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Characterization of Cassava Mill Effluent Under Different Environmental Conditions and Its Suitability as a Biostimulant for Soil Remediation

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Abstract

Cassava mill effluent (CME) is a high-volume byproduct of cassava processing in Nigeria, posing environmental hazards but also offering potential as a low-cost biostimulant for soil remediation. This study characterized fresh CME and monitored its physicochemical evolution over seven days under three storage regimes: covered at room temperature, uncovered at room temperature, and uncovered under sunlight. Parameters including pH, cyanide, electrical conductivity (EC), total suspended solids (TSS), organic carbon, macronutrients (N, P, K, Ca, Mg, Na), cation exchange capacity (CEC), and heavy metals were analyzed using standard methods. Fresh CME was moderately acidic (pH 6.23) with high cyanide (437.67 µg/mL), EC (3506.33 µS/cm), and organic matter (233.64 g/L). Storage conditions significantly altered effluent properties. Exposed storage increased pH (6.70–6.87), cyanide (up to 884.67 µg/mL), EC (up to 9884.33 µS/cm), and TSS (4657 mg/L), while covered storage promoted acidification (pH 3.53) and reduced cyanide (230.67 µg/mL). Nutrient analysis revealed exceptionally high potassium (65,519 mg/L) and moderate nitrogen (121 mg/L) and phosphorus (16.99 mg/L). Heavy metals were within regulatory limits. These findings confirm that CME is a nutrient-dense organic amendment, but its application must be carefully managed particularly for cyanide and salinity under optimized storage conditions to ensure safe and effective bioremediation.

Keywords: Cassava mill effluent, cyanide, biostimulation, hydrocarbon remediation, soil amendment, organic waste management.

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INTRODUCTION

Petroleum hydrocarbon pollution remains a persistent environmental threat, particularly in oil-producing regions such as the Niger Delta, where decades of exploration, refining, and accidental spills have rendered large areas of arable land infertile (Ite *et al.*, 2013; Mekonnen *et al.*, 2024). These contaminants alter soil physicochemical properties, reduce microbial diversity, and inhibit plant growth, thereby threatening food security and ecosystem stability (Megharaj *et al.*, 2015). Conventional remediation strategies, including excavation, soil washing, and chemical oxidation, are often prohibitively expensive, technically complex, and may generate secondary pollutants (Sharma, 2020). Consequently, there is increasing interest in sustainable, low-cost, and ecologically compatible bioremediation strategies.

Bioremediation using organic amendments offers a dual advantage: it enhances the metabolic activity of indigenous hydrocarbon-degrading microorganisms while simultaneously improving soil structure and fertility (Das & Chandran, 2011). Among potential organic amendments, agro-industrial wastes are particularly attractive due to their availability, nutrient richness, and low cost. Cassava (*Manihot esculenta*) processing generates large volumes of liquid waste—cassava mill effluent (CME) which is often discharged untreated into the environment, leading to odour nuisance, surface water pollution, and phytotoxicity (Nnaji *et al.*, 2016; Olukanni *et al.*, 2019). Nigeria, as the world's largest cassava producer, generates millions of litres of CME annually, yet its utilization as a resource remains underexplored.

Recent studies have demonstrated that CME contains significant concentrations of nitrogen, phosphorus, potassium, and organic carbon key nutrients required for microbial proliferation and hydrocarbon degradation (Jude *et al.*, 2022). However, the application of CME for bioremediation is constrained by critical knowledge gaps. First, the persistence and dynamics of cyanogenic glycosides and their toxic breakdown product, cyanide, under different storage and environmental conditions are poorly understood (Martínez Toledo *et al.*, 2018). Second, the physicochemical variability of CME especially pH, electrical conductivity, and suspended solids—under different handling regimes has not been systematically characterized. This lack of empirical data hinders the development of standardized protocols for safe and effective field application.

This study was therefore designed to: (i) provide a comprehensive physicochemical characterization of fresh cassava mill effluent; (ii) monitor the evolution of key parameters (pH, cyanide, EC, TSS, nutrients) over a seven-day period under three environmentally relevant storage conditions; and (iii) evaluate the suitability of CME as a biostimulant for hydrocarbon-contaminated soils based on its nutrient profile, heavy metal content, and cation exchange capacity. By establishing evidence-based storage and application protocols, this work aims to facilitate the safe conversion of CME from an environmental pollutant into a value-added resource for soil restoration.

MATERIALS AND METHODS

Sample Collection

Fresh cassava mill effluent (CME) was collected directly from the discharge point of a cassava processing facility in Benin City, Edo State, Nigeria (6°20' N, 5°37' E). The effluent was collected in sterile 20-litre high-density polyethylene (HDPE) containers, transported to the laboratory within one hour, and homogenized by stirring prior to subsampling and experimental setup.

