of resistivity value in corresponding of the increasing of the concentration. Overall, as the value of R^2 showing its closer to 1.000, thus it can be referred that the calibration curve provided a great details and references as the data collected was quite accurate.

Keywords: Nitrate-nitrogen, Electrical Resistivity Value, Calibration Curve, Neutral pH

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1. Introduction

Water quality which affected by the quantity and quality from organic and inorganic presence of salts in water. For the past few years, fertilizers have been one of the few causes for water pollution to increase gradually. [1] stated that, water is one of the life essential for living things. Therefore, the accessibility of the water supply should be safe and adequate for daily purpose use. Thus, with the significant number of benefits on health can be obtained resulting from the improvement of accessibility by safe drinking water. Safe-drinking water indicates that the water is pollution free. Water pollution occurs when harmful substances, such as chemicals or microbes, pollute and decrease the quality of water in a specific water body for instance lake, river, ocean, or aquifer. This in resulting to the contribution of toxicity level in water where it is unsafe for consumption usage. Since water nature acting as the universal solvent, it could dissolve a various amount of substance. This can be an issue, though, because water is prone to pollutant exposure. Hence, the study conducted to investigate whether the possibility of pollution of water with the presence of certain substances in a water body.

One of the geophysical methods available is electrical resistivity. Seismic, gravity magnet and electromagnet and induced polarization are included as examples of indirect or surface geophysical methods available. Based on previous case study in Malaysia, resistivity and seismic method known to has mobility of contrast result and high rates of successfulness. One of the factors affecting the resistivity value that contribute above is pore of fluid and geomaterials grain matrix[2]. In addition, when a relatively slow rate of the currents that was carried by ions, the electric current can spread through the electrolysis process in geomaterials[3]. The earth and electric current flow have an impact towards the electrical resistivity's reaction. Table below shows different comparison of characteristic for resistivity.

Furthermore, when conductivity is high, water sample will have low resistivity as the relationship between water resistivity and conductivity is reciprocal. Resistivity and conductivity methods can be applied for both to measure the purity of water sample. This study focussing on the solution in neutral pH range where presumably strong acidic solution gives high conductivity. This strongly happens due to where hydrogen with high concentration of ion in the acidic solution. On the other hand, the lower the concentration of ions in a solution or soil, the lower the electrical conductivity (EC). Generally, the conducting of electricity is better with both strong acidic and strong alkaline but pH-neutral material are less conducive. Resistivity is measured in the units of Ohm-meter. Material that has low resistivity easily flow the electricity through a material and vice-versa. [4] stated that high resistivity is similar to low conductivity whereby low resistivity is similar to high conductivity.

Electrical resistivity has been known to shown to be a good predictor of several soil hydraulic parameters including water content, saturation, and hydraulic conductivity, according to the study[5]. A soil box resistivity meter is utilized to measure the electrical resistivity of liquid. The concentration and type of salts dissolved in the formation fluids determine its resistivity[6]. Measurement of electrical resistivity can be done by using soil box and two electrodes connecting to the soil box resistivity meter. The soil box filled with fluid sample were let it flow with the constant voltage of current. This will give effect to resistivity's result with the amount of ion contained. Electrons flows through the solutions by ions. Therefore, higher concentration of ion or salt dissolves in the sample, the lower the reading of the resistivity value obtained.

For this study, resistivity soil box was used for the laboratory resistivity method. Therefore, an experiment was conducted with Nitrate-nitrogen in the neutral range of different concentration to generate the calibration curve between Nitrate-nitrogen and electrical resistivity value obtained.

2. Materials and Methods

2.1 Preparation for Solution in Neutral Condition

The neutral condition has a range of pH 6.4 to pH 7.4 were considered for the preparation of standard solution.

2.2 Preparation of Solution

Throughout the experiment, there were other reagents included for preparation of the standard solution of nitrate-nitrogen.

2.2.1 Preparation of Decon 90 Liquid Detergent

All apparatus needed for the experiment should be cleaned thoroughly to prevent any addition of contamination. For the Decon 90 liquid detergent solution, 10ml of Decon 90 was put into 500ml of volumetric flask and filled with distilled water until reached its meniscus level. The solution was kept aside and used to clean any apparatus any time needed.

2.2.2 Preparation of Sodium Nitrate Stock Solution

All apparatus needed for the experiment should be cleaned thoroughly with Decon 90 solution and rinsed well to prevent any addition of contamination. The solution for 1000 mg/L were prepared with 6.07g dried Sodium Nitrate transferred to 1000ml volumetric flask using plastic funnel. The funnel was rinsed with ultra-pure water to prevent any leftover. Volumetric flask that has been filled then drop little by little with ultra-pure water using a plastic pipette until reached its meniscus level before close its cap and shake it 5 times until fully dissolved. All steps were repeated with 12.14g of Sodium Nitrate for 2000 mg/L solution after transferred. All the solution were transferred and kept into Duran bottle.

2.2.3 Preparation of Dilution Solution

All apparatus needed for the experiment should be cleaned thoroughly with Decon 90 solution and rinsed well to prevent any addition of contamination. The Sodium Nitrate stock solution with 100 mg/L, 1000 mg/L, and 2000 mg/L were being prepared by diluting it according to the Table 1 shown below in 1000ml volumetric flask with ultra-pure water and shaken it to mixed well. All the solution were transferred and kept into Duran bottle.

Table 1: Volume of stock solution with different concentration

Concentration (mg/L)	2	4	6	8	10	20	40	60
Stock Solution Volume (mL)	20	40	60	80	100	20	40	60

Table 1: (continued)

Concentration (mg/L)	80	100	200	400	600	800	1000
Stock Solution Volume (mL)	80	100	100	200	300	400	1000

2.2.4 Preparation of Balancing Solution

All apparatus needed for the experiment should be cleaned thoroughly with Decon 90 solution and rinsed well to prevent any addition of contamination. Firstly, preparation for base solution Sodium Hydroxide (NaOH) started with weighing 4g of NaOH pallet and transferred into 100ml volumetric flask and filled it with 100ml of ultra-pure water. The volumetric flask was shaken well 5 times after closed it tightly to mixed well the solution and labelled as 1M NaOH. All the solution were transferred into Amber bottle and kept in the chiller. Steps are repeated with different mass as shown in Table 2.

Table 2: Mass of various mol NaOH

Mole of NaOH (M)	0.5	1	2
Mass of NaOH (g)	2	4	8

Secondly, preparation for acid solution Sulphuric Acid (H_2SO_4) started with measuring 0.54ml of Sulphuric Acid and transferred into 100ml volumetric flask and filled it with 100ml of ultra-pure water. The volumetric flask was shaken well 5 times after closed it tightly to mixed well the solution and labelled as 1M H_2SO_4. All the solution were transferred into Amber bottle and kept in the chiller. Steps are repeated with different mass as shown in Table 3.

Table 3: Mass of various mol H_2SO_4

Mole of H_2SO_4 **(M)**	0.5	1	2
Mass of H_2SO_4 **(mL)**	0.54	2.72	5.43

2.2.5 Procedure for Resistance Reading

In these experiments, firstly after all electrodes were connected to the soil box resistivity meter, 250ml of 2 mg/L of Sodium Nitrate solution starting with pH 6.4 were transferred into it. Then, move the selector range switch that label "Ohms Multiply By" to the 100K setting and set the "Balance Dial" button to "10" when the approximate solution resistance is unknown. Next, switch the "Null Sensitivity" to "Low" position. When the meter needle movement went right, it represents as null and indicate the resistance setting too high. While holding the "Null Sensitivity", the 10K, 1K, 100K and etc resistance ranges need to be adjust so that the meter needle will move to the left of the null position (also left from center position). The position of the "Balance Dial" are changed until its needle position back at the meter center of the null. Multiply the range setting "Balance Dial" by setting the resistance value on the switch labelled "Ohms Multiply By". Use the resistance value obtained for resistivity calculation. All the procedure were repeated for different pH (6.6, 6.8, 7.0, 7.2, 7.4) and concentration as shown in Table 4 below.

Table 4: Low, medium, and high concentration

Type of Concentration	**Concentration Value (mg/L)**				
Low	2	4	6	8	10
Medium	20	40	60	80	100
High	200	400	600	800	1000

3. Results and Discussion

The resistivity value of pH value for each concentration (low, medium, and high) were tabulated and analyzed its data. Each of reading were taken 3 times to get average value for accurate data as in Table 5.

Table 5: R^2 value with different pH and concentration level

pH	Concentration	R^2
6.4	Low	0.9626
	Medium	0.5439
	High	0.8739
6.6	Low	0.9911
	Medium	0.5916
	High	0.7738
6.8	Low	0.9874
	Medium	0.5973
	High	0.7738
7.0	Low	0.9449
	Medium	0.5720
	High	0.7738
7.2	Low	0.9674
	Medium	0.6288

	High	0.8136
7.4	Low	0.8043
	Medium	0.5745
	High	0.7738

Concentration (mg/L):
Low – 2, 4, 6, 8, 10
Medium – 20, 40, 60, 80, 100
High – 200, 400, 600, 800, 1000

3.1.1 pH 6.4

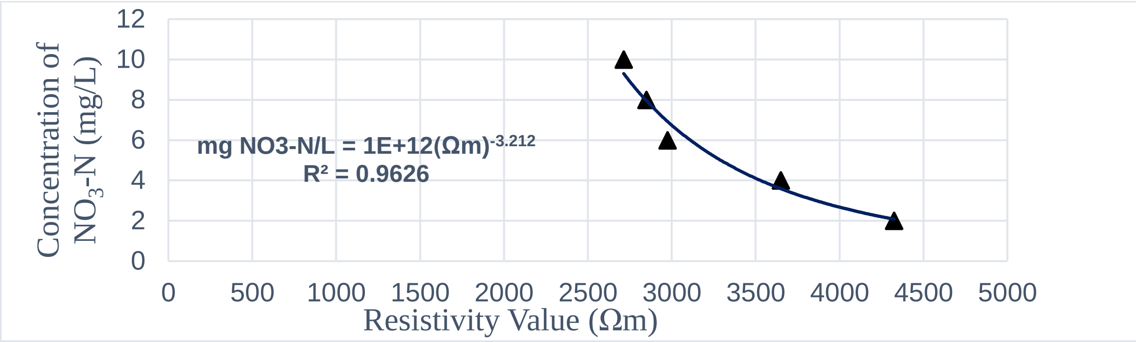

Figure 1: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 6.4 (low concentration)

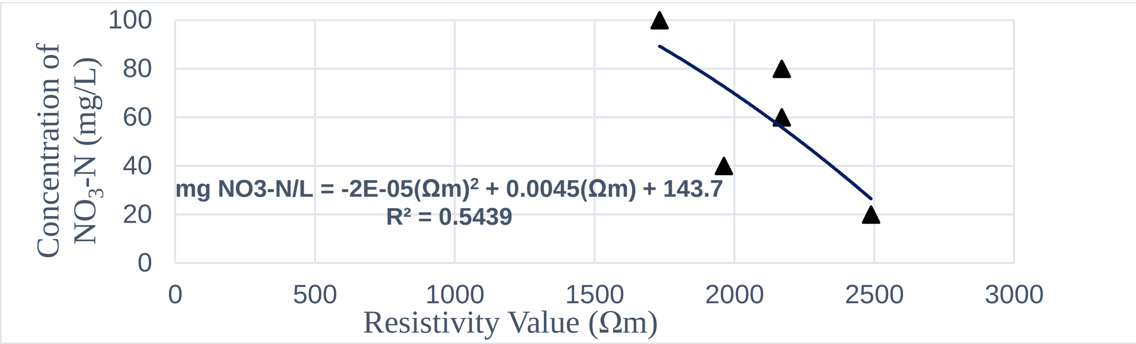

Figure 2: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 6.4 (medium concentration)

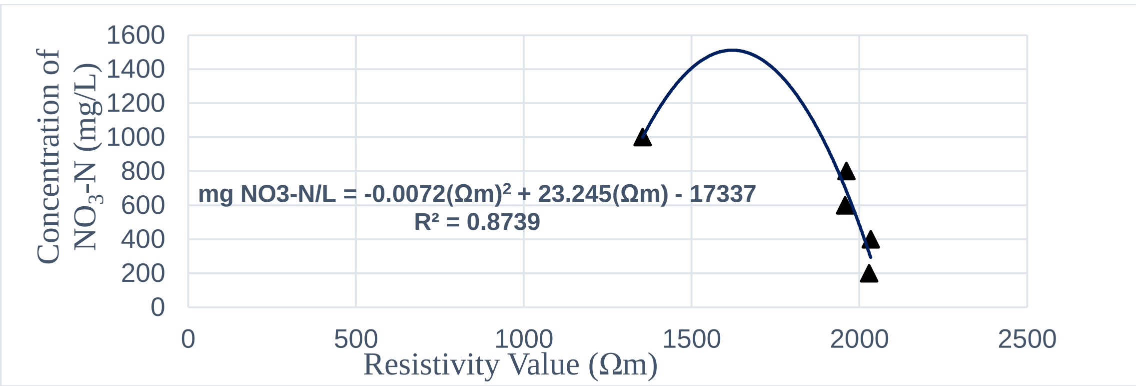

Figure 3: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 6.4 (high concentration)

3.1.2 pH 6.6

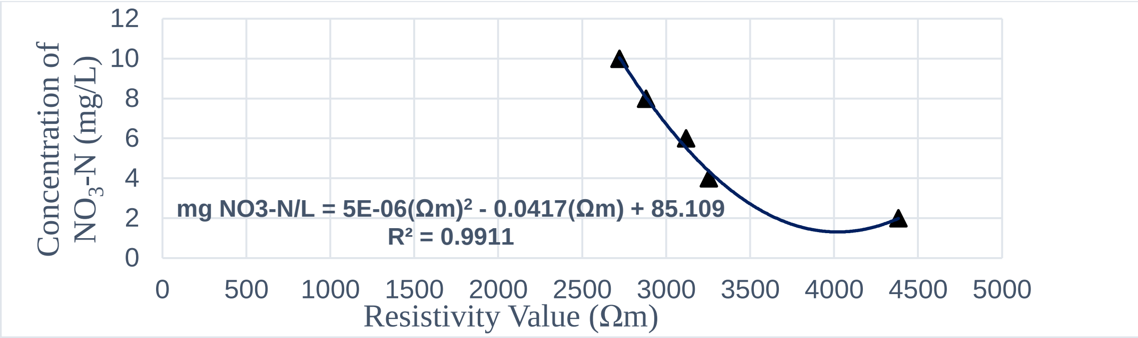

Figure 4: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 6.6 (low concentration)

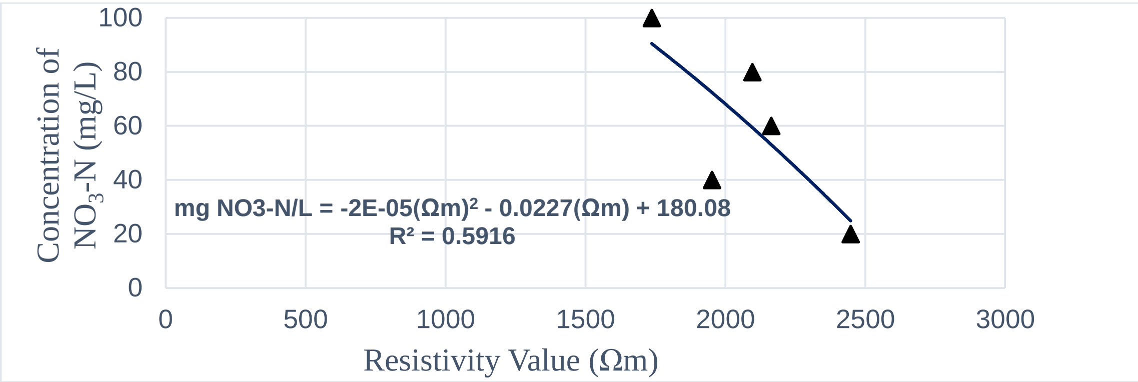

Figure 5: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 6.6 (medium concentration)

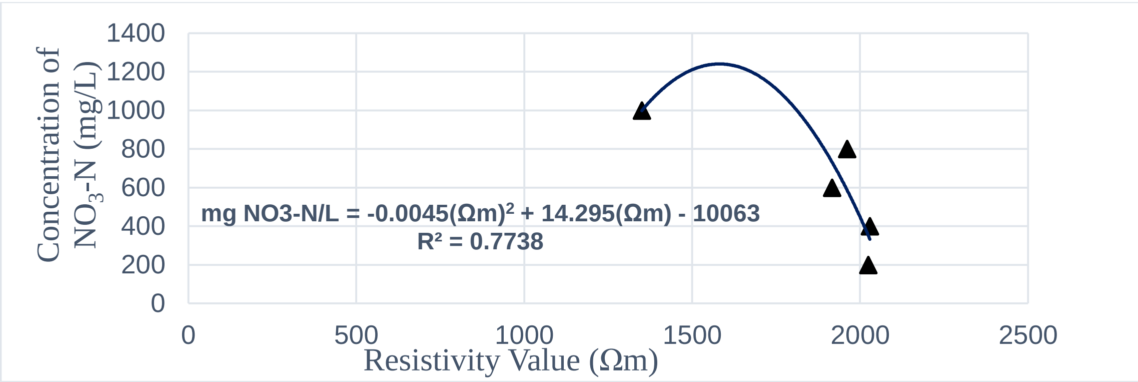

Figure 6: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 6.6 (high concentration)

3.1.3 pH 6.8

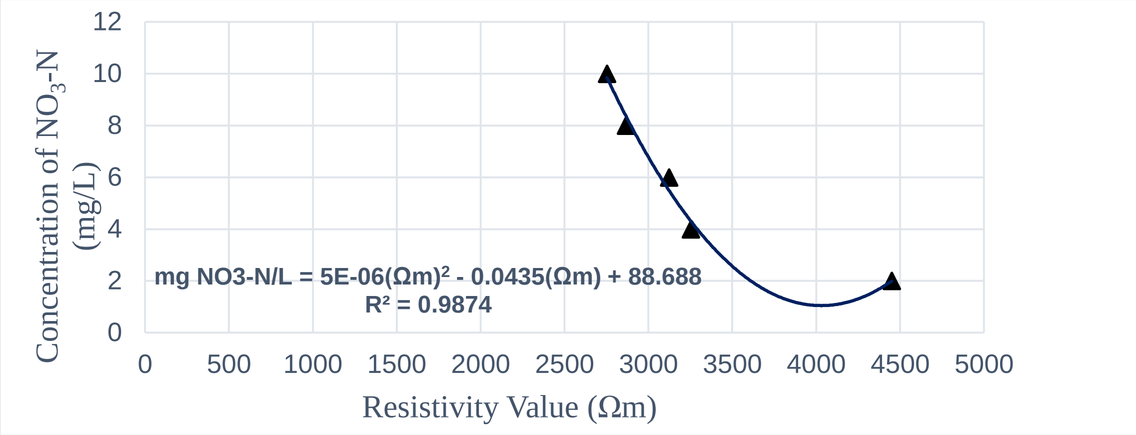

Figure 7: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 6.8 (low concentration)

