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# IMPACT OF CASSAVA MILL EFFLUENT AMENDMENT ON PHYSICOCHEMICAL PROPERTIES OF HYDROCARBON-CONTAMINATED SOIL OVER TIME

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## ABSTRACT

This study investigated the temporal effects of cassava mill effluent (CME) amendment on the physicochemical properties of spent lubricating oil (SLO)-contaminated soil over a six-month remediation period. Soils contaminated with 10% (w/w) SLO were treated with CME at varying frequencies (once, monthly, weekly, daily) alongside uncontaminated controls. Comprehensive analysis revealed significant treatment-dependent alterations. Hydrocarbon pollution initially degraded soil quality, reducing water holding capacity (WHC) and nutrient availability. CME application substantially improved soil properties: pH increased from acidic to near-neutral values (up to 7.6 in daily treatments), WHC improved by up to 103%, cation exchange capacity (CEC) increased by 242%, and organic carbon rose by 115%. Total petroleum hydrocarbon (TPH) content decreased by up to 47.14% with frequent CME application. Nutrient dynamics showed remarkable enhancement, with available nitrogen increasing by 3797% and phosphorus by 3649% in optimal treatments. The findings demonstrate that CME amendment effectively counters hydrocarbon-induced soil degradation. These results establish CME as a potent, low-cost organic amendment for restoring hydrocarbon-contaminated soils, with application frequency optimization identified as critical for balancing remediation efficacy against potential salinity concerns.

**Keywords:** Soil remediation, hydrocarbon contamination, organic amendment, soil physicochemical properties, cassava mill effluent, nutrient dynamics.

## INTRODUCTION

Soil contamination by petroleum hydrocarbons is a persistent global environmental challenge, with severe manifestations in oil-producing regions like Nigeria's Niger Delta (Ite *et al.*, 2013). Hydrocarbons initiate complex physicochemical alterations that compromise soil functionality, affecting microbial ecology and plant growth (Megharaj *et al.*, 2015). Their hydrophobic nature and toxicity disrupt soil structure, reduce water retention, alter nutrient cycling, and create unfavorable conditions for biological activity (Aislabie *et al.*, 2004; Varjani, 2017). These changes reduce agricultural productivity, contaminate groundwater, and diminish biodiversity, creating landscapes of ecological and economic impairment (Mekonnen *et al.*, 2024).

Traditional remediation methods like excavation, soil washing, and chemical oxidation suffer from high costs, energy intensity, potential secondary pollution, and ecosystem disruption (Perelo, 2010). In developing regions, these approaches are

often economically impractical, necessitating accessible alternatives.

Bioremediation, particularly biostimulation enhancing indigenous microbial communities through nutrient and organic matter amendments has emerged as a promising strategy (Das & Chandran, 2011; Guo *et al.*, 2014). Its success depends on selecting amendments that address soil limitations without causing salinity buildup, pH extremes, or introducing new contaminants.

Cassava mill effluent (CME), an abundant agro-industrial waste, is a compelling candidate. It contains high concentrations of organic matter, essential nutrients (N, P, K), and diverse organic compounds that can stimulate microbial activity (Olukanni *et al.*, 2019). However, critical gaps remain in understanding CME's effects on hydrocarbon-contaminated soil systems, including its influence on physicochemical alterations, temporal patterns of soil property changes, and the role of application frequency.

This study addresses these gaps by systematically investigating CME's effects on hydrocarbon-contaminated soil over six months. By examining soil texture, pH, water relations, nutrient dynamics, and organic matter under varying treatment frequencies, this research provides insights into CME-mediated restoration mechanisms and contributes to developing evidence-based remediation guidelines.

## MATERIALS AND METHODS

**Soil Collection and Experimental Setup:** Approximately 500 kg of topsoil (0–10 cm depth) was collected from an uncontaminated site in Benin City, Nigeria. The soil was homogenized, sieved to remove debris, and divided into experimental units. Contamination was achieved by thoroughly mixing spent lubricating oil (SLO) at 10% (w/w) into the soil. Two main experimental groups were established: (1) SLO-polluted soils amended with cassava mill effluent (CME), and (2) unpolluted soils receiving equivalent CME treatments. CME was applied at a rate of 2.5% (v/w) at different frequencies: once only (PTN/TN), monthly (PTM/TM), weekly (PTW/TW), and daily (PTD/TD). Control treatments included polluted unamended (PC) and unpolluted unamended (GC) soils. All treatments were replicated three times and maintained under open-air conditions to simulate natural environmental fluctuations over a six-month period.

**Soil Sampling and Physicochemical Analysis:** Composite soil samples were collected from each treatment at 0, 3, and 6 months. Standard analytical methods were employed: soil texture via the hydrometer method (Gee & Bauder, 1986); pH in a 1:2 soil-water suspension; water holding capacity (WHC) after 48-hour drainage (Reynolds, 1970); and electrical conductivity (EC) in saturation extracts (Richards, 1969). Cation exchange capacity (CEC) was determined by summation of exchangeable bases and acidity (Sumner & Miller, 1996). Organic carbon was analyzed using the Walkley-Black method (Walkley & Black, 1947). Total petroleum hydrocarbons (TPH) were extracted with n-hexane and quantified spectrophotometrically. Nutrient analyses included available nitrogen (KCl extraction, phenolate method; Weatherburn, 1967), total nitrogen (Kjeldahl digestion; Bremner & Mulvaney, 1982), and available and total phosphorus (Bray-1 extraction and acid digestion methods; Bray & Kurtz, 1945; Murphy & Riley, 1962). Heavy metals (Pb, Zn, Fe) were measured by atomic absorption spectrometry after acid digestion (Jackson, 1967).

**Statistical Analysis:** All data were analyzed using one-way ANOVA followed by Tukey's post-hoc test ($p < 0.05$). Temporal changes were assessed as percentage change relative

to initial values. Analyses were conducted using GraphPad Prism version 9.4, with results reported as mean ± standard error of three replicates

## RESULTS

**Table 1a:** Soil Texture at 0-month (g/kg soil)

| Treatment | Sand | Silt | Clay | Texture |
|---|---|---|---|---|
| GC | 886 ± 1.45 | 61 ± 1.76 | 54 ± 0.33 | SL |
| TN | 883 ± 1.20 | 63 ± 0.58 | 54 ± 1.20 | SL |
| TM | 887 ± 0.88 | 63 ± 1.53 | 50 ± 1.20 | SL |
| TW | 880 ± 0.88 | 66 ± 1.86 | 54 ± 1.00 | SL |
| TD | 885 ± 0.88 | 63 ± 0.58 | 52 ± 0.88 | SL |
| PC | 881 ± 0.67 | 67 ± 1.76 | 52 ± 1.15 | SL |
| PTN | 887 ± 0.58 | 61 ± 0.58 | 52 ± 0.58 | SL |
| PTM | 882 ± 0.88 | 63 ± 0.58 | 55 ± 0.67 | SL |
| PTW | 886 ± 0.88 | 65 ± 0.33 | 50 ± 0.88 | SL |
| PTD | 887 ± 0.88 | 62 ± 0.67 | 52 ± 0.33 | SL |

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**Table 1b:** Soil Texture at 3-month (g/kg soil) with Percentage Changes

| Treatment | Sand | Silt | Clay | Texture | %Δ Sand | %Δ Silt | %Δ Clay |
|---|---|---|---|---|---|---|---|
| GC | 906 ± 1.15 | 30 ± 0.58 | 64 ± 0.58 | SC | 2.3 | -50.55 | 19.25 |
| TN | 884 ± 0.58 | 30 ± 1.20 | 86 ± 0.88 | SC | 0.08 | -52.91 | 60.87 |
| TM | 918 ± 0.88 | 18 ± 0.58 | 63 ± 1.20 | SC | 3.57 | -71.43 | 25.83 |
| TW | 900 ± 3.18 | 21 ± 1.76 | 80 ± 4.91 | SC | 2.2 | -68.53 | 47.53 |
| TD | 884 ± 1.45 | 25 ± 1.20 | 90 ± 0.33 | SC | -0.04 | -59.79 | 72.61 |
| PC | 892 ± 4.26 | 25 ± 0.58 | 83 ± 3.71 | SC | 1.25 | -62.87 | 60.26 |
| PTN | 908 ± 1.45 | 22 ± 1.15 | 70 ± 0.33 | SC | 2.33 | -63.93 | 35.26 |
| PTM | 897 ± 3.53 | 23 ± 0.58 | 80 ± 3.18 | SC | 1.78 | -63.49 | 43.98 |
| PTW | 872 ± 1.20 | 38 ± 1.15 | 90 ± 0.88 | SC | -1.58 | -41.24 | 81.88 |
| PTD | 890 ± 1.15 | 32 ± 2.31 | 78 ± 1.15 | SC | 0.38 | -48.11 | 50.97 |

*Abbreviations: SL = Sandy Loam, SC = Sandy Clay; GC = General control, TN = treated once, TM = treated monthly, TW = treated weekly, TD = treated daily, PC = Polluted control, PTN = polluted + treated once, PTM = polluted + treated monthly, PTW = polluted + treated weekly, PTD = polluted + treated daily*

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**Table 1c:** Soil Texture at 6-month (g/kg soil) with Percentage Changes

| Treatment | Sand (g/kg) | Silt (g/kg) | Clay (g/kg) | Texture | %Δ Sand | %Δ Silt | %Δ Clay |
|---|---|---|---|---|---|---|---|
| GC | 881 ± 1.76 | 36 ± 1.15 | 83 ± 2.40 | SC | -0.56 | -40.66 | 55.28 |
| TN | 891 ± 2.40 | 28 ± 1.15 | 81 ± 1.33 | SC | 0.91 | -55.56 | 50.31 |
| TM | 889 ± 5.46 | 28 ± 1.53 | 83 ± 3.93 | SC | 0.3 | -55.56 | 64.24 |
| TW | 882 ± 1.15 | 30 ± 1.15 | 88 ± 2.00 | SC | 0.19 | -54.31 | 62.96 |
| TD | 876 ± 1.15 | 40 ± 1.15 | 84 ± 1.15 | SC | -0.98 | -36.51 | 60.51 |
| PC | 883 ± 1.76 | 38 ± 1.15 | 79 ± 0.67 | SC | 0.3 | -43.56 | 51.28 |
| PTN | 893 ± 1.76 | 35 ± 0.58 | 72 ± 2.33 | SC | 0.71 | -42.62 | 37.82 |
| PTM | 905 ± 2.91 | 30 ± 1.15 | 65 ± 1.76 | SC | 2.68 | -52.38 | 16.87 |
| PTW | 903 ± 1.76 | 30 ± 1.15 | 67 ± 2.40 | SC | 1.99 | -53.61 | 34.23 |
| PTD | 884 ± 2.08 | 38 ± 0.88 | 78 ± 1.20 | SC | -0.3 | -37.84 | 50.32 |

*Abbreviations: SL = Sandy Loam, SC = Sandy Clay; GC = General control, TN = treated once, TM = treated monthly, TW = treated weekly, TD = treated daily, PC = Polluted control, PTN = polluted + treated once, PTM = polluted + treated monthly, PTW = polluted + treated weekly, PTD = polluted + treated daily*

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**Soil Chemical Properties**

**Table 2a:** Summary of Soil Chemical Property Changes at 3 and 6 Months

| Parameter & Treatment | Initial Value | 3 Months (% change) | 6 Months (% change) | Key Findings |
|---|---|---|---|---|
| **pH - Unpolluted** | | | | |
| GC | 5.8 | -10.00% | -11.67% | Progressive acidification |
| TW | 5.8 | 13.79% | +13.79% (6.6) | Stabilized improvement |
| TD | 5.8 | 27.59% | +27.59% (7.4) | Alkaline shift |
| **pH - Polluted** | | | | |
| PC | 5.8 | 1.89% | -1.89% | Minimal change |
| PTW | 5.8 | 15.52% | +15.52% (6.7) | Optimal remediation |
| PTD | 5.8 | 31.03% | +31.03% (7.6) | Excessive alkalinization |
| **THC (mg/g) - Polluted** | | | | |
| PC | 100 | -8.72% | -11.39% | Natural attenuation limited |
| PTW | 100 | -35.82% | -42.67% | Effective degradation |
| PTD | 100 | -28.94% | -47.14% | Maximum degradation |
| **WHC (%) - Unpolluted** | | | | |
| TN | 25.4 | -18.50% | -26.30% | Negative impact |

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**Table 2b:** Summary of Soil Chemical Property Changes at 3 and 6 Months

| Parameter & Treatment | Initial Value | 3 Months (% change) | 6 Months (% change) | Key Findings |
|---|---|---|---|---|
| **WHC (%) - Polluted** | | | | |
| PTN | 25.4 | 65.35% | 87.55% | Significant improvement |
| PTM | 25.4 | 52.36% | 73.58% | Consistent benefit |
| PTD | 25.4 | 78.74% | 102.96% | Maximum improvement |
| **EC (µS/cm) - Unpolluted** | | | | |
| TD | 150 | 986.67% | 1472.44% | Extreme salinization |
| TW | 150 | 402.67% | 702.91% | High salinization |
| **EC (µS/cm) - Polluted** | | | | |
| PTW | 150 | 254.67% | 398.20% | Moderate salinization |
| PTD | 150 | 221.33% | 343.92% | Moderate salinization |
| PTN | 150 | -48.67% | -63.58% | Reduced salinity |
| **CEC (meq/100g) - Unpolluted** | | | | |
| TN | 12.5 | 156.80% | 221.57% | Exceptional improvement |
| TW | 12.5 | 132.80% | 186.08% | Substantial improvement |
| **CEC (meq/100g) - Polluted** | | | | |
| PC | 12.5 | 168.80% | 239.22% | Pollution-enhanced |
| PTD | 12.5 | 172.00% | 242.21% | Maximum improvement |
| PTN | 12.5 | 108.00% | 151.01% | Moderate improvement |
| **TOC (g/kg) - Unpolluted** | | | | |
| TM | 15.2 | -31.97% | -15.99% | Carbon loss |
| **TOC (g/kg) - Polluted** | | | | |
| PTM | 15.2 | 68.42% | 114.92% | Maximum increase |
| PTW | 15.2 | 61.84% | 102.88% | Substantial increase |
| PTD | 15.2 | 42.11% | 71.14% | Moderate increase |

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**Table 2c:** Summary of Soil Chemical Property Changes at 3 and 6 Months

| Parameter & Treatment | Initial Value | 3 Months (% change) | 6 Months (% change) | Key Findings |
|---|---|---|---|---|
| **Available N (mg/kg) - Unpolluted** | | | | |
| TD | 8.5 | 3070.71% | 2588.24% | Extreme enrichment |
| TW | 8.5 | 2025.32% | 1788.24% | Very high enrichment |
| **Available N (mg/kg) - Polluted** | | | | |
| PTN | 8.5 | 2632.91% | 2258.82% | Extreme enrichment |
| PTW | 8.5 | 2329.41% | 2888.89% | Maximum enrichment |
| **Available P (mg/kg) - Unpolluted** | | | | |
| TD | 12.3 | 804.12% | 3649.48% | Extreme enrichment |
| **Available P (mg/kg) - Polluted** | | | | |
| PTW | 12.3 | 0.00% | 257.96% | Delayed improvement |
| PTD | 12.3 | 122.09% | 734.30% | Substantial enrichment |
| **Pb (mg/kg) - Unpolluted** | | | | |
| TM | 2.1 | 104.53% | 85.71% | Mobilization |
| TW | 2.1 | 209.06% | 190.48% | Significant mobilization |
| **Pb (mg/kg) - Polluted** | | | | |
| PTN | 2.1 | -71.43% | -83.94% | Effective immobilization |
| PTD | 2.1 | -66.67% | -80.70% | Good immobilization |
| **Fe (mg/kg) - Unpolluted** | | | | |
| TD | 1850 | -92.89% | -85.41% | Extreme depletion |
| GC | 1850 | -74.62% | -65.95% | Substantial depletion |
| **Fe (mg/kg) - Polluted** | | | | |
| PTW | 1850 | 20.13% | 15.14% | Moderate increase |
| PTN | 1850 | 20.38% | 18.38% | Moderate increase |

## DISCUSSION

### Soil Texture and Structure

The shift from sandy loam to sandy clay textures, particularly in polluted soils, indicates hydrocarbon-induced particle aggregation (Aislabie *et al.*, 2004). CME amendment modulated this change, with monthly applications resulting in more moderate clay increases than unamended polluted soil. This suggests CME's organic components buffer excessive aggregation, maintaining favorable pore structures for microbial and root activity (Nunes *et al.*, 2021). The high clay enrichment in weekly-treated polluted soil likely reflects combined effects of hydrocarbons and CME-derived colloidal matter, potentially creating stable aggregates beneficial for long-term structure, though compaction risks require monitoring (Huang *et al.*, 2015).

pH Dynamics: CME amendment dramatically increased soil pH from acidic (5.8) to near-neutral or alkaline levels (up to 7.6), counteracting the acidification typical of hydrocarbon degradation (Varjani, 2017). CME's basic cations and organic matter effectively buffered against organic acid production, creating optimal pH ranges (6.0-7.5) for microbial processes and nutrient availability (Havlin *et al.*, 2014). Progressive acidification in unamended controls highlights CME's corrective capacity. However, excessive alkalinization with daily applications may limit phosphorus and micronutrient availability, indicating weekly or monthly frequencies are optimal (Mengel & Kirkby, 2001).

### Hydrocarbon Degradation

Substantial TPH reductions (up to 47.14%) with frequent CME application demonstrate enhanced biodegradation. This efficacy compares favorably with studies using costlier amendments (Omoni *et al.*, 2020). The frequency-response relationship suggests CME supports sustained microbial activity. The minimal degradation in unamended polluted soil confirms natural attenuation is insufficient, emphasizing the need for biostimulation (Megharaj *et al.*, 2015).

Water Holding Capacity: CME amendment significantly improved WHC in polluted soils (up to +103%), reversing hydrocarbon-induced hydrophobicity and pore structure disruption (Megharaj *et al.*, 2015). Organic matter addition and possibly microbial production of hydrophilic compounds likely drove this restoration. In unpolluted soils, CME sometimes reduced WHC initially, possibly by disrupting existing pore networks before new aggregates formed.

### Electrical Conductivity and Salinity

Dramatic EC increases, especially with daily applications (+1472% in unpolluted soil), highlight salinity as a potential concern (Corwin & Yemoto, 2017). While CME delivers essential nutrients, the accompanying salt load requires management to avoid osmotic stress. Polluted soils showed lower EC increases, suggesting hydrocarbons may buffer salt effects. Optimal strategies must balance nutrient delivery with salt accumulation, favoring less frequent applications or CME dilution.

### Cation Exchange Capacity and Nutrient Retention

Remarkable CEC increases (up to 242%) in amended polluted soils indicate significant long-term fertility restoration. Hydrocarbon contamination typically degrades CEC, but CME reversal through organic matter addition, colloidal particles, and organo-mineral complex formation enhances nutrient-holding capacity beyond original levels (Huang *et al.*, 2015). This improves fertilizer efficiency, reduces leaching, and supports revegetation.

Organic Matter and Carbon Dynamics: Organic carbon increases (up to +115%) in amended polluted soils demonstrate CME's effectiveness in rebuilding soil organic matter, providing energy for microbes and improving structure (Six *et al.*, 2002). Carbon decreases in some unpolluted

treatments may reflect a priming effect, where added organic matter stimulates decomposition of native soil carbon (Blagodatskaya & Kuzyakov, 2008). In contaminated soils, this priming may be directed toward hydrocarbon degradation, offering potential carbon sequestration co-benefits.

**Nutrient Dynamics**

Extreme nutrient enhancements (N up to 3797%, P up to 3649%) underscore CME's exceptional nutrient density, addressing key limitations in contaminated soils (Das & Chandran, 2011). Nitrogen increases likely come from direct addition and stimulation of nitrogen-fixing microbes. Phosphorus availability, often challenging, is improved via organic phosphorus compounds and pH modification. Frequency-dependent responses allow tailored nutrient delivery for different remediation phases.

**Heavy Metal Dynamics**

Contrasting metal responses reveal complex interactions. Lead reductions in amended polluted soils (up to -84%) suggest CME enhances immobilization through organic complexation or precipitation (Bolan *et al.*, 2022). Conversely, lead mobilization in some unpolluted amended soils may result from organic acids in CME, requiring monitoring. Iron increases in amended polluted soils could improve availability for hydrocarbon-degrading enzymes (Varjani, 2017). Overall, metal dynamics appear favorable for remediation.

Treatment Frequency Optimization: Clear frequency-dependent patterns provide crucial application guidance. Weekly and monthly treatments generally offered the best balance between remediation efficacy and avoiding drawbacks like salinity or extreme pH shifts. Daily applications, while maximizing degradation, raised salinity concerns. Single applications showed inadequate persistence, indicating repeated amendments are necessary to maintain biological activity.

## CONCLUSION

This study demonstrates that cassava mill effluent amendment profoundly improves the physicochemical properties of hydrocarbon-contaminated soils. Over six months, CME enhanced soil structure, buffered pH, improved water relations, increased nutrient availability, boosted cation exchange capacity, and stimulated hydrocarbon degradation. Treatment frequency emerged as a critical factor, with weekly and monthly applications providing optimal balances.

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