Experimental Design and Storage Conditions
The homogenized CME was aliquoted into 2-litre transparent glass containers and subjected to three storage treatments in triplicate:

1. **Covered, room temperature (RT-C):** Containers sealed with screw caps, stored in the laboratory (ambient temperature 29.6 ± 0.5°C).
2. **Uncovered, room temperature (RT-E):** Containers open to air, stored in the laboratory.
3. **Uncovered, sunlight (Sun-E):** Containers open to air, placed outdoors under direct sunlight (temperature range 27.5–30.5°C; photoperiod approximately 12 h light/12 h dark).

Samples were collected at baseline (Day 0) and on Days 3 and 7 for physicochemical analysis. All analyses were performed in triplicate.

Physicochemical Analysis
pH was measured using a calibrated Jenway 3510 digital pH meter. Electrical conductivity (EC) was determined using a HANNA HI-9835 conductivity meter and expressed in µS/cm. Total suspended solids (TSS) were quantified gravimetrically using a TSS meter (HACH DR/2000).

Cyanide content was determined using the alkaline picrate colorimetric method described by Onwuka (1996). Briefly, 5 mL of sample was distilled into an alkaline picrate solution, and absorbance was read at 490 nm using a Jenway 7305 UV-Vis spectrophotometer. Cyanide concentration was extrapolated from a standard curve prepared using KCN. Organic carbon was analyzed using the Walkley–Black dichromate oxidation method (Walkley & Black, 1947). Briefly, 1 g of dried sample was oxidized with 10 mL of 1N $K_2Cr_2O_7$ and 20 mL of concentrated H_2SO_4. The mixture was titrated with 0.5N $FeSO_4$ after cooling. Organic carbon was calculated from the titre difference; organic matter was derived by multiplying organic carbon by 1.724 (Bao, 2000). Exchangeable cations (K^+, Na^+, Ca^{2+}, Mg^{2+}) were extracted with 1N ammonium acetate (pH 7.0) following the method of Black & Evans (1965). Potassium and sodium were analysed by flame photometry (Jenway PFP7); calcium and magnesium were determined by atomic absorption spectrophotometry (Buck Scientific 210VGP). Cation exchange capacity (CEC) was calculated as the sum of exchangeable bases. Total nitrogen was determined using the Kjeldahl digestion-distillation method (Bremner & Mulvaney, 1982). Available phosphorus was extracted using Bray-1 solution and quantified colorimetrically via the molybdate blue method at 882 nm (Murphy & Riley, 1962). Total phosphorus was determined after perchloric acid digestion. Heavy metals (Pb, Zn, Fe) were analyzed after acid digestion. Exactly 1 g of dried effluent sample was digested with 15 mL of concentrated HNO_3 and 5 mL of $HClO_4$ (4:1 v/v) at 120°C until a clear digest was obtained (Jackson, 1967). The digest was cooled, filtered (Whatman No. 42), and made up to 50 mL with deionized water. Metal concentrations were determined using atomic absorption spectrophotometry (Buck Scientific 210VGP) with appropriate hollow cathode lamps and air-acetylene flame.

Spent lubricating oil (SLO)
Collected from a mechanic workshop in Benin City was similarly characterized for comparative purposes.

Statistical Analysis
All data are expressed as mean ± standard error of the mean (SEM) of triplicate determinations. Differences among treatments and sampling days were analyzed using one-

way analysis of variance (ANOVA) followed by Tukey's honestly significant difference (HSD) post-hoc test. Statistical significance was set at $p < 0.05$. Analyses were performed using GraphPad Prism version 9.4 for Windows (GraphPad Software, San Diego, CA, USA).

RESULTS AND DISCUSSION

Table 1: Physicochemical characteristics of cassava mill effluent (CME) under different storage conditions over seven days

Time	Condition	Temp (°C)	pH	Cyanide (µg/mL)	EC (µS/cm)	TSS (mg/L)
Day 0	Fresh (Baseline)	29.6	6.23 ± 0.09a	437.67 ± 1.45b	3506.33 ± 0.67a	1692.33 ± 1.45a
Day 3	RT, Covered	29.6	4.17 ± 0.09c	327.00 ± 2.31c	4271.67 ± 2.31b	2117.50 ± 0.33b
	RT, Exposed	29.6	4.33 ± 0.07c	373.00 ± 4.93c	4823.33 ± 0.67c	2422.00 ± 1.15c
	Sun, Exposed	27.5	3.80 ± 0.06d	388.00 ± 6.08c	4957.33 ± 1.20d	2460.67 ± 1.20c
Day 7	RT, Covered	30.5	3.53 ± 0.09d	230.67 ± 4.91d	4748.67 ± 2.33c	2403.33 ± 2.03c
	RT, Exposed	30.5	6.87 ± 0.03b	699.00 ± 1.15e	9884.33 ± 2.73e	4657.00 ± 4.62d
	Sun, Exposed	30.5	6.70 ± 0.06b	884.67 ± 3.76f	7934.00 ± 6.35f	4106.33 ± 7.36e