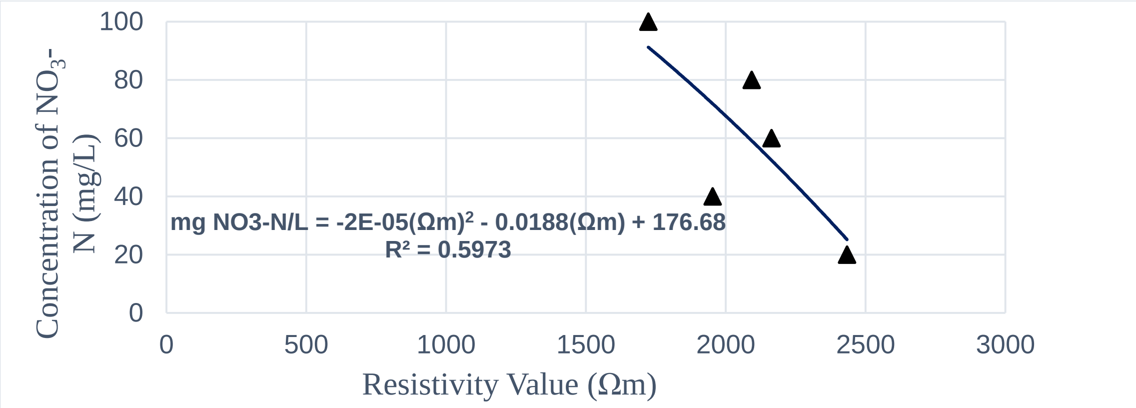

Figure 8: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 6.8 (medium concentration)

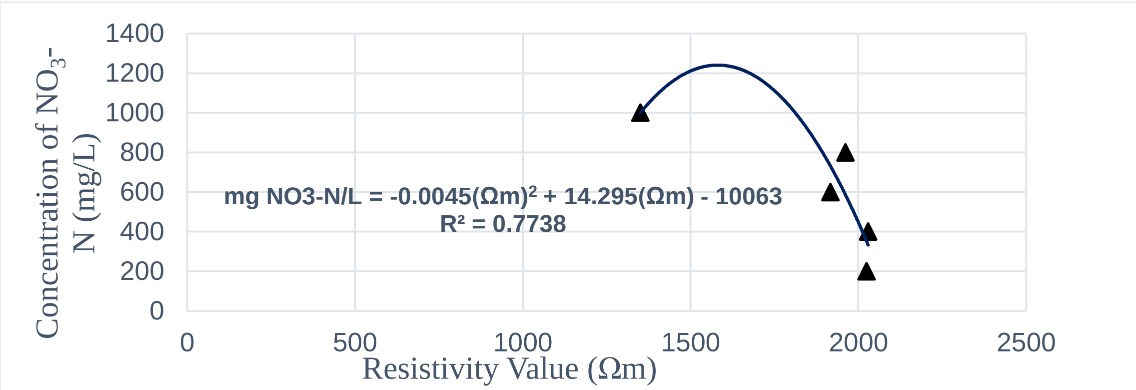

Figure 9: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 6.8 (high concentration)

3.1.4 pH 7.0

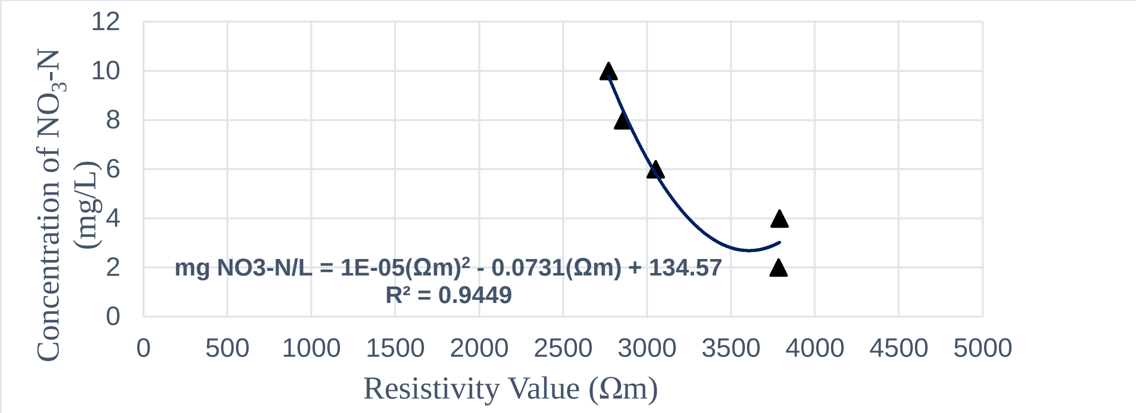

Figure 10: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 7.0 (low concentration)

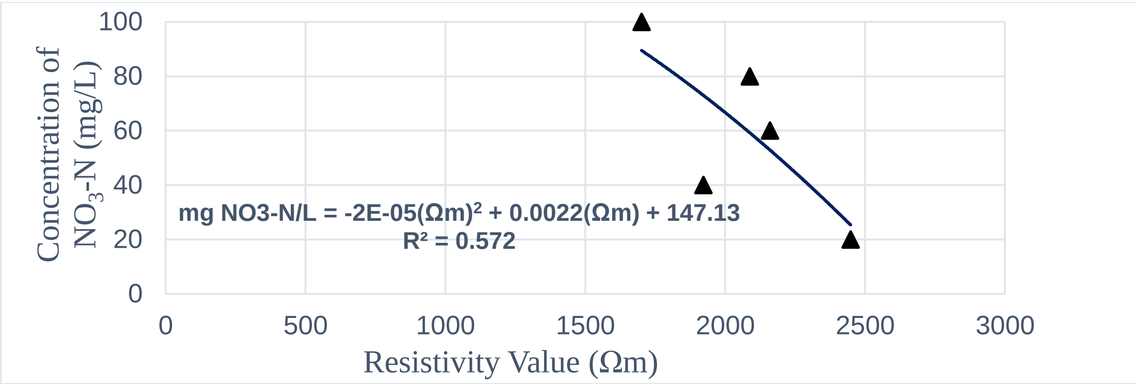

Figure 11: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 7.0 (medium concentration)

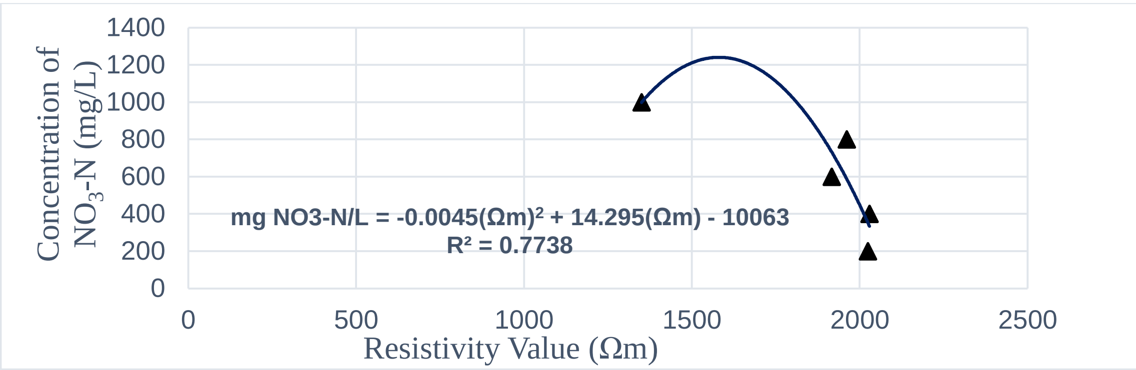

Figure 12: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 7.0 (high concentration)

3.1.5 pH 7.2

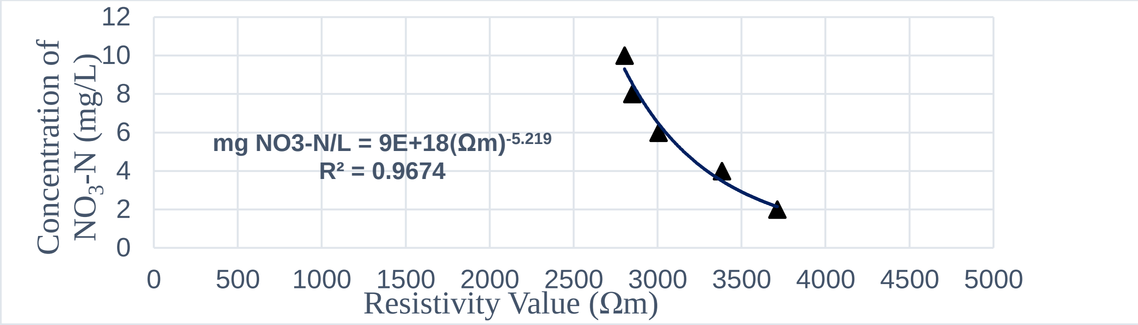

Figure 13: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 7.2 (low concentration)

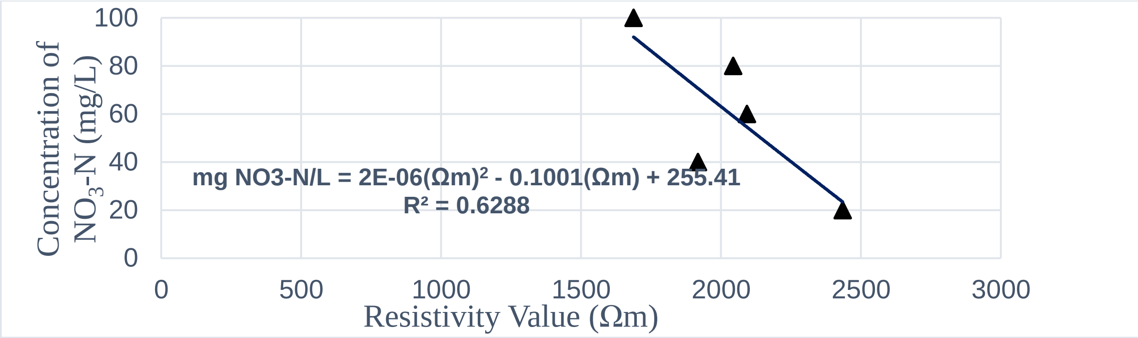

Figure 14: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 7.2 (medium concentration)

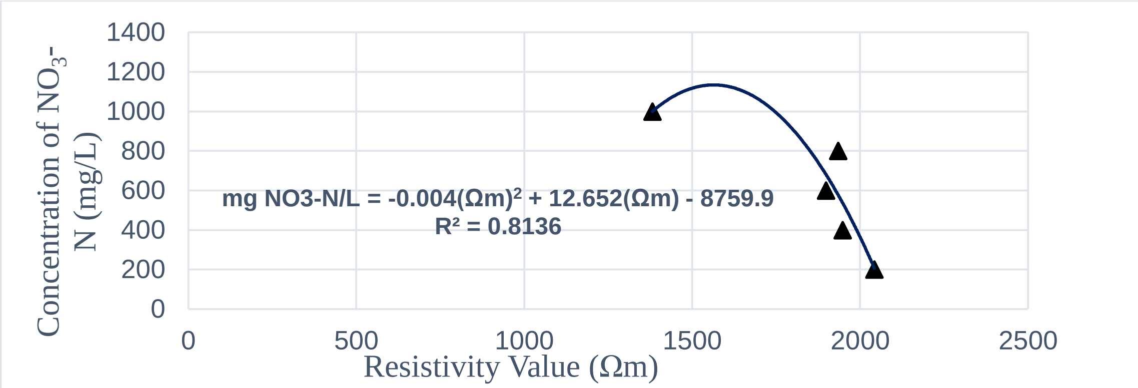

Figure 15: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 7.2 (high concentration)

3.1.6 pH 7.4

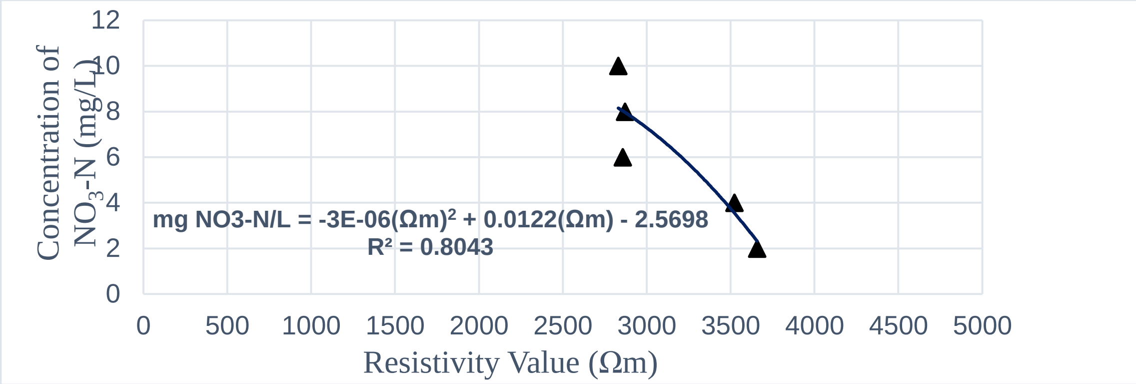

Figure 16: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 7.4 (low concentration)

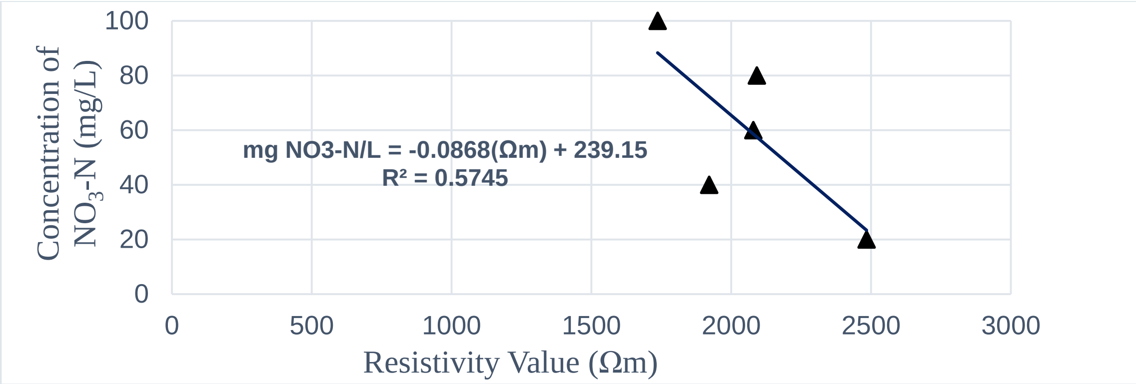

Figure 17: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 7.4 (medium concentration)

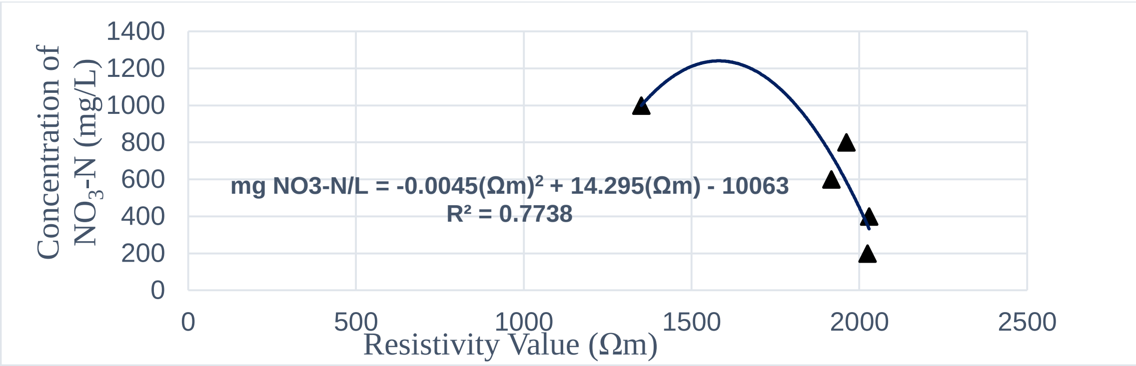

Figure 18: Calibration curve of Nitrate-Nitrogen concentration against resistivity value at pH 7.4 (high concentration)

Based on the previous researchers finding by [2] which researching on soil moisture with laboratory resistivity, it was found that the correlation shows a curvilinear trendline. Therefore, from the Figure 1 to Figure 18, most of the calibration curve were showing a polynomial trendline function where it is illustrated the R^2 reading that is higher compared to the others function; power, linear, logarithmic, and exponential. Besides, from previous findings stated above, the value of moisture content will increase as the soil resistivity value decrease. This is because, when over saturated dry soil was continuously added with water, the flow of current dissemination in soil were increased, thus resulting from high to low resistivity value. Low moisture content in soil makes the ions in pore fluid hardly to disseminate, thus resulting in low soil conductivity and high resistivity value. From previous phenomenon which related in field resistivity, dry condition will always resulting erroneous observations. This proves the important of water when applying for the soil box resistivity method [2]. This condition can be applied with nitrate-nitrogen concentration against the electrical resistivity correlation. The value of electrical resistivity is decreasing gradually with an increasing of the concentration solutions, thus proving that due to the increasing of the ions present it subsequently decreasing the electrical resistivity value since conductivity is the inversely proportional to resistivity. Hence, at higher concentration, more ions present in the solution makes electric current flow with ease. Overall, the R^2 value from each of calibration curve that were generated shows closer to 1.000 although slightly vary result were observed in medium concentration range which related to the possibility of the neutral solution that existed in it as the R^2 was showing less than 0.6500.

4. Conclusion

From It can be concluded that the laboratory experiment of soil box resistivity test value has been successfully conducted. The conclusions can be concluded based on the three (3) objectives stated. Hence, for the first objective is to determine the electrical resistivity value of nitrate-nitrogen concentration at neutral range. The value of electrical resistivity obtained which took the consideration within the range of neutral pH of 6.4, 6.6, 6.8, 7.0, 7.2, and 7.4. The pH adjustment was using two (2) kind of solution which is H_2SO_4 and NaOH. Next, the second objective for this study is to determine the electrical resistivity value at low, medium, and high Nitrate-Nitrogen concentration. It's range of concentration of solutions were sorted out from low, medium and high and its sequence were listed with systematically so that the data were properly sorted. The third objective that has been achieved is to generate the calibration curve between Nitrate-Nitrogen and resistivity value based on the objective (1) and (2) data. Thus, as shown in the Figure 3.1 to Figure 3.18, the curves that were generated showing the relationship between the concentration of solution within certain range and the value of electrical resistivity at neutral pH. The graph of calibration curve was interpreted by the legend indicators at the bottom with grid and colours. This to ensure that the information was clear when the calibration curve need to be referred.

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