International Journal of Advanced Biochemistry Research

ISSN Print: 2617-4693
ISSN Online: 2617-4707
IJABR 2024; 8(4): 539-544
www.biochemjournal.com
Received: 09-01-2024
Accepted: 19-03-2024

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Exploration of electrode materials for in-situ soil fertilizer concentration measurement by electrochemical method

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DOI: https://doi.org/10.33545/26174693.2024.v8.i4g.1011

Abstract
The electrochemical methodology investigates the manner in which chemical and electrical effects interact with one another, focusing on the chemical processes that produce electrical energy as well as the chemical changes carried out by the movement of electricity. The determination of soil fertilizer levels is a significant use for this technology. Three distinct electrode materials were examined in this work: graphite, aluminum, and silver, at various concentrations and nutritional combinations. The open circuit method was used to experiment. Samples of soil were taken from the experimental site, an artificial paddy field constructed out of concrete. The samples were immersed in electrolytes, with electrodes serving as the anode and cathode. The recorded voltage was measured using a digital multimeter, and nutrient levels in soil samples were calibrated using a polynomial equation. We next determined the pH values for every fertilizer concentration using a digital pH meter. Four plots, one in the field and one for the control group without fertilizer, were used for the experiment. The amount of fertilizer progressively decreased over time. The voltage rose and then gradually decreased as fertilizer was applied. All three electrodes satisfactorily predicted the soil fertilizer content in the field. The pH values that were measured ranged from 4.5 (lowest) to 7.7 (highest). This work demonstrates that it is possible to identify macronutrients in soil using a handheld, user-friendly electrode-based instrument. Furthermore, the choice of electrode material had a significant impact on repeatability, stability, and corrosion resistance. Only silver electrodes performed satisfactorily in terms of repeatability and lack of clogging impact among the materials examined.

Keywords: Electrochemical approach, Soil fertilizer concentration, Nutrient monitoring, Portable electrode device, In-situ sensing

1. Introduction

Like people and animals, plants need to be fed, have access to water, and be protected from pests and diseases in order to succeed. Commercial fertilizers provide vital minerals in precise amounts that plants can effectively absorb and use for growth and development, including nitrogen (N), phosphate (P), potassium (K), iron (Fe), zinc (Zn), copper (Cu), and boron (B) [1]. Fertilizers are essential for boosting crop yields, growth rates, and soil productivity since they contain nitrogen, phosphorus, and potassium. India and other developing nations have seen increases in agricultural output because of chemical fertilizers. India is now among the biggest producers and consumers of fertilizer worldwide as a result. However, because of inefficient nutrient consumption, leaching, and evaporation, overuse of synthetic agrochemicals has resulted in persistent toxicity and environmental damage [2, 3]. To preserve agricultural crop productivity while reducing the adverse impacts of fertilization, investigators are exploring other ways to maintain soil fertility. Using organic fertilizers like compost, which not only give the soil vital nutrients but also enhance its structure and capacity to hold water, serves as an example of a sustainable replacement. Additionally, by decreasing the need for chemical inputs and fostering natural nutrient cycle processes, techniques like crop rotation and cover crops can support the preservation of soil fertility and health. Soil analysis findings are impacted by depth-dependent fluctuations in soil nutrient levels [4]. Assessing the appropriate depth for the sample is the aim of the soil test.

Before planting, soil should be sampled at a depth of 15 to 30 cm, covering the area of active root activity, to estimate the amount of fertilizer needed. When sampling perennial crops that receive frequent surface fertilization, a depth of 5 to 10 cm is sufficient. It could be necessary to use a variety of sampling depths to diagnose issues in orchards. For the purpose of making precise nutrient recommendations and directing fertilization techniques, soil testing is essential [5]. The overview of this article talks about the pros and cons of potentiometric electrochemical sensors, like ion-sensitive field-effect transistors (ISFETs) and ion-selective electrodes (ISEs), for finding NPK in soil.

The development of proximal soil sensors is essential to advancing precision agriculture and deepening our understanding of soil variability. Modern transportable proximal soil sensors have the benefit of being quick and inexpensive to measure for soil analysis [6]. On the other hand, there persists a need for the direct measurement of soil nutrient concentration. Ion-selective electrodes (ISEs) and ion-sensitive field-effect transistors (ISFETs) are two examples of electrochemical sensors that show promise for this application. A multi-ion measurement system (MIMMS) that can be utilized for mobile proximal soil nutrient sensing is still being developed by researchers, despite the technology not being fully there anymore [7]. An efficient ammonia sensor that is inexpensive, easy to use, and produces little pollution was developed by Zhang and Wei [8] for in-situ ammonia detection. Additional studies (Ramane, Patil [9]; Rubio and Gil-Sotres [10]; Jarosiewicz and Tomaszewska (11)) have examined sensor techniques to measure NPK concentrations in soil, emphasizing the significance of this information in improving crop fertility and soil quality.

The objective of the study is to evaluate the levels of nitrogen (N), phosphorus (P), and potassium (K) in soil using electrochemical methods in conjunction with conductivity testing. These elements are essential for recognizing soil fertility and maximizing crop growth. The main objective of the research is to determine the relationship between the electrical signals detected by electrochemical measurements and the amounts of nutrients by testing various electrode materials to see how well they detect NPK levels in soil. The research will also evaluate these electrodes in the lab and in the field. This study tries to assess these electrodes' effectiveness in the field and in lab environments, with a particular emphasis on how effectively they work with various crops. In order to ascertain which electrode material yields the most precise and trustworthy findings when detecting NPK levels in soil samples, researchers may explore several electrode materials in the future, including metal oxides, graphite, and carbon nanotubes. Conducting trials in both controlled laboratory and real-world agricultural settings could be helpful in determining how well these electrodes work for precisely monitoring nutrient amounts in various crop species. In order to enable precise fertilizer management based on crop requirements, the study also attempts to establish a quantifiable relationship between the levels of nutrients present in soil and the electrical signals obtained by electrochemical measurements. This will facilitate the management of fertilizer on-site in accordance with the crops' present needs. The goal of this extensive study is to aid in the creation of feasible and effective soil nutrient monitoring systems for precision agriculture, which has the potential to transform agricultural methods and increase crop yields.

2. Materials and Methods

2.1 Selection of an Experimental Site

The Department of Agricultural Engineering, Assam University, Silchar, was selected as an experimental site. Geographically, the location of the site is latitude 24.68˚N and longitude 92.75˚E. The selection of the site took topography, climate, and soil type into account. Clay loam soil, which is typical of rice agriculture areas, was identified as the soil type at the site. Paddy crops might thrive in the subtropical region because of its warm, humid weather and 2196 mm of yearly precipitation on average [12]. The site's relatively flat topography makes it perfect for paddy farming because it makes effective water management possible. The subtropical climate combined with the clay loam soil made the ideal growing conditions for paddy crops, which need a lot of moisture. The 2196 mm of precipitation on average per year also guaranteed that crop productivity would not be constrained by the availability of water. All things considered, the location was excellent for agricultural engineering research and paddy cultivation studies.

The test crop was chosen to be rice (*Oryza sativa*), also referred to as paddy, because of its importance in the area and extensive cultivation. The three main nutrients that are necessary for rice growth are nitrogen (N), phosphorus (P), and potassium (K). The individual nutritional requirements of the several growth phases of the rice plant were taken into consideration when designing nutrient management strategies. To ensure that the soil had the right amount of nutrients, these procedures included applying both organic and inorganic fertilizers [13]. Regular soil testing was also done to keep an eye on nutrient levels and modify fertilizer applications as necessary. Farmers were able to produce higher yields of superior rice through effective nutrient management, which enhanced the region's food security and economic growth. The effectiveness of these methods demonstrated how crucial it is to recognize and satisfy each crop's unique nutritional requirements in order to optimize agricultural output.

Fig 1: Stages of experimental plots

At the experimental site, concrete plots measuring two meters in length and width were built, with each plot having an area of four meters square, as illustrated in Fig. 1. According to the literature of Zakaria *et al*. (2021) [14], the plots were divided into sub-plots with four replications for each treatment, utilizing a randomized complete block design (RCBD). The sub-plots were given varying combinations of NPK nutrients and a control plot (one that didn't get any fertilizer). The control plot assisted in creating a standard against which to compare the impact of applying nutrients. Based on the findings of the soil test and the particular growth stages of the rice crop, the chosen NPK nutrients were applied to the corresponding sub-plots at the prescribed rates. The application rates adhered to best practices and instructions provided by the area agricultural extension. The techniques of Jørgensen and Jørgensen (2007) [15] were followed while filling the concrete plots with soil from the adjoining rice field, with care given to maintain uniformity in the application of fertilizers.

2.2 Soil property

Before applying fertilizer to each sub-plot and at regular intervals during the crop's growth phases, soil samples were taken. Soil samples were taken for each sampling occasion from several randomly chosen places within the sub-plot, and the samples were combined to create a composite sample. After being gathered, the soil samples were labeled, cleaned, and brought to the lab for additional examination. The soil samples were air-dried in the lab and then sieved to get rid of any debris. Using the usual laboratory techniques of Liao *et al.* (2011) [16], a number of the soil's physicochemical characteristics, including pH, organic matter content, cation exchange capacity (CEC), and nutrient levels, were examined. A pH meter was used to assess the pH of the soil, and Järvan, Vettik [17] used standard chemical extraction techniques to measure the amount of nutrients present.

2.3 Development of an electrochemical setup

The electrochemical method of measuring soil fertilizer content was developed using electrodes and a pH meter. The literature of Tilman *et al.* (2002) [2] recommended the electrodes constructed of graphite, aluminum, and silver (Fig. 2) due to their conductivity and stability under soil conditions. With an exact thickness of 3 mm for graphite, 2 mm for aluminum, and 1 mm for silver, each electrode measured 20 mm by 50 mm. The electrodes were placed in pairs and held in place with plastic glue and epoxy, with a 10 mm insulating space between them. To measure the open circuit voltage, titanium wire was used to link them to an electrical circuit. A pH meter was used to measure the hydrogen-ion activity in the soil solution, which was a necessary step for electrochemical analyses.

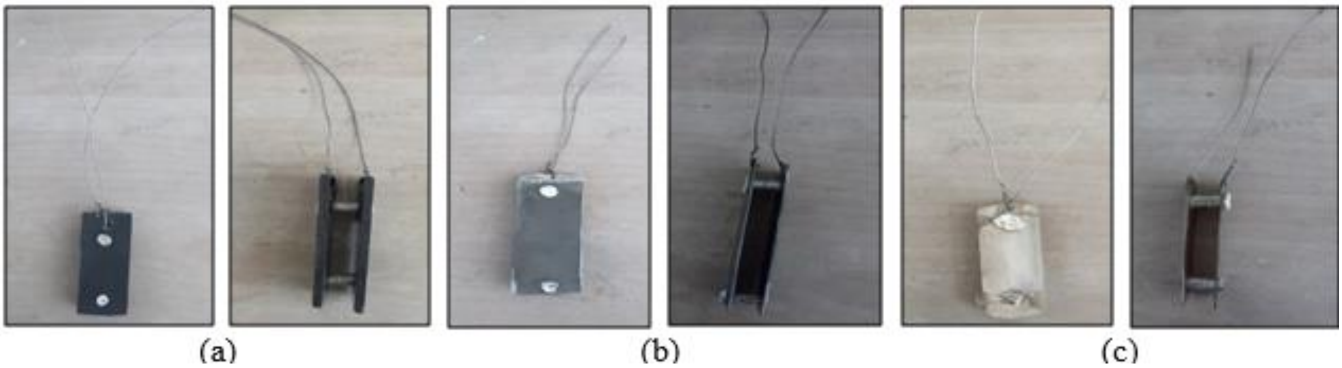

Fig 2: Electrodes: (a) graphite, (b) aluminium, and (c) silver

The electrodes were appropriately calibrated using standard solutions with established pH and nutrient contents before each measurement, in accordance with Kim and Hummel's protocol (17). Throughout the electrochemical examination, the calibration guaranteed precise and reliable readings. To avoid cross-contamination between soil samples, the electrodes were also meticulously washed and rinsed with deionized water (Fig. 3). The aluminum electrode was picked for its conductivity and price, while the graphite electrode was chosen for its longevity and stability. Because of its great sensitivity to nutrient concentrations, the silver electrode was incorporated. Any possible faults or inaccuracies were reduced by calibrating and cleaning the electrodes before each test, enabling accurate and dependable results from the electrochemical examination of the soil samples. Overall, the success of the experiment was assured by the careful selection and maintenance of the electrodes.

Fig 3: Electrode set-up and calibration for concentration measurement of NPK

2.4 Electrochemical measurement of soil fertilizer concentration: The potential difference (voltage) between the soil sample and the reference electrode was measured using the electrochemical setup (Fig. 3). For statistical analysis and result reliability, the potential difference was recorded for every soil sample, and several readings were obtained. The known amounts of NPK nutrients discovered through the physicochemical analysis of soil samples were associated with the observed potential differences. The calibration of the electrode configuration for determining the amount of NPK nutrients in soil samples was made possible by this correlation. A calibration curve was developed to precisely ascertain the NPK concentration in the soil by analyzing the possible variations with the known amounts. This technique made it possible to monitor soil fertility and nutrient levels quickly and effectively, which aided in the optimization of fertilizer application for crop growth and production.

3. Results and Discussion

3.1 Soil physical characteristics

Using a variety of standard instruments and Chaudhari *et al.* [18] methodologies, the physical parameters of the soil, including its bulk density, moisture content, and texture, were examined for varying depths in the experimental site, which was a concrete plot. Using the hydrometer method [19], it was possible to assess the depth-wise soil texture status of the experimental plot. At a depth of 0 to 10 cm, the soil type was silt loam; at 10 to 20 cm, it was sandy loam; and at 20 to 30 cm, it was clay loam and loam. The soil was determined to be clay loam and loam, respectively, based on the USDA textural categorization triangle. An important factor in affecting the soil's ability to retain water and the availability of nutrients is its texture. Crop growth performance and soil productivity can be impacted by the differing composition of sand, silt, and clay at different depths. Making educated judgments about irrigation, fertilizing, and general soil management techniques to maximize agricultural yield in the experimental plot requires an understanding of the soil texture. By employing the oven drying method and the standard equation [20], the moisture content analysis was completed. The association between soil nutrition level and moisture content was important during the ripening stage, when irrigation was discontinued, even though during the crop growth stage, saturated soil moisture was maintained and had little importance.

3.2 Soil chemical characteristics

The chemical properties of the soil, including pH, voltage (mV), and fertilizer concentration (g/m2), were measured at the experimental site using slightly modified Rayment and Lyons [17] techniques. However, the study took into account the total nitrogen, phosphorus, and potassium soil nutrient content statuses of 16.5, 62.4, and 4.4 g/m2, respectively [18]. The findings demonstrated that, with a standard variation of 0.25 to 1.25, the average moisture content in the various plots varied from 1.97 to 2.46. With minor adjustments, the established protocols for analyzing soil chemical properties were followed when determining the soil's pH, voltage, and fertilizer content. Furthermore, as stated in earlier research, the soil nutrient content status of total nitrogen, phosphorous, and potassium was determined to be 16.5, 62.4, and 4.4 g/m2, respectively.

3.3 Calibration curve

Using pair electrodes (graphite, aluminum, and silver) and a multimeter, respectively, the "open circuit method" [19] was used to calculate the fertilizer concentration for soil profiles. Soil samples were combined with a predetermined quantity of N, P, and K, and the beaker's calibration process involved maintaining saturated soil moisture levels. Table 1 displays the calibration data for the graphite, aluminum, and silver electrodes for N, P, and K. (Fig. 4) displays the curve equation. The electrical response of each electrode to known concentrations of potassium, phosphorus, and nitrogen in solution was measured as part of the calibration procedure. The calibration curves that were produced made it possible to precisely determine the quantities of fertilizer in soil profiles, providing exact nutrient management for the best possible plant growth.

Table 1: Calibration of graphite, aluminium, and silver electrodes (NPK)

Conc. (g)	Graphite (N)		Graphite (P)		Graphite (K)		Aluminum (N)		Aluminum (P)		Aluminum (K)		Silver (N)		Silver (P)		Silver (K)	
	Voltage (mV)	pH	Voltage (mV)	pH	Voltage (mV)	pH	Voltage (mV)	pH	Voltage (mV)	pH	Voltage (mV)	pH	Voltage (mV)	pH	Voltage (mV)	pH	Voltage (mV)	pH
1	60	7.5	39.41	4.8	-11.3	7.4	-1.42	6.9	104.5	6.6	102.8	7.5	1.8	5.4	25.6	4.8	118.5	6
2	57.5	7.3	42.65	4.9	-9.7	7.3	-1.51	6.7	104.4	6.5	100.3	7.4	1.46	5.6	25.3	4.8	113	6.1
3	44.48	7.7	36.1	5	-9.65	7.2	-1.62	6.2	103.8	6.4	99.2	7.4	1.02	6	25	4.9	103.7	6.2
4	35.09	7.6	29.18	5.1	-9.62	7.2	-1.82	6.1	103.7	6.2	80.2	7.1	-1.7	6.1	24.82	5	82.7	6.5
5	28.32	7.1	24.44	5.2	-10.45	7.1	-1.9	6	103.2	5.9	72.3	6.9	-1.99	6.4	24.76	5.2	81.6	6.7
6	25.32	6	19.68	5.4	-8.76	7	-2.7	5.9	102.2	5.7	62.3	6.5	-1.86	6.7	23.24	5.3	87.5	6.8
7	20.98	5.8	16.49	5.6	-6.54	6.9	-2.79	5.7	100.2	5.5	52.6	6.7	-1.98	7	23	5.4	67.8	6.9
8	17.53	5.9	14.15	5.7	-3.82	6.8	-2.82	5.6	100	5.4	50.1	6.9	-1.83	7.1	22.82	5.6	61.3	7.1
9	16.53	5.8	12.01	5.8	-3.03	6.7	-3.12	5.5	90.3	5.3	40.2	6.8	-2.01	7.3	22.3	5.7	58.12	7
10	14.98	5.9	9.92	5.9	-3.69	6.5	-3.31	5.3	85.5	5.2	38.2	6.5	-2.15	7.2	21.9	5.9	29.6	7.3
11	15.27	5.8	8.79	6	-0.2	6.4	-3.42	5.2	80.2	5.2	35.4	6.4	-2.3	7.5	21.6	6	30.58	7.4
12	13.46	5.6	7.32	6.1	-0.58	6.3	-3.53	5.2	75.55	5.1	30.2	6.2	-2.7	7.7	21.2	6.2	27.04	7.6
13	11.3	5.6	7.11	6.2	-0.22	6.2	-3.59	5.1	60.5	5.2	28.6	6.1	-2.9	7.5	20.8	6.3	22.08	7.3
14	10.6	5.5	6.11	6.3	0.13	6.1	-3.71	5.5	55.8	5.2	25.2	6.1	-3.2	7.2	19.8	6.4	20.1	7.4
15	9.58	5.3	5.69	6	0.2	6	-3.82	5.4	54.5	5.1	22.3	6	-3.6	7.2	19.2	6.7	19.5	7.5
16	8.76	5.1	5.43	6.2	0.45	5.9	-3.91	5.8	53.5	5.1	20.6	6.1	-3.9	7.3	18.3	6.5	18.2	7.6

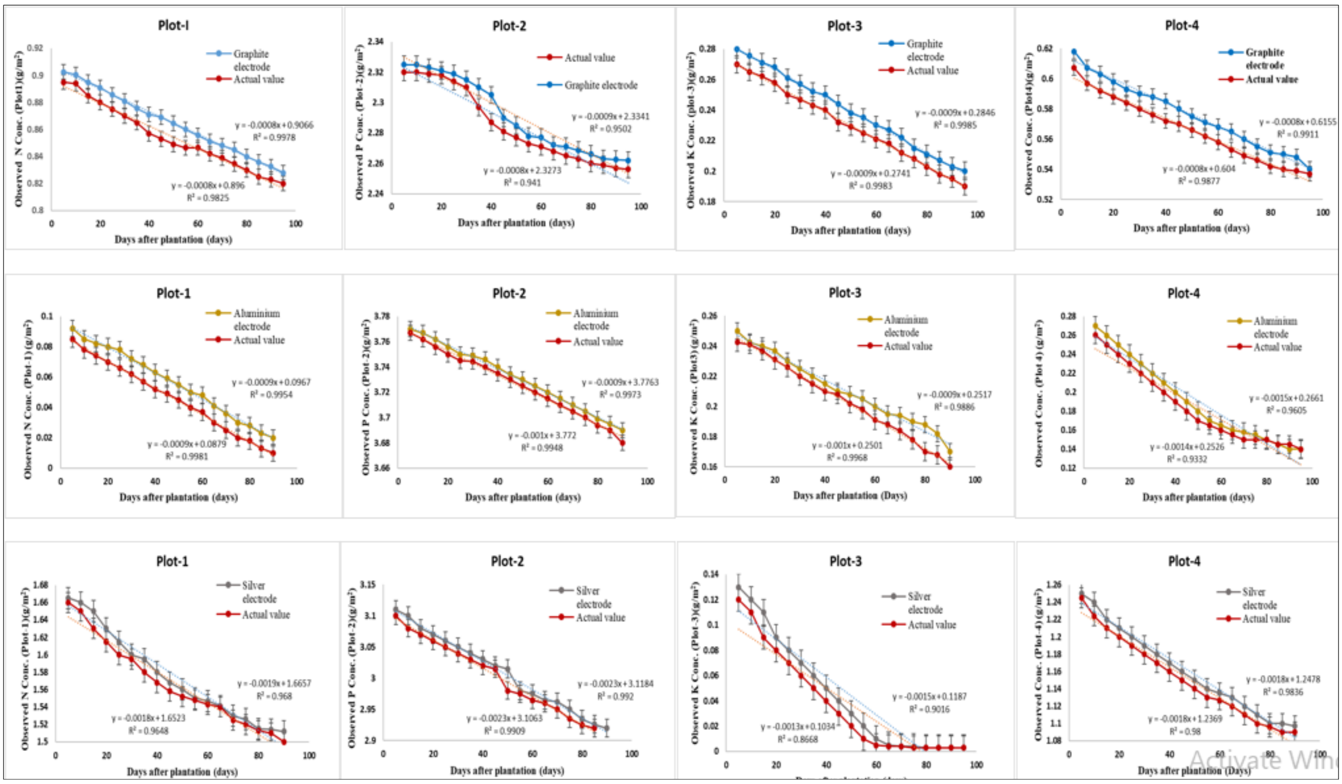

Fig 4: Comparison of fertilizer concentration inside the laboratory and on the field using (i) graphite electrodes, (ii) aluminium electrodes, and (iii) silver electrodes

3.4 Measurement of fertilizer concentration

The electrochemical approach was used to conduct the experiment in four plots: Plot 1 (N), Plot 2 (P), Plot 3 (K), and Plot 4 both in the field and at the experimental site within the laboratory. It has been seen that the concentration is getting lower every day. Additionally, it was noted that the voltage first increases during the fertilizer application process before gradually declining. The pH was found to range from 4.5 (lowest) to 7.7 (highest). Following the acquisition of the known concentration, the actual value measured in the laboratory is compared. Fig. 4 shows the comparison of fertilizer concentrations.

3.5 Comparison of fertilizer concentrations for different electrodes

For assessing NPK content, the calibration curves for graphite, aluminum, and silver showed consistency with a linear fit equation and an R^2 value ranging from 0.886 to 0.998. With a tiny discrepancy between the actual value achieved in the lab and the known amount, the real concentration measurement was almost exactly in line with the calibrated curve. This discrepancy could be caused by a variety of reasons, such as application methods, soil composition, and environmental conditions. Further research is required to determine the measurement's accuracy and identify any potential sources of error in the data collection process. Moreover, the range of pH values from 4.5 to 7.7 points to a dynamic environment that could influence the efficiency of fertilizer application. In each instance, the observed open circuit voltage trended downward, and the pH level decreased as fertilizer concentration increased.

The best-fit curve in Fig. 4 (R^2 = 0.9981, 0.9948, and 0.9968) suggests that the aluminum electrode was more suitable for measuring the concentrations of potassium, phosphorus, and nitrogen. The graphite electrode was suitable for measuring potassium (R^2 value of 0.9983), while the silver electrode showed adequate consistency with an R^2 value of 0.9909 for detecting phosphorous concentration. It was also observed that the plant routinely absorbed the nutrients and that the concentration was measured with reasonable accuracy. Plot-4 fertilizer concentration, on the other hand, varied marginally for aluminum electrodes but exhibited a similar pattern for graphite and silver electrodes. This suggests that this inexpensive approach to detecting fertilizer concentration can be used to approximate the concentration of fertilizer in the soil. It was also observed that soil microbial activity led to the formation of a thicker biofilm on the graphite electrode. The silver electrode held up well even after several days of use, while the aluminum electrode needed regular cleaning due to rust. It is evident that silver is a superior electrode material; however, more investigation is required for the closed-circuit study.

4. Conclusion

Nutrient levels gradually decreased during fertilization, as shown by the electrode calibration and in-situ validation. This allowed for the creation of an approximate method for determining the amount of nutrients (NPK) in the soil. The concentrations of phosphorous and silver were successfully measured by the graphite electrode, while the concentrations of phosphorous, silver, and potassium were successfully measured by the aluminum electrode. As a whole, the study made clear how important it is to understand the complexities of soil composition and how they may affect soil treatment's function. Researchers can efficiently track changes in nutrient levels over time by calibrating silver electrodes, which provides important data for the use of sustainable soil management techniques. Further research on the long-term effects of different soil treatments on soil health and nutrient levels is necessary in order to optimize agricultural output and guarantee environmental sustainability. The results highlight how important it is to classify soil in order to regulate fertilizer and apply sustainable agricultural techniques. The information obtained from this study is a useful tool for managing crops and soil in an informed manner. In order to improve its broader application, more research on a range of soil types and climates is advised. To evaluate the long-term impacts of interventions on crop productivity and soil health, ongoing monitoring is crucial. All things considered, this study offers insightful information about improving environmental sustainability and securing food security.

5. Statements and Declarations

The authors would like to state that there is no conflict of interest and that no funding has been invested in this research.

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