Values are mean ± SEM (n = 3). Different superscript letters within the same column indicate significant differences ($p < 0.05$; Tukey's HSD). RT = room temperature; EC = electrical conductivity; TSS = total suspended solids.

pH Dynamics

The pH of fresh CME (6.23 ± 0.09) indicates moderate acidity, consistent with values reported by Nnaji *et al.* (2016) and Ajao *et al.* (2024), who documented pH ranges of 4.00–6.5 for cassava processing wastewater. This slight acidity is attributable to organic acids released during cassava fermentation. Under covered storage, progressive acidification to pH 3.53 ± 0.09 by Day 7 was observed. This finding aligns with Zhang *et al.* (2017), who demonstrated that anaerobic conditions promote accumulation of volatile fatty acids through fermentative microbial metabolism. Singh *et al.* (2015) similarly reported that sealed storage of organic amendments enhances acidification due to CO_2 accumulation. In contrast, exposed storage conditions resulted in significant alkalization by Day 7, with pH rising to 6.87 ± 0.03 (RT-E) and 6.70 ± 0.06 (Sun-E). This corroborates Martínez Toledo *et al.* (2018), who reported pH increases from 5.2 to 7.1 in aerated cassava wastewater, attributing this to volatilization of fatty acids, ammonification, and photosynthetic activity. Ajao *et al.* (2025) demonstrated that bioaugmentation with cyanide-degrading bacteria increased soil pH from 4.8 to 7.4, supporting the role of microbial activity in pH normalization.

Cyanide Dynamics

The initial cyanide concentration of 437.67 ± 1.45 µg/mL represents a significant toxicological concern. This value is comparable to ranges reported by Olukanni *et al.* (2019) and exceeds WHO (2004) drinking water guidelines. The 47.3% reduction in cyanide under covered storage (to 230.67 µg/mL by Day 7) is consistent with Ugbune *et al.* (2025), who reported progressive cyanide decreases in CME-contaminated soil treated with abattoir sludge over 42–56 days. Kuyucak and Akcil (2013) attributed anaerobic cyanide attenuation to spontaneous hydrolysis and precipitation.

However, the dramatic increase under exposed conditions reaching 884.67 ± 3.76 µg/mL (Sun-E) by Day 7 represents a critical finding. This paradox may result from continued enzymatic hydrolysis of cyanogenic glycosides outpacing degradation (Chandra *et al.*, 2014), evaporative concentration, and microbial cyanogenesis (Martínez Toledo *et al.*, 2018). Ajao *et al.* (2025)

identified several cyanide-degrading bacteria (*Pseudomonas fluorescens*, *Bacillus pumilus*) in CME environments, suggesting that under aerobic conditions, initial hydrolysis may temporarily release cyanide before subsequent degradation.

Electrical Conductivity and Total Suspended Solids

Baseline EC (3506.33 ± 0.67 µS/cm) increased dramatically under exposed conditions, reaching 9884.33 ± 2.73 µS/cm (RT-E) by Day 7. This 182% increase exceeds the FAO irrigation guideline of 3000 µS/cm (Corwin & Yemoto, 2017) and indicates significant salinity risk if applied undiluted. Isimah *et al.* (2025) documented elevated EC in CME-affected soils, emphasizing the need for controlled discharge. TSS followed similar trends, with exposed treatments reaching 4657.00 ± 4.62 mg/L. These values exceed ranges reported by Ajao *et al.* (2024) (2709–2812 mg/L), suggesting evaporative concentration and microbial biomass accumulation (Bansal *et al.*, 2020.

Table 2: Comprehensive physicochemical characterization of fresh cassava mill effluent (CME) and spent lubricating oil (SLO), with regulatory comparison

Parameter	CME (This Study)	SLO (This Study)	FEPA (1991) Limit	WHO (2004) Limit
Total N (mg/L)	121.00 ± 0.31	68.40 ± 0.91	—	50
Available N (mg/L)	5.56 ± 0.01	2.97 ± 0.01	—	—
Total P (mg/L)	16.99 ± 0.10	21.70 ± 0.16	5	10
Available P (mg/L)	3.71 ± 0.23	0.33 ± 0.01	—	—
TOC (g/L)	135.84 ± 0.13	—	—	—
OM (g/L)	233.64 ± 1.00	—	—	—
K (mg/L)	65519.00 ± 1.20	59669.00 ± 7.51	—	—
Na (mg/L)	1564.00 ± 0.88	1447.00 ± 4.98	200	200
Ca (mg/L)	121.00 ± 0.88	59.67 ± 1.45	—	200
Mg (mg/L)	64.00 ± 0.88	1.90 ± 0.08	—	50
CEC (meq/100g)	67361.00 ± 9.94	61178.00 ± 7.69	—	—
Pb (mg/L)	ND	0.13 ± 0.03	0.1	0.1
Zn (mg/L)	1.97 ± 0.05	1.00 ± 0.00	5	5
Fe (mg/L)	78.00 ± 2.60	108.00 ± 2.60	20	20

Values are mean ± SEM (n = 3). ND = Not Detected; FEPA = Federal Environmental Protection Agency (Nigeria); WHO = World Health Organization; TOC = total organic carbon; OM = organic matter; CEC = cation exchange capacity. "—" indicates no established standard.

Nitrogen, Phosphorus, and Organic Matter

Total nitrogen (121.00 ± 0.31 mg/L) and total phosphorus (16.99 ± 0.10 mg/L) in fresh CME are comparable to liquid anaerobic digestates used as biostimulants (Olukanni *et al.*, 2019). Available nitrogen (5.56 mg/L) and phosphorus (3.71 mg/L) substantially exceed SLO values, confirming CME's superior biostimulation potential. Das and Chandran (2011) emphasized that nitrogen and phosphorus availability limits hydrocarbon biodegradation. However, total

phosphorus exceeds FEPA (1991) and WHO (2004) discharge limits, indicating eutrophication potential if released untreated. Organic matter content (233.64 ± 1.00 g/L) is exceptionally high, providing energy substrates for heterotrophic bacteria and improving soil structure (Six *et al.*, 2002; Nunes *et al.*, 2021).

Exchangeable Cations and Cation Exchange Capacity

Potassium (65,519.00 ± 1.20 mg/L) dominates the cation profile, reflecting cassava's potassium-intensive nature (Havlin *et al.*, 2014). Sodium (1564 mg/L) exceeds regulatory limits, requiring dilution prior to application. However, high divalent cations (Ca^{2+}: 121 mg/L; Mg^{2+}: 64 mg/L) mitigate sodicity risk (Richards, 1969). The exceptional CEC (67,361 meq/100g) far exceeds typical mineral soils (5–40 meq/100g), reflecting the colloidal nature of effluent particulates (Huang *et al.*, 2015). This property indicates CME application could significantly enhance soil nutrient retention. Omotosho *et al.* (2024) demonstrated that 25% CME concentration provided optimal benefits for soil amendment.

Heavy Metal Profile

Lead was not detected in CME, contrasting with SLO (0.13 mg/L) and with CME-affected soils studied by Abasiekong *et al.* (2025). Zinc (1.97 mg/L) is well below regulatory limits. Iron (78 mg/L) exceeds FEPA/WHO limits but is substantially lower than SLO (108 mg/L) and is amenable to biological treatment Ajao *et al.* (2025) achieved >70% heavy metal reduction using cyanide-degrading bacteria. Iron is an essential micronutrient for microbial electron transport systems involved in hydrocarbon oxidation. The favorable heavy metal profile supports CME's safety as a soil amendment, consistent with findings that treated effluent can be applied without adverse effects (Omotosho *et al.*, 2024).

Conclusion and Recommendations

This study demonstrates that cassava mill effluent is a chemically dynamic material with significant potential as a biostimulant for soil remediation, but its properties—particularly pH, cyanide, and salinity—change profoundly under different storage conditions. Fresh CME exhibits high nutrient content (K: 65,519 mg/L; N: 121 mg/L; P: 16.99 mg/L), exceptional organic matter (233.64 g/L), remarkable CEC (67,361 meq/100g), and a favorable heavy metal profile, supporting its bioremediation value. However, the condition-dependent variability documented requires careful management: covered storage reduces cyanide by 47% but causes extreme acidification (pH 3.53), while exposed storage normalizes pH but paradoxically elevates cyanide to potentially toxic levels (884 μg/mL) and dramatically increases salinity (EC: 9884 μS/cm).

Based on these findings and recent literature, the following recommendations are proposed:

1. **Storage Protocol**: Store CME under covered conditions for 7–14 days prior to use to reduce cyanide burden through natural attenuation (Ugbune *et al.*, 2025).
2. **Pre-Application Analysis**: Conduct routine analysis of pH, cyanide, and EC before field application due to the significant variability documented.
3. **Dilution**: Dilute CME with clean water at ratios of at least 1:5 (CME:water) to ameliorate salinity risks. Omotosho *et al.* (2024) demonstrated optimal benefits at 25% concentration.
4. **Bioaugmentation**: Consider co-application with cyanide-degrading bacteria (*Pseudomonas* and *Bacillus* species) to enhance detoxification, as Ajao *et al.* (2025) achieved >80% cyanide reduction and >70% heavy metal removal within 20 days.

5. **Regulatory Compliance**: Ensure application rates do not result in accumulation of phosphorus or sodium beyond regulatory limