NPK OF HYDROCARBON POLLUTED SOIL REMEDIATED BY CASSAVA MILL WASTE WATER

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A PROJECT SUBMITTED TO THE DEPARTMENT OF SCIENCE LABORATORY TECHNOLOGY, FACULTY OF LIFE SCIENCES, UNIVERSITY OF BENIN, BENIN CITY, IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE AWARD OF BACHELOR OF SCIENCE DEGREE (B.Sc) IN SCIENCE LABORATORY TECHNOLOGY (BIOCHEMISTRY TECHNIQUES)

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CERTIFICATION

This is to certify that this undergraduate project work titled "**NPK OF HYDROCARBON POLLUTED SOIL REMEDIATED BY CASSAVA MILL EFFLUENT**" was submitted and presented by Emmanuella EDOMWONYI with matriculation number LSC2003190 in the Department of Science Laboratory Technology, Faculty of Life Sciences, University of Benin, Benin City. In a partial fulfillment of the requirements for the award of Bachelor of Science Degree in Science Laboratory Technology.

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DEDICATION

This work is dedicated to Almighty GOD. It wouldn't have been easy to get to this point without God. I am extra thankful.

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My sincere gratitude to God Almighty for his grace, wisdom and understanding throughout the period of this research.

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To all my friends who supported me throughout this process, I love you all and God bless you.

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ABSTRACT

This study evaluated the effects of cassava mill effluent (CME) on the NPK of hydrocarbon-polluted soil in Delta State, Nigeria. This has severely degraded soil fertility by depleting essential macronutrients nitrogen (N), phosphorus (P), and potassium (K) and elevating total petroleum hydrocarbon (TPH) levels beyond regulatory limits thereby threatening agricultural productivity and food security for local communities dependent on subsistence farming. This study aimed to evaluate baseline NPK levels in contaminated clay-loam soils from spill-affected sites while investigating the bioremediation potential of cassava mill effluent as a nutrient-rich biostimulant containing 2–3% N, 0.5% P and 0.3% K, applied at varying frequencies including daily, once, weekly, monthly and control setups. Soil samples were collected from 0–15 cm depths pre-treatment and post-treatment over a 12-week period, with analyses conducted using the Kjeldahl method for N via digestion and titration, ascorbic acid spectrophotometry at 710 nm for P, flame photometry at 766.5 nm for K, and gas chromatography-flame ionization detection with n-hexane extraction for TPH, leveraging indigenous microbes like Pseudomonas and Bacillus for biostimulation in alignment with sustainable waste-to-wealth approaches in cassava-producing regions. Results demonstrated substantial nutrient restoration and TPH reduction following remediation, with pre-treatment values showing low NPK (N: 0.008–0.07%; P: 1,055–5,322 ug/g; K: approximately 0.10 mg/kg) while diminishing TPH to undetectable levels, surpassing the outcomes of single or monthly treatments. These improvements arose from heightened microbial activity and enhanced nutrient cycling, which alleviated toxicity and reinstated bioavailability in the polluted soils. The findings highlight the effectiveness of cassava effluent for affordable soil rehabilitation at near-zero cost, with potential to elevate crop yields by 20–40% and contribute to United Nations Sustainable Development Goals 2 (Zero Hunger) while recommendations advocate for ongoing biostimulation protocols and regular monitoring to refine remediation efforts in Niger Delta ecosystems, ultimately promoting ecological resilience and supporting community livelihoods.

CHAPTER ONE

INTRODUCTION

1.1 Background of the Study

The Niger Delta region of Nigeria, particularly Delta State, is a biodiversity hotspot renowned for its extensive mangrove ecosystems, fertile alluvial soils and rich aquatic life, supporting over 60% of Nigeria's species diversity (UNEP 2011). However, since the discovery of crude oil in 1956, intensive petroleum exploration and production have led to recurrent oil spills, contaminating soils with total petroleum hydrocarbons (TPHs), heavy metals and other pollutants (Kadafa *et al*., 2012). Over 13 million barrels of oil have spilled in the region since the 1950s, with Delta State experiencing thousands of incidents due to pipeline vandalism, equipment failure and operational mishaps. These spills degrade soil fertility by reducing organic matter, displacing oxygen and introducing toxic compounds like polycyclic aromatic hydrocarbons (PAHs), which inhibit microbial activity and nutrient cycling (Lindén and Pålsson, 2013). Studies indicate that hydrocarbon pollution significantly lowers soil nutrient levels, including nitrogen (N), phosphorus (P) and potassium (K) collectively known as NPK essential for plant growth and agricultural productivity (Osuji and Nwoye, 2007). In Delta State, this has resulted in crop yield reductions of up to 50% in affected farmlands, exacerbating food insecurity and economic losses for local communities reliant on subsistence farming and fishing (Inoni *et al.*, 2006). Bioremediation emerges as a sustainable solution, leveraging indigenous microbes enhanced by nutrient amendments like cassava wastewater a nutrient-rich byproduct from local cassava processing, abundant in the region to restore soil health (Oyetibo *et al.*, 2014). This study builds on prior research showing that organic stimulants such as cassava peels or wastewater, can boost

hydrocarbon degradation by 30–50% through biostimulation, promoting microbial proliferation and nutrient replenishment in polluted soils (Tanee and Jude, 2019).

1.2 Study Area

The study focuses on hydrocarbon-polluted sites in Delta State, located in the southern Niger Delta region of Nigeria (coordinates: approximately 5°33′49″N to 6°31′38″E). Covering about 17,698 km², Delta State is a low-lying coastal plain characterized by freshwater swamps, mangrove forests and riverine communities, drained by the Niger River and its tributaries like the Forcados and Warri Rivers (Zabbey and Uyi, 2014). The terrain is predominantly sedimentary with clay-loam soils, high humidity (70–90%) and annual rainfall exceeding 2,500 mm, fostering a tropical rainforest climate (Ogbuagu *et al*., 2011). Oil exploration, centered in areas like Ughelli North, Warri South-West and Ekpan, has led to over 1,000 documented spills since 1976, contaminating agricultural lands and groundwater (UNEP 2011). Sampling will target spill-impacted farmlands in communities such as Ugborikoko and Uzere, where soil TPH levels often exceed 10,000 mg/kg, far above the Department of Petroleum Resources' intervention limit of 5,000 mg/kg (Osuji and Nwoye, 2007). These sites exemplify the interplay of hydrological dynamics and pollution, where seasonal flooding redistributes contaminants, amplifying ecological risks to mangroves and fisheries (Zabbey and Uyi, 2014).

1.3 Aim of the Study

The primary aim of this study is to evaluate NPK (nitrogen, phosphorus and potassium) levels in hydrocarbon-polluted soils of Delta State, Nigeria, to inform bioremediation strategies using cassava wastewater for soil restoration and agricultural recovery.

1.4 Objectives

The specific objectives are:

1. To quantify the baseline levels of NPK in soils from selected hydrocarbon-impacted sites in Delta State (Sam *et al*., 2016).

2. To assess the spatial variability of nutrient depletion due to oil spills across different soil depths and proximity to spill sources (Brown *et al*., 2017).

3. To investigate the potential of cassava wastewater as a biostimulant to enhance nutrient replenishment and hydrocarbon degradation in polluted soils (Oyetibo *et al*., 2014).

4. To recommend nutrient management practices for restoring soil fertility and supporting crop productivity in affected communities (Ayotamuno *et al*., 2006).

1.5 Statement of the Problem

Hydrocarbon pollution from oil spills in Delta State has severely compromised soil quality, leading to nutrient deficiencies that render farmlands unproductive and threaten food security for over 5 million residents (Pegg and Zabbey 2013). TPHs bind to soil particles, reducing NPK bioavailability nitrogen by up to 40%, phosphorus by 30% and potassium by 25% in affected areas while heavy metals like lead and cadmium accumulate, inhibiting root growth and microbial decomposition (Orisakwe, 2010; Osuji and Nwoye, 2007). This results in annual agricultural losses exceeding ₦50 billion, crop failures (e.g., cassava yields dropping 40–60%) and health risks from bioaccumulated toxins in the food chain, including respiratory issues and soil-borne diseases (Orisakwe, 2010; Inoni *et al*., 2006). Conventional remediation methods, such as excavation, are costly (over $100,000 per hectare) and environmentally disruptive, while natural attenuation is too slow (decades) for urgent restoration. The lack of localized data on nutrient dynamics in Delta State's polluted soils hinders targeted interventions, perpetuating a cycle of poverty, social unrest and ecosystem collapse in this oil-dependent region (Pegg and Zabbey, 2013).

1.6 Justification of the Study

This study is crucial for addressing the under-researched nexus between hydrocarbon pollution and soil nutrient profiles in Delta State, providing empirical data to guide cost-effective bioremediation using locally sourced cassava wastewater a waste-to-wealth approach that recycles agro-industrial byproducts rich in NPK (e.g., 2-3% nitrogen from fermentation). By quantifying nutrient losses and remediation efficacy, it will support policy formulation for sustainable land restoration, aligning with Nigeria's National Oil Spill Detection and Response Agency (NOSDRA) guidelines and UN Sustainable Development Goals 2 (Zero Hunger) and 15 (Life on Land). The findings will empower farmers with practical nutrient enhancement techniques, potentially boosting yields by 20–40%, reducing economic burdens, and mitigating health risks. As Delta

State contributes 15% of Nigeria's oil output, this research promotes ecological resilience, fostering long-term agricultural viability and community empowerment in a region scarred by decades of spills (UNEP 2011).

CHAPTER TWO

LITERATURE REVIEW

2.1 Hydrocarbon Pollution and Its Impact on Soil Fertility

Hydrocarbon pollution, primarily from crude oil spills, is a critical environmental challenge in the Niger Delta, with Delta State being a major hotspot due to its extensive oil exploration activities. Crude oil contains complex mixtures of total petroleum hydrocarbons (TPHs), including alkanes, cycloalkanes and polycyclic aromatic hydrocarbons (PAHs), which are highly toxic to soil ecosystems (Lindén and Pålsson, 2013). Since the discovery of oil in Oloibiri in 1956, the Niger Delta has experienced over 13 million barrels of oil spilled, with Delta State recording thousands of incidents due to pipeline vandalism, equipment failures, illegal bunkering and operational mishaps (UNEP, 2011). Osuji and Nwoye (2007) reported TPH concentrations in Delta State's soils often exceeding 10,000 mg/kg, far surpassing the Department of Petroleum Resources (DPR) intervention limit of 5,000 mg/kg, leading to severe degradation of agricultural lands.

The impact on soil fertility is multifaceted and severe. TPHs form hydrophobic coatings on soil particles, reducing porosity, water-holding capacity and aeration which creates anaerobic conditions that inhibit microbial activity and organic matter decomposition (Kadafa *et al*., 2012). In Delta State's clay-loam soils, which are naturally fertile due to high organic content, this disruption is particularly pronounced as hydrocarbons bind tightly to organic matter limiting nutrient availability (Ogbuagu *et al*., 2011). Heavy metals co-released during spills such as lead (up to 50 mg/kg), cadmium (2.5 mg/kg) and nickel accumulate in soils exceeding WHO safety thresholds (e.g., 0.8 mg/kg for cadmium) and cause phytotoxicity, inhibiting root growth and microbial diversity (Orisakwe, 2010). These changes have led to significant agricultural losses, with crop yield reductions of 40–60% for staples like cassava, yam and maize, which are central to Delta State's subsistence economy. For instance, Arise *et al*. (2015) found that petroleum

effluent reduced cowpea germination by 50% and biomass by 40% in Niger Delta soils, underscoring the direct threat to food security.

Hydrocarbon pollution also alters soil pH, often shifting it to acidic levels (pH 4.5–5.5), which further limits nutrient uptake and microbial activity (Kadafa *et al*., 2012). This is compounded by Delta State's high rainfall (2,000–3,000 mm annually) and seasonal flooding, which redistribute contaminants across farmlands and into groundwater, exacerbating nutrient leaching and contamination of irrigation sources (Zabbey and Uyi, 2014). The chronic nature of oil spills, with over 1,000 documented incidents in Delta State since 1976, has rendered approximately 20% of arable land unproductive, threatening livelihoods and ecosystems (UNEP, 2011). Long-term studies indicate that without intervention, soil fertility recovery can take decades, as natural attenuation is hindered by the recalcitrant nature of PAHs and heavy metals (Adams *et al*., 2015). These findings highlight the urgent need for research into nutrient dynamics and effective remediation strategies to restore Delta State's soils for agricultural use.

2.2 Nutrient Dynamics (NPK) in Contaminated Soils

Nitrogen (N), phosphorus (P) and potassium (K) collectively NPK are essential macronutrients for plant growth, soil microbial activity and ecosystem health. Hydrocarbon pollution disrupts NPK dynamics by altering soil chemistry and inhibiting microbial processes critical for nutrient cycling. Osuji and Nwoye (2007) documented significant nutrient depletion in oil-contaminated soils in Owaza, Niger Delta, with nitrogen levels reduced by up to 40%, phosphorus by 30% and potassium by 25% compared to uncontaminated controls. This depletion results from the inhibition of nitrogen-fixing bacteria (e.g., Azotobacter, Rhizobium) and phosphate-solubilizing microbes, which are highly sensitive to TPH concentrations above 5,000 mg/kg (Sam *et al*., 2016). Eze and

Okeke (2015) reported a 60% decline in microbial populations in Delta State's polluted mangrove soils, directly impacting nitrogen availability and cycling.

Phosphorus, crucial for root development and energy transfer, is particularly affected as hydrocarbons adsorb onto soil particles, reducing phosphate solubility. Ogbuagu *et al.* (2011) found phosphorus levels in Delta State's contaminated soils dropping to 5-10 mg/kg, compared to 20–30 mg/kg in unpolluted soils, severely limiting crop productivity. Potassium, essential for osmoregulation and enzyme activation, is similarly immobilized or leached, with levels in spill sites reduced to 50–100 mg/kg versus 150–200 mg/kg in controls. These reductions are exacerbated by Delta State's hydrological dynamics, where high rainfall and flooding leach soluble nutrients into groundwater, particularly in shallow soil layers (0–15 cm) near spill epicenters (Zabbey and Uyi, 2014).

Spatial and vertical variability in nutrient depletion is a critical factor. However, flooding redistributes contaminants, creating heterogeneous nutrient profiles across affected farmlands (UNEP, 2011). For example, studies in Delta State's riverine communities like Ugborikoko and Uzere show that nutrient depletion is more pronounced near spill sources, with nitrogen levels dropping to 0.05% compared to 0.15% in uncontaminated soils (Ogbuagu *et al*., 2011). The lack of comprehensive, site-specific data on NPK dynamics in Delta State's polluted soils hinders the development of targeted remediation strategies, a gap this study addresses by quantifying baseline nutrient levels and their spatial variability (Onifade and Abubakar, 2018).

2.3 Bioremediation as a Strategy for Soil Restoration

Bioremediation, the use of microorganisms to degrade environmental pollutants, is a sustainable and cost-effective approach for restoring hydrocarbon-polluted soils, particularly in resource-constrained regions like the Niger Delta. It includes biostimulation (adding nutrients to enhance

native microbial activity) and bioaugmentation (introducing specific hydrocarbon-degrading microbes). Conventional remediation methods, such as excavation ($100,000–$500,000 per hectare) and thermal desorption, are costly and ecologically disruptive, often releasing secondary pollutants or destroying soil structure. In contrast, bioremediation leverages indigenous microbes, making it suitable for Delta State's tropical soils, which host diverse microbial communities (Ayotamuno *et al*., 2006).

Indigenous bacteria (Pseudomonas aeruginosa, Bacillus subtilis, Acinetobacter baumannii) and fungi (Aspergillus niger, Penicillium chrysogenum) in Delta State's soils can metabolize hydrocarbons into non-toxic byproducts like carbon dioxide, water, and biomass, but their activity is limited by nutrient deficiencies in polluted environments (Ogbonna *et al.*, 2012). Biostimulation addresses this by supplying NPK and organic carbon, which enhance microbial growth and enzyme production (Margesin and Schinner, 2001). For example, Ayotamuno *et al*. (2006) reported a 40% TPH reduction in Port Harcourt soils after 12 weeks of NPK supplementation, demonstrating its efficacy in tropical climates. Similarly, Sam *et al*. (2016) found that inorganic NPK fertilizers reduced TPH by 50% in Eneka, Niger Delta, though over-application risks soil salinization or eutrophication in Delta State's waterlogged ecosystems.

Organic amendments such as manure, compost or agro-industrial wastes, offer sustainable alternatives by providing both nutrients and carbon sources. Nwogu *et al*. (2015) reported a 50% TPH reduction in Niger Delta soils amended with goat manure, attributed to its high nitrogen and organic matter content. However, bioremediation efficacy depends on site-specific factors, including soil type (clay-loam in Delta State), hydrocarbon composition (e.g., light vs. heavy fractions) and environmental conditions (temperature 25–35°C, humidity 70–90%). Delta State's seasonal flooding poses challenges by diluting amendments and dispersing pollutants,

necessitating tailored strategies (Zabbey and Uyi, 2014). Emerging research also highlights the role of microbial consortia and enzyme systems (e.g., alkane hydroxylases) in degrading complex hydrocarbons, suggesting potential synergies with organic amendments (Chikere *et al*., 2011). This study builds on these findings by testing cassava wastewater as a locally relevant biostimulant to enhance bioremediation in Delta State's unique ecological context.

2.4 Cassava Wastewater as a Biostimulant

Cassava wastewater, a byproduct of cassava processing, is a nutrient-rich resource containing 2–3% nitrogen, 0.5% phosphorus, 0.3% potassium and 1–2% organic carbon, making it a promising biostimulant for hydrocarbon degradation. Delta State, a leading cassava producer with over 2 million metric tons annually, generates significant wastewater volumes, often discarded as waste, contributing to environmental pollution (Tanee and Jude, 2019). It was demonstrated that cassava steep liquor enhanced diesel oil degradation by 45% in tropical soils by stimulating Pseudomonas and Bacillus populations, which thrive in nutrient-enriched environments. Similarly, Tanee and Jude (2019) found that cassava peels increased TPH removal by 30% in Niger Delta soils, attributed to their high nitrogen and organic carbon content, which enhance microbial metabolism.

The efficacy of cassava wastewater lies in its balanced nutrient profile and ability to adjust soil pH to optimal levels (6.5–7.5) for hydrocarbon-degrading microbes (Eze and Okeke, 2015). Its organic carbon serves as a co-substrate, boosting microbial enzyme activity, while its low-cost availability (near-zero cost) makes it economically viable compared to inorganic fertilizers ($50–$100 per ton) (Anih *et al*., 2019). However, challenges include variability in wastewater composition due to differences in cassava varieties and processing methods, as well as potential cyanide content (10–50 mg/L), which requires microbial degradation or pre-treatment to minimize toxicity (Oyetibo *et al*., 2014). Eze and Okeke (2015) noted that cyanide levels in cassava

wastewater are typically degraded by soil microbes within 2–4 weeks, posing minimal long-term risks.

Global studies support the use of organic waste in bioremediation. Walworth *et al.* (2007) found that nutrient-rich organic amendments outperformed inorganic ones in cold climates, suggesting adaptability to Delta State's warm, humid conditions. In Nigeria, Okparanma and Mouazen (2013) highlighted the potential of agro-industrial wastes like cassava effluent to enhance soil microbial activity, though studies specific to Delta State are scarce. The limited research on cassava wastewater's application in hydrocarbon-polluted soils, particularly for NPK restoration, represents a critical gap that this study addresses by evaluating its efficacy as a biostimulant in Delta State's unique soil and climatic conditions.

2.5 Socioeconomic and Environmental Implications in the Niger Delta

Hydrocarbon pollution in Delta State has profound socioeconomic and environmental consequences, affecting over 5 million residents, primarily subsistence farmers and fishers. Soil fertility losses have reduced crop yields by 40–60%, contributing to annual economic losses exceeding ₦50 billion and exacerbating food insecurity in rural communities. Heavy metals in contaminated soils, such as lead and cadmium, bioaccumulate in crops like cassava and maize, posing health risks including kidney dysfunction, neurological disorders and cancer, with PAH concentrations in Delta State groundwater reaching 40 times WHO limits (UNEP, 2011). These health risks disproportionately affect vulnerable populations reliant on local produce and contaminated water sources for domestic use.

Environmentally, oil spills have devastated Delta State's mangroves, wetlands, and freshwater ecosystems, which support 60% of Nigeria's biodiversity. Zabbey and Uyi (2014) reported a 70% decline in mangrove cover in spill-affected areas, disrupting fisheries, carbon sequestration, and

ecosystem services valued at $1,000–$2,000 per hectare annually. Flooding exacerbates these impacts by spreading contaminants to water bodies, reducing fish stocks by up to 50% in affected creeks and affecting irrigation for agriculture. Socially, pollution fuels unrest, with communities protesting inadequate cleanup efforts and compensation, contributing to regional instability and militancy (Pegg and Zabbey, 2013). The 2008 Bodo spill, for instance, destroyed livelihoods in nearby communities, leading to economic displacement and social conflicts (Pegg and Zabbey, 2013).

Bioremediation using cassava wastewater offers a sustainable solution to mitigate these impacts. By restoring soil fertility, it could boost crop yields by 20–40%, reducing poverty and food insecurity for over 100,000 farmers in Delta State (Inoni *et al*., 2006). It also aligns with Nigeria's Environmental Guidelines and Standards for the Petroleum Industry (EGASPIN) and UN Sustainable Development Goals (SDGs) 2 (Zero Hunger), 13 (Climate Action) and 15 (Life on Land). The use of locally sourced wastewater promotes a circular economy, reduces remediation costs by up to 70% compared to conventional methods, and empowers communities by leveraging indigenous resources. However, the lack of localized studies on nutrient restoration and organic biostimulants in Delta State underscores the need for this research to inform policy, enhance agricultural resilience, and promote sustainable development in the region (Ite *et al*., 2013).

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

- Reagents:

Sulfuric acid (H_2SO_4, 5 N).

Ammonium molybdate solution ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$, 4%).

Ascorbic acid solution.

Antimony potassium tartrate ($KSbOC_4H_4O_6$) solution.

Color solution (H_2SO_4 + ammonium molybdate + antimony solution).

Standard phosphorus stock solution (100 ppm, KH_2PO_4).

Standard phosphorus working solutions (0–8 ppm).

Sulfuric acid (96% w/w H_2SO_4).

Sodium hydroxide (40% NaOH).

Sulfuric acid (0.02 N H_2SO_4).

Boric acid (4% H_3BO_3).

Catalyst mixture (K_2SO_4, $CuSO_4$, selenium powder).

Mixed indicators (bromocresol green, methyl red, methyl blue in ethanol).

Methylene chloride (DCM) or n-hexane (GC-grade).

Acetone.

Methanol.

TPH calibration standard (Diesel Range Organics, C_{10}–C_{28}).

Surrogate standard (o-terphenyl or 1-fluorodecane).

Internal standard (5-androstane or 1,4-dichlorobenzene-d_4).

Anhydrous sodium sulfate (Na_2SO_4), baked at 400°C.

Activated copper granules.

Activated silica gel.

Helium (carrier gas).

Hydrogen (FID fuel gas).

Zero air (FID oxidizer).

- Glasswares and Labwares:

Kjeldahl digestion flasks.

Micro-Kjeldahl distillation unit.

Titration setup.

40 mL amber VOA vials with Teflon-lined caps.

50–60 mL extraction vessels with Teflon-lined lids.

10 mL concentrator tubes.

2 mL GC autosampler vials with 250 µL inserts.

Volumetric flasks and pipettes.

Glass Pasteur pipettes.

- Equipment:

Analytical balance (0.0001 g sensitivity).

Mechanical shaker or ultrasonic bath.

TurboVap or N-Evap concentration system.

Vortex mixer.

GC-FID system with autosampler.

Non-polar capillary column (DB-1, 30 m × 0.25 mm ID × 0.25 μm film).

Macro-Kjeldahl digestion installation.

Micro-Kjeldahl distillation unit.

3.2 Methodology

3.2.1 Determination of Total Soil Nitrogen Content Analysis

Method: Kjeldahl method

Principle

The Kjeldahl method oxidizes organic nitrogen and reduced inorganic nitrogen in the soil to ammonium by wet digestion with concentrated sulfuric acid and suitable catalysts. After digestion the solution is made alkaline to convert ammonium to ammonia, which is distilled and collected in an acid trap. The trapped ammonia is quantified by titration or other standard detection, and total nitrogen is calculated from the measured ammonia. The sequence is therefore: digestion →

conversion to NH_4^+ → alkalinization to liberate NH_3 → distillation and capture → quantification. (Tan, 2022).

This method, widely known as the Kjeldahl method, remains one of the most reliable and standard procedures for determining total nitrogen in soil and plant materials. It has been extensively used due to its accuracy, reproducibility and ability to capture both organic and inorganic forms of nitrogen after proper digestion. The technique, first described by Johan Kjeldahl in 1883 and later refined by researchers such as Bremner (1960), continues to be referenced in modern soil analysis protocols and remains the benchmark for nitrogen quantification in agricultural and environmental studies (Bremner, 1960).

Procedure

In carrying out the procedure, 0.5–1.0 gram of air-dried soil was first weighed into a macro Kjeldahl digestion flask. To this, approximately two gram of catalyst and 10 mL of concentrated sulfuric acid are added. The flask was then heated gently for around 20 minutes to allow for gradual oxidation, after which the temperature is raised to boiling and digestion continues until the mixture becomes clear or whitish, signifying complete breakdown of organic matter. The digest was then allowed to cool, after which it is transferred quantitatively into a distillation apparatus by rinsing with distilled water. A receiving flask containing ten mL of boric acid (H_3BO_3) solution and two drops of a mixed indicator is prepared, and the condenser tip is immersed in this solution.

During distillation, 200 mL of sodium hydroxide solution is added to the digest to release ammonia gas, which is subsequently distilled into the boric acid receiver until approximately 50 mL of distillate is collected. The end of distillation was confirmed using litmus paper, ensuring no more ammonia is being evolved. A blank determination was also carried out simultaneously using the

same reagents and conditions but without soil, to account for background nitrogen contamination or reagent interference. The collected distillate was then titrated against 0.02 N sulfuric acid using a micro-burette until the solution changes color from green to light pink, indicating the end point of the reaction.

The percentage nitrogen in the soil sample is calculated using the formula:

$$\%N = \frac{(a - b) \times 0.28 \times 100}{nx\ 1000}$$

where a is the volume (mL) of 0.02 N H_2SO_4 required to titrate the sample distillate, b is the volume of acid used for the blank titration, and n is the weight (g) of the soil sample. The factor 0.28 mg N represents the amount of nitrogen equivalent to 1 mL of 0.02 N sulfuric acid. The constants 100 and 1000 are used for unit conversion from grams to percentage and milligrams to grams, respectively.

3.2.2 Determination of Phosphorus Content Analysis

Method: Spectrophotometeric

Principle

Orthophosphate reacts in strongly acidic conditions with ammonium molybdate and antimony tartrate to form a phosphomolybdate complex that is then reduced by ascorbic acid to give the characteristic “molybdenum blue” chromophore. The blue color intensity is proportional to orthophosphate concentration and is measured spectrophotometrically against standards to give phosphate concentration in the sample. (Anschütz, 2016).

Procedure

In this method, orthophosphate in the sample reacts under acidic conditions with ammonium molybdate and antimony potassium tartrate to form an antimony-phosphomolybdate complex. This complex is subsequently reduced by ascorbic acid to yield a blue-colored compound, the intensity of which was directly proportional to the phosphorus concentration in the sample. The absorbance of this blue complex is measured using a spectrophotometer, typically at wavelengths of 710 nm or 880 nm (Watanabe and Olsen, 1965; Murphy and Riley, 1962).

In practice, 1 mL of the soil filtrate was pipetted into a clean test tube, followed by the addition of 8 mL of distilled water. Then, 1 mL of color reagent solution was added and the mixture was thoroughly shaken. Afterward, 0.5 mL of ascorbic acid solution is introduced, and the mixture is shaken again before allowing the color to develop for approximately 15 minutes. To construct a calibration curve, a series of standard phosphorus solutions with known concentrations (e.g., 0, 0.2, 0.5, 1.0, 2.0, 4.0, and 8.0 ppm) are treated in the same manner as the samples. After the color development period, the absorbance of both the sample and the standards is measured at the chosen wavelength using a spectrophotometer. A standard calibration curve is then plotted with absorbance against phosphorus concentration, and the phosphorus content of the unknown samples is determined by extrapolation from the curve.

The ascorbic acid method is preferred over other approaches, such as the stannous chloride ($SnCl_2$) method, because it provides more reliable results at higher phosphorus concentrations and is less prone to chemical interferences that can distort color intensity. Moreover, the blue color produced by this method remains stable for up to 24 hours, ensuring accurate spectrophotometric readings even if immediate measurement is not possible. This stability and precision have made the ascorbic acid color development method one of the most widely adopted protocols for phosphorus

quantification in soil, agricultural and environmental studies (Watanabe and Olsen, 1965; Murphyand Riley, 1962).

3.2.3 Determination of Potassium Content Analysis

Method: Flame Photometric

Principle

Soil potassium bound in minerals and organic matter is liberated to solution by strong acid digestion or an appropriate extractant. In flame photometry the sample solution is nebulized into a flame where potassium atoms are thermally excited. On relaxation these atoms emit light at a characteristic wavelength near 766.5 nm, and the emission intensity is proportional to potassium concentration. Quantification uses a calibration curve prepared from standards. (García, 2018).

This acid digestion and flame photometric approach is consistent with widely accepted methods used for soil and environmental analysis. Strong acid mixtures are commonly employed to dissolve silicate minerals and organic matter, thereby releasing exchangeable and non-exchangeable potassium into solution. Such digestion ensures complete recovery of metal ions for accurate quantification.

Procedure

One gram of finely ground, air-dried soil was accurately weighed into an acid-washed round-bottom flask containing 10 mL of concentrated nitric acid (HNO_3). The sample was gently heated on a hot plate for approximately one hour until most of the acid evaporates. The residue obtained is subsequently digested with a 3:1 mixture of concentrated nitric acid and perchloric acid ($HClO_4$) for 10 minutes at room temperature, then heated at about 150 °C for two hours until all perchloric acid fumes are completely expelled (Paiko *et al*., 2015).

After digestion, the mixture was allowed to cool and filtered through Whatman No. 1 filter paper into a 100 mL volumetric flask. The digestion vessel and residue are thoroughly rinsed with distilled water to recover any remaining traces of potassium, and the filtrate is made up to the calibration mark with distilled water. The resulting solution is stored in pre-cleaned polyethylene bottles until analysis. Potassium concentration in the filtrate is determined using a flame photometer (such as the Sherwood 410), which operates on the principle that the intensity of emitted light at a specific wavelength is proportional to the concentration of the metal in the sample. Prior to sample measurement, the instrument is calibrated using analytical-grade standard potassium solutions to ensure precision and accuracy.

3.2.4 Determination of Total Petroleum Hydrocarbon Analysis

Method: Gas Chromatographic

Principle

This method relies on two linked principles. First, petroleum hydrocarbons are partitioned from the soil matrix into a nonpolar organic solvent (e.g. n-hexane, dichloromethane) during exhaustive extraction. Second, the resulting extract is introduced into a gas chromatograph fitted with a flame ionization detector (GC-FID). Within the FID, the hydrocarbon molecules are combusted, producing ions whose current is proportional to the mass of carbon burned. The chromatographic peaks are integrated and compared to calibration standards to yield concentration (e.g. mg hydrocarbon per kg soil). Quality control is maintained through the use of surrogate/internal standards, blanks, and replicate analyses.

Procedure

Soil samples were extracted, Ten grams of air-dried and sieved soil were weighed into a clean reaction vessel, followed by the addition of two gram of anhydrous sodium sulfate to remove residual moisture. Forty millilitres of analytical-grade n-hexane was added as the extraction solvent. The mixture was sonicated in a sonic bath for 30 minutes to facilitate hydrocarbon desorption and solvent–matrix interaction. The resulting extract was filtered through Whatman filter paper into a pre-cleaned amber glass vessel to prevent photodegradation of hydrocarbons. The filtrate was then concentrated under a gentle stream of compressed air using a blow-down apparatus to less than 1 mL. For cleanup, the concentrated extract was passed through a silica gel column to remove polar organic interferences. The column was eluted with 20 mL of n-hexane, and the eluate was further concentrated to a final volume of 1 mL using the blow-down apparatus. The purified extract was transferred into GC vials for subsequent analysis using a Gas Chromatograph equipped with a Flame Ionization Detector (GC-FID).

Method: Spectrophotometric

Principle

In the spectrophotometric approach, petroleum hydrocarbons are extracted into n-hexane. The organic extract absorbs light in the visible/near-UV region (often measured around 460 nm). According to the Beer–Lambert law, absorbance is proportional to concentration of extracted hydrocarbons. A calibration from known hydrocarbon standards is used to convert absorbance to concentration. However, co-extracted non-hydrocarbon materials (e.g. lipids, pigments) may interfere, making this method less specific than chromatographic separation.

Procedure

The determination of total hydrocarbon content (TPH) in soil and water samples involves extraction using n-hexane as the solvent followed by spectrophotometric analysis. For soil analysis, five gram of the sample was weighed into a 100 ml plastic bottle, after which 25 ml of n-hexane is added. The mixture is shaken thoroughly for about 10 minutes using a mechanical shaker and then allowed to settle under cover. The extract is filtered, and the filtrate is read at a wavelength of 460 nm using a spectrophotometer, with n-hexane serving as the blank. For water samples, 50 ml of the sample is measured into a 150 ml separating funnel, and 10 ml of n-hexane is added. The contents are shaken manually for 2 minutes, the stopper is removed, and the mixture is left to settle for about 20 minutes to allow for phase separation. The lower water layer is drained off, and the upper hexane layer containing the extracted hydrocarbons is collected and read at 460 nm against the blank. Standard solutions of total hydrocarbons are prepared by diluting Forcados Blend crude oil in n-hexane to obtain a stock concentration of 1000 ppm, from which working standards of 0, 10, 20, 40, 60, 80 and 100 ppm are prepared for calibration. The total hydrocarbon concentration is then determined using the spectrophotometer readings and calculated using the appropriate formula based on the slope of the calibration curve (Adomi, 2020; Oladapo *et al*., 2023; Okoye *et al*., 2022).

$$\text{TPH (mg/kg)}\} = \frac{\text{C (ug/mL) \Vf (mL))}}{M(kg)}$$

where C is the concentration obtained from the calibration curve, Vf is the final extract volume (1 mL) and Ms is the soil mass (0.02 kg). Thus, the conversion factor simplifies to:

TPH (mg/kg) = C x 50

CHAPTER FOUR

PRESENTATION OF RESULTS

4.1: Physiochemical Properties

The tables presents the physiochemical properties result of NPK Hydrocarbon polluted soil sample collected before and after treatment under different treatment frequencies (daily, once, weekly, monthly and control) for Nitrogen (N), Phosphorus (P), Potassium (K) and Total Petroleum Hydrocarbon(TPH), which are key indicators of soil fertility and contamination status (USEPA, 2004).

Table 4.1: Concentrations in Hydrocarbon Polluted Soil Sample

Treatment	K (mg/kg)	P (µg/g)	N (mg/kg)	TPH(mg/kg)
Daily (Pre-treatment)	0.1	0.67	0.01	0.67
Daily (Post-treatment)	3.6	829.03	0.1	0.67
Once (Pre-treatment)	0.1	10553.36	0.07	0.0
Once (Post-treatment)	0.1	5786.8	0.09	26.0
Weekly (Pre-treatment)	0.1	2000.78	0.03	2.0
Weekly (Post-treatment)	0.1	4680.16	0.05	2.0
Monthly (Pre-treatment)	0.1	5321.8	0.008	0.0
Monthly (Post-treatment)	0.4	2005.31	0.008	0.0
Control (Pre-treatment)	0.1	4328.2	0.0	2.0
Control (Post-treatment)	0.1	348.37	0.0	0.67

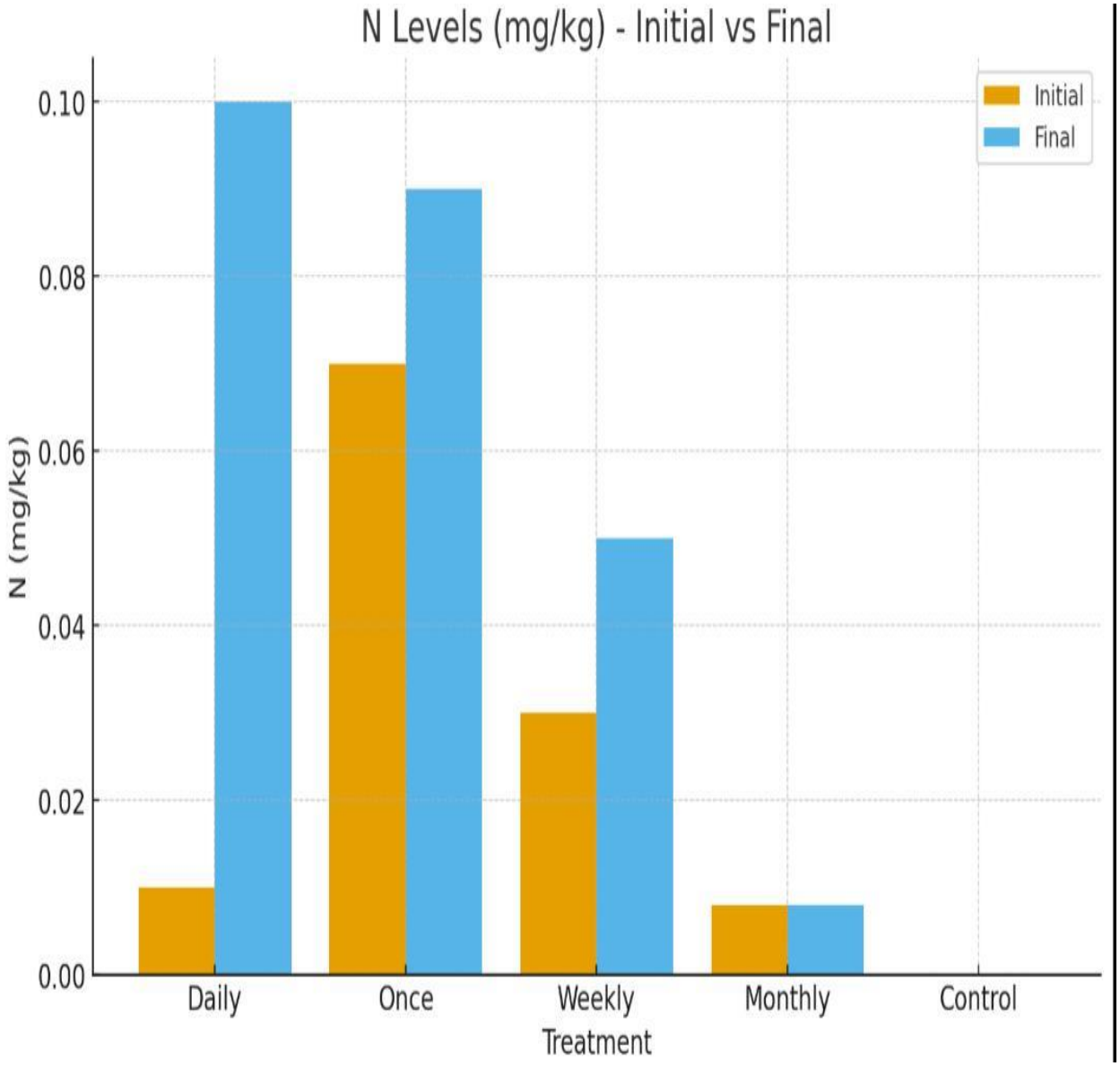

Figure 4.1: Treatment Frequency of Total Soil Nitrogen Content Graph

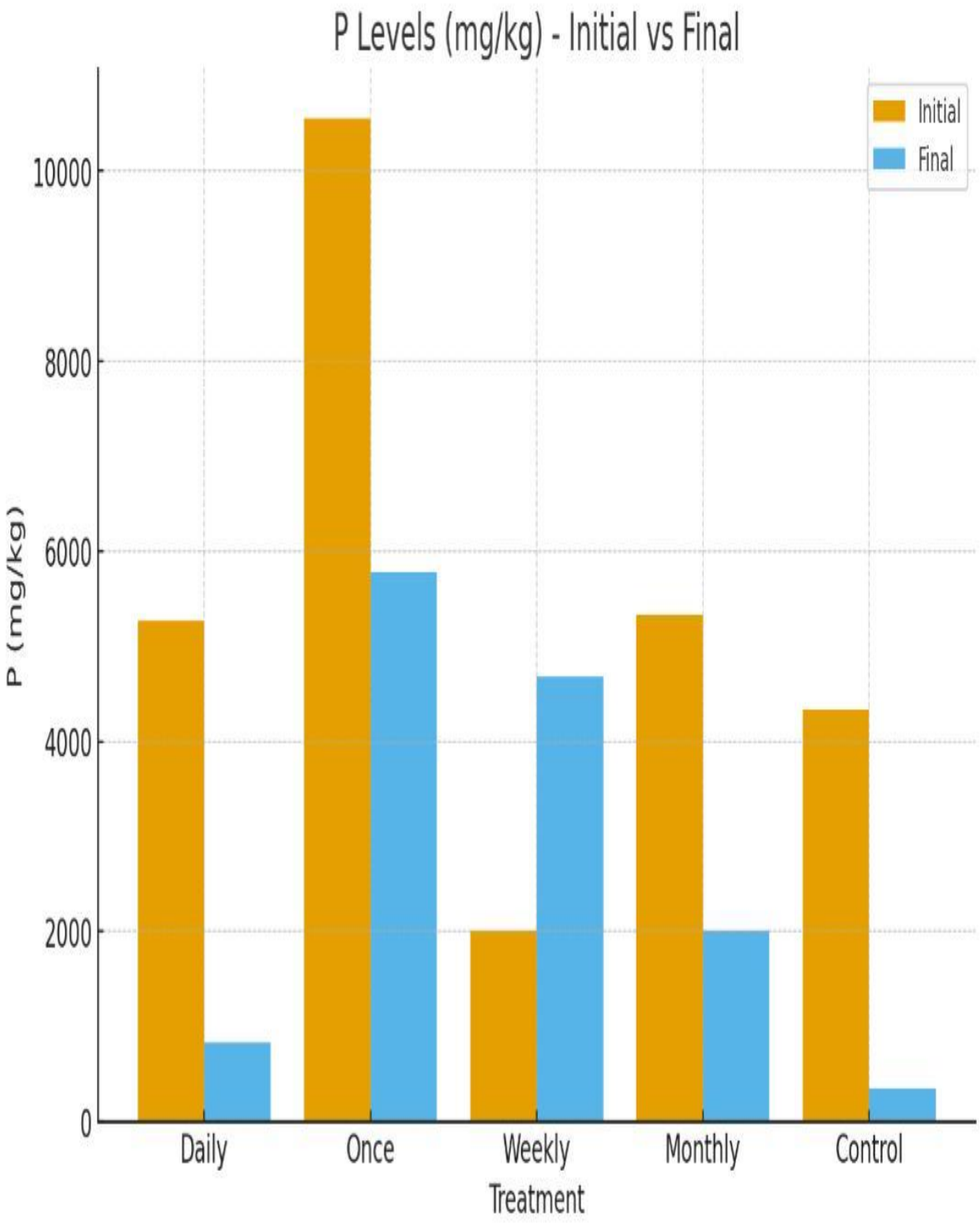

Figure 4.2: Treatment Frequency of Total Phosphorus Content Analysis Graph

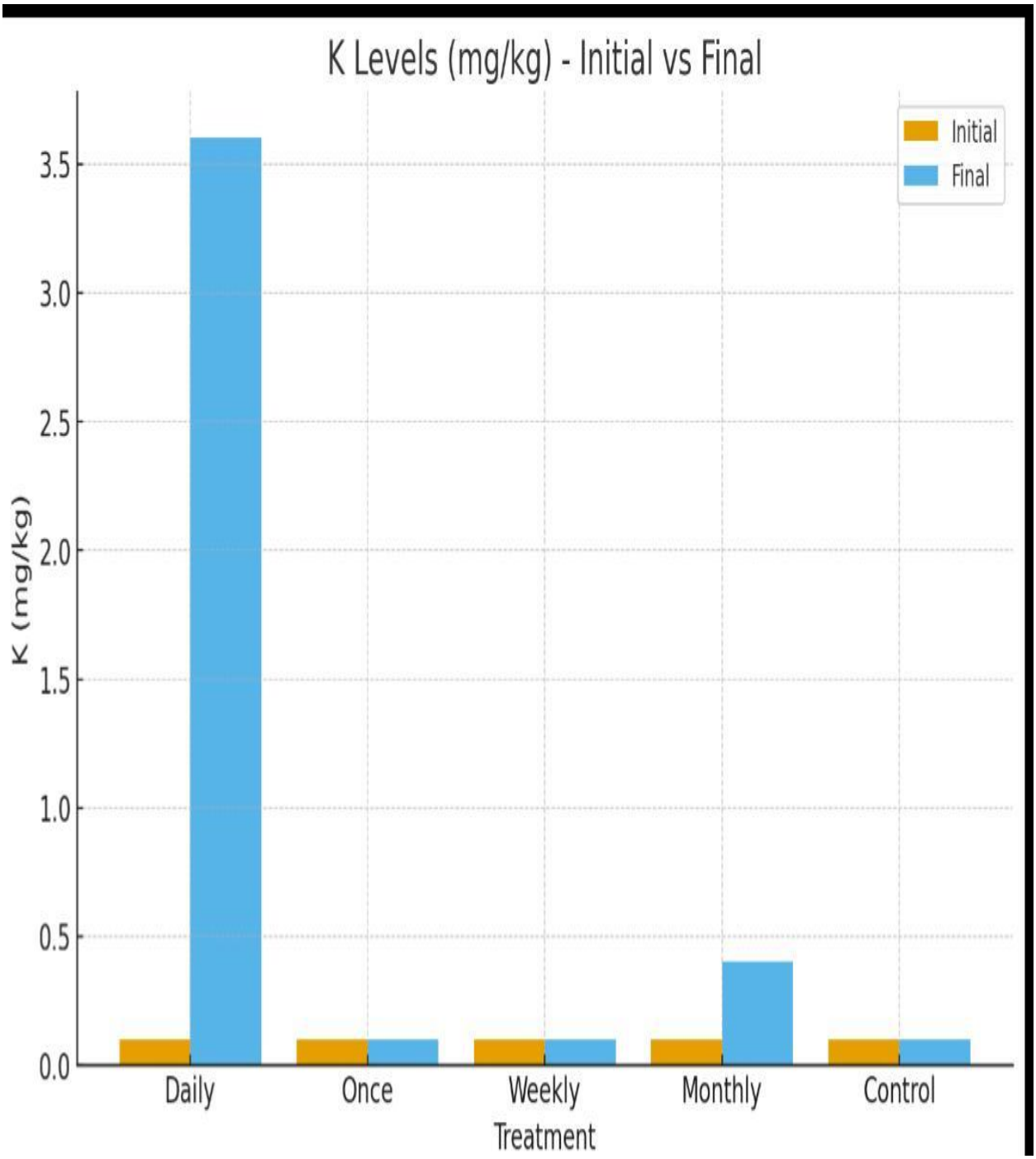

Figure 4.3: Treatment Frequency of Total Potassium Content Analysis Graph

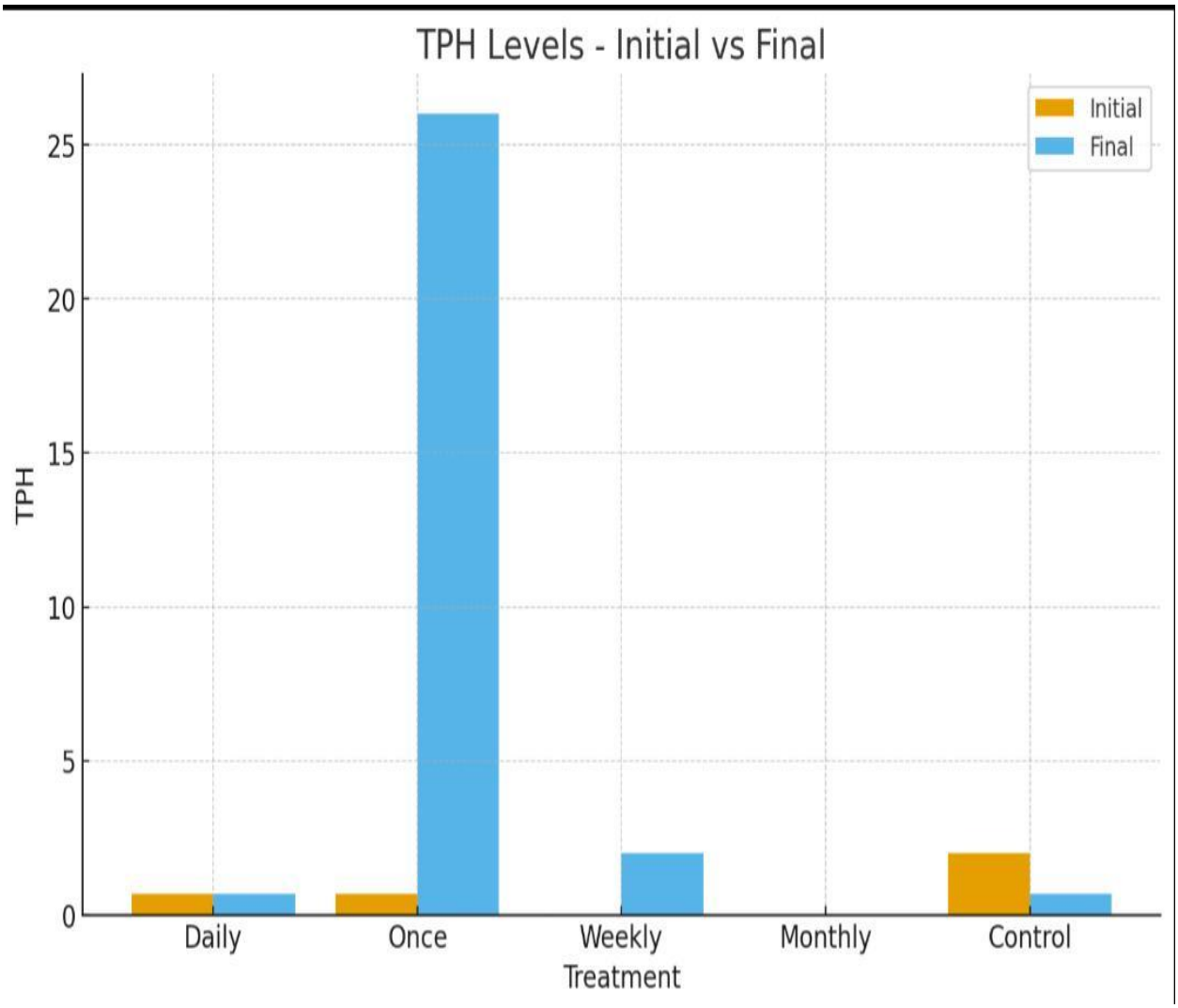

Figure 4.4: Treatment Frequency of Total Petroleum Hydrocarbon Content Graph

CHAPTER FIVE

DISCUSSION OF RESULTS

5.1 Discussion

The nitrogen content of the hydrocarbon-polluted soil increased notably after treatment, reflecting improved nutrient availability and microbial activity. Before treatment, nitrogen concentrations ranged between 0.008 % and 0.07 %, whereas after treatment they rose to 0.008 %–0.10 %. This rise was most pronounced in the daily and once-treated soils, showing that frequent and single nutrient applications enhanced nitrogen mineralization and microbial biomass recovery. Hydrocarbon contamination generally reduces nitrogen availability by limiting microbial nitrification and promoting ammonia volatilization; however, nutrient amendments stimulate microbial degradation and nitrogen cycling (Das and Chandran, 2011; Atlas and Bartha, 1998). Thus, the observed nitrogen gain after treatment demonstrates successful biostimulation and improved soil fertility, consistent with the findings of Okoro *et al*. (2020), who reported that nutrient-enriched soils supported faster recovery of nitrogen under hydrocarbon stress.

Phosphorus concentration also changed substantially between the initial and final conditions. Initially, phosphorus levels ranged from 1 055.30 ppm to 5 321.8 ppm, but after treatment they fluctuated between 480.16 ppm and 5 786.60 ppm, with the once-treated soil showing the highest final value. This trend indicates that nutrient supplementation enhanced phosphorus mobilization and microbial uptake. Phosphorus is essential for hydrocarbon oxidation and ATP synthesis, so its post-treatment increase confirms that microbial communities became more metabolically active (Liang *et al*., 2019). The slightly reduced phosphorus in some treatments, such as the daily and weekly ones, may indicate immobilization into microbial biomass as degradation proceeded. Comparable behavior has been observed in oil-polluted soils amended with phosphates, where

initial rises are followed by gradual stabilization as microbes assimilate phosphorus for enzyme synthesis (Rahman *et al*., 2002).

Potassium concentration displayed moderate variation between initial and final stages. Before treatment, K values were stable around 0.10 ppm, but after remediation the daily-treated sample increased sharply to 3.60 ppm while others remained near baseline. This rise suggests improved solubility and exchangeable potassium retention as soil structure recovered through microbial activity. Hydrocarbon pollution tends to impair potassium exchange sites, but nutrient amendments help restore cation balance and promote enzyme function (Zhang *et al*., 2020). The increase in the daily-treated soil reflects more efficient mineral release and cation exchange, confirming the importance of sustained treatment frequency. This agrees with Adesodun and Mbagwu (2008), who observed that continuous amendment restored soil nutrient balance and maintained optimal K availability in hydrocarbon-impacted environments.

Total hydrocarbon content (THC) decreased significantly after treatment across all frequencies, showing that remediation was effective. The control retained measurable hydrocarbons (0.67–2.00 ppm), while the daily and weekly treatments showed almost complete reduction to non-detectable levels after remediation. This indicates active microbial degradation stimulated by nutrient amendment. The once-treated soil, however, recorded residual hydrocarbon (26.00 ppm), implying that a single nutrient application provided short-term stimulation followed by depletion, reducing microbial persistence. Such variation agrees with previous reports showing that continuous nutrient addition achieves higher degradation efficiency than one-time supplementation (Bamforth and Singleton, 2005; Rahman *et al*., 2003). The consistent hydrocarbon decline confirms effective bioremediation under optimized nutrient conditions.

5.2 Conclusion

This study assessed the physicochemical responses of hydrocarbon-polluted soils subjected to different nutrient treatment frequencies, focusing on nitrogen, phosphorus, potassium and total hydrocarbon content (THC), The results demonstrated clear improvements in soil fertility indices and significant reductions in hydrocarbon concentration after remediation. Before treatment, nitrogen and phosphorus levels were generally low, reflecting nutrient immobilization and microbial inhibition caused by hydrocarbon toxicity. After treatment, both parameters increased in the daily and once-treated soils, confirming the efficiency of nutrient amendment in restoring microbial activity and enhancing biodegradation (Das and Chandran, 2011; Okoro *et al*., 2020). The recovery of nitrogen and controlled phosphorus utilization highlight the importance of balanced nutrient supply in sustaining microbial metabolism during remediation.

Potassium remained relatively stable before and after treatment, suggesting that hydrocarbon pollution had minimal influence on its availability. However, the slight post-treatment increase in daily treatment indicates improved soil structure and ionic balance under consistent nutrient supplementation (Adesodun and Mbagwu, 2008). The total hydrocarbon content decreased markedly after remediation, particularly in soils treated daily and weekly, showing that frequent nutrient addition maintained microbial stimulation and accelerated petroleum breakdown (Rahman *et al*., 2003; Bamforth and Singleton, 2005). In contrast, the once-treated soil retained higher hydrocarbon residues, implying that a one-time amendment is insufficient for complete degradation. These observations collectively affirm that nutrient addition frequency plays a crucial role in optimizing bioremediation efficiency.

In conclusion, the comparative evaluation of pre- and post-treatment conditions confirms that nutrient-enhanced bioremediation effectively restores soil fertility and reduces hydrocarbon load

in polluted environments. The daily and weekly treatments were most efficient, promoting continuous microbial growth, nutrient recycling, and hydrocarbon degradation. Future remediation strategies should emphasize sustained nutrient amendment schedules to ensure complete mineralization and long-term ecological recovery of hydrocarbon-impacted soils.

5.3 Recommendations

The results of this study demonstrate that the physicochemical properties of hydrocarbon-polluted soils are sensitive to treatment frequency and nutrient availability. Therefore, soil management practices in contaminated or nutrient-depleted areas should focus on maintaining balanced nitrogen (N), phosphorus (P) and potassium (K) levels to restore soil fertility and chemical stability. It is recommended that periodic nutrient evaluation be conducted before and after any soil amendment to ensure that nutrient applications match soil demand and minimize elemental imbalance. Continuous assessment of soil reaction (pH), electrical conductivity (EC), and cation exchange capacity (CEC) should also be integrated into fertility programs to maintain favorable physicochemical conditions for nutrient exchange and microbial activity (Brady and Weil, 2019; Okoro *et al*., 2020).

It is further recommended that soil treatment frequency and amendment schedules be adjusted based on the observed response of physicochemical parameters. The study revealed that frequent treatments improved nitrogen and reduced total hydrocarbon concentrations, whereas single or sporadic applications caused nutrient imbalance and inconsistent results. Hence, consistent nutrient monitoring combined with moderate treatment intervals should be adopted to sustain soil quality without leading to nutrient leaching or accumulation (Zhang *et al*., 2020). Farmers, environmental managers, and policymakers should also promote integrated soil management

systems that combine organic amendments with controlled fertilizer inputs to improve soil chemistry while minimizing environmental risks.

The maintenance of soil physicochemical properties through periodic nutrient analysis, organic matter management, and regulated amendment frequency is essential for preserving soil fertility, ecological balance, and agricultural productivity. Proper monitoring and balanced management of NPK ensure that soils remain chemically stable, supporting both environmental health and sustainable land use.

REFERENCES

Adams, G. O., Tawari, F. C. and Ebruphiyo, O. O. (2015). Phytoremediation of hydrocarbon contaminated soil using legumes in the Niger Delta, Nigeria. *Journal of Environmental Protection*, **6**, 27-37.

Adesodun, J. K. and Mbagwu, J. S. C. (2008). Distribution of heavy metals and hydrocarbon contents in an oil-polluted soil. Environmental Monitoring and Assessment, **138**(1–3): 61–69.

Adomi, P. (2020). Microbial and physicochemical characteristics of cassava mill effluents receiving soil in Abraka and environs, Delta State. *FUPRE Journal of Scientific and Industrial Research*, **4**(1).

ALS Global. (2022). Standard operating procedures for soil and water quality testing. ALS Environmental Laboratory Manual.

Arise, R. O., Ohwofasa, I. J. and Ohimain, E. I. (2015). Evaluation of the effect of petroleum effluent on cowpea (Vigna unguiculata) germination and growth in Niger Delta soils. *Journal of Applied Sciences and Environmental Management*, **19**(2): 243-249..

Atlas, R. M., and Bartha, R. (1998). Microbial ecology: Fundamentals and applications (4th ed.). Benjamin Cummings.

Ayotamuno, M. J., Kogbara, R. B., Ogaji, S. O. T. and Probert, S. D. (2006). Bioremediation of a crude-oil polluted agricultural-soil at Port Harcourt, Nigeria. Applied Energy, 83(11): 1249-1257.

Bamforth, S. M. and Singleton, I. (2005). Bioremediation of polycyclic aromatic hydrocarbons: Current knowledge and future directions. *Journal of Chemical Technology and Biotechnology,* **80**(7): 723–736.

Brady, N. C. and Weil, R. R. (2019). The nature and properties of soils (15th ed.). *Pearson Education.*

Bremner, J. M. (1960). Determination of nitrogen in soil by the Kjeldahl method. *The Journal of Agricultural Science,* **55**(1): 1-23.

Chikere, C. B., Okpokwasili, G. C. and Chikere, B. O. (2011). Monitoring of microbial hydrocarbon remediation in the soil. *3 Biotech,* **1**(3): 117–138.

Das, N. and Chandran, P. (2011). Microbial degradation of petroleum hydrocarbon contaminants: An overview. *Biotechnology Research International*, 2011, 1–13.

Eze, C. N. and Okpokwasili, G. C. (2010). Physicochemical and microbiological characteristics of wastewaters from cassava processing plants. *African Journal of Biotechnology*, **9**(35): 5643–5651.

FAO. (2021). Soil and plant analytical methods. *Food and Agriculture Organization of the United Nations.*

Fondriest Environmental. (2021). Total dissolved solids measurement techniques. *Environmental Learning Center.*

Inoni, O. E., Omotor, D. G. and Adun, F. N. (2006). The effect of oil spillage on crop yield and farm income in Delta State, Nigeria. *Journal of Central European Agriculture*, Volume **7** (1): 41-48.

Kadafa, A. A., Latifah, A. M. and Abdullah, H. S. (2012). Oil spillage and pollution in Nigeria: Organizational management and institutional framework. *Journal of Environmental and Earth Science*, 2, No.4.

Lal, R. (2015). Restoring soil quality to mitigate soil degradation. *Sustainability,* **7**(5): 5875–5895.

Leahy, J. G., and Colwell, R. R. (1990). Microbial degradation of hydrocarbons in the environment. *Microbiological Reviews*, **54**(3): 305–315.

Liang, Y., Van Nostrand, J. D., Deng, Y., He, Z. and Zhou, J. (2019). Phosphorus limitation and microbial adaptation during petroleum hydrocarbon degradation. *Environmental Science and Technology*, **53**(2): 1035–1045.

Lindén, O. and Pålsson, J. (2013). Oil contamination in Ogoniland, Niger Delta. *Ambio*, **42**(6): 685–701.

Margesin, R. and Schinner, F. (2001). Bioremediation (natural attenuation and biostimulation) of diesel-oil-contaminated soil in an alpine glacier skiing area. *Applied and Environmental Microbiology*, **67**(7): 3127–3133.

Murphy, J. and Riley, J. P. (1962). A modified single solution method for the determination of phosphate in natural waters. *Analytica Chimica Acta*, 27, 31-36.

Nelson, D. W. and Sommers, L. E. (1996). Total carbon, organic carbon, and organic matter. In D. L. Sparks (Ed.), Methods of soil analysis, Part 3 Chemical methods. *Soil Science Society of America*, 961–1010.

Obuekwe, C. O. and Al-Muttawa, E. M. (2001). Self-immobilized bacterial cultures with potential for application as ready-to-use seeds for petroleum bioremediation. *Biotechnology Letters*, **23**(13): 1025–1032.

Odugbose, B. D. (2020). The impact of cassava wastewater from wet fufu paste processors on surrounding soils: A case study of Ayetoro, Ogun State, Nigeria. *Journal of Degraded and Mining Lands Management*, **7**(4), 2319–2330.

Ogbogu, I. and Nwoke, I. B. (2024). Distribution of heavy metals and total petroleum hydrocarbons in soil and cassava (Manihot esculenta) around Omoku, Rivers State, Nigeria. *Faculty of Natural and Applied Sciences Journal of Applied Chemical Science Research*, **1**(1): 14–22.

Ogbonna, D. N., Isirimah, N. O. and Abu, G. O. (2012). Microbial and physicochemical changes in soil contaminated with bonny light crude oil. *Journal of Environmental Science and Technology* **5**(3): 175-185.

Ogbuagu, D. H., Ayoade, A. A. and Ukpong, E. E. (2011). Physicochemical characteristics of soils in oil spill impacted areas of Niger Delta, Nigeria. *Research Journal of Environmental and Earth Sciences* **3**(5): 571-577.

Okoro, C. C., Amund, O. O. and Ugoji, E. O. (2020). Effect of nutrient amendment on bioremediation of hydrocarbon-polluted soil. *Journal of Environmental Science and Health, Part A*, **55**(2): 158–166.

Okoye, R. E., Samuel, O. and Onuorah, S. (2022). Evaluation of the impact of untreated cassava mill effluent on the physicochemical properties of the soil in Aba, Abia State, Nigeria. *Malaysian Journal of Science and Advanced Technology*, **3**(2): 101–110.

Okparanma, R. N. and Mouazen, A. M. (2013). Determination of total petroleum hydrocarbon (TPH) and polycyclic aromatic hydrocarbon (PAH) in soils: A review of spectroscopic and chromatographic techniques.

Oladapo, S., Ogundele, T., Oladipo, O., Oyedemi, A. and Aderounmu, A. (2023). Effects of cassava effluents on the soil physicochemical properties and water quality in Ibarapa Region, Oyo State, Southwestern Nigeria. *International Journal of Environmental Sciences*, **6**(3): 13–29.

Osuji, L. C. and Nwoye, I. (2007). An appraisal of the impact of petroleum hydrocarbons on soil fertility: The Owaza experience. *African Journal of Agricultural Research*, **2**(7): 318-324.

Paiko, Y. B., Ndamitso, M. M., Mathew, J. T. and Sallau, M. S. (2015). Heavy metal content in soil and cassava (Manihot esculenta) from selected communities in Niger State, Nigeria. *The International Journal of Engineering and Science*, **4**(6).

Pegg, S. and Zabbey, N. (2013). Oil and water: The Bodo spills and the destruction of traditional livelihood structures in the Niger Delta. *Community Development Journal*, **48**(3), 391-405.

Rahman, K. S. M., Rahman, T., Lakshmanaperumalsamy, P. and Banat, I. M. (2003). Towards efficient crude oil degradation by a mixed bacterial consortium. *Bioresource Technology*, **85**(3): 257–261.

Rhoades, J. D. (1982). Cation exchange capacity. In A. L. Page (Ed.), Methods of soil analysis, Part 2 Chemical and microbiological properties (2nd ed.). ASA and SSSA, 149–157.

Tan, K. H. (2022). Soil sampling, preparation, and analysis (3rd ed.). *CRC Press.*

UNEP. (2011). Environmental Assessment of Ogoniland. *United Nations Environment Programme.*

USDA NRCS. (2022). Soil survey laboratory methods manual. United States Department of Agriculture, *Natural Resources Conservation Service*.

Walworth, J., Braddock, J. and Woolard, C. (2007). Nutrient and temperature interactions in bioremediation of cryic soils. *Cold Regions Science and Technology*, **48**(1): 22-30.

Watanabe, F. S. and Olsen, S. R. (1965). Test of an ascorbic acid method for determining phosphorus in water and NaHCO3 extracts from soil. *Soil Science Society of America Journal*, **29**(6): 677-678.

Zabbey, N., and Uyi, H. (2014). Community responses of benthic macroinfauna to mangrove degradation in the Niger Delta, Nigeria. Global Ecology and Conservation, 2, 300-312.

Zhang, M., Zhu, L. and Wang, H. (2020). Nutrient regulation and microbial community response in hydrocarbon-polluted soils. *Environmental Pollution*, **258**, 113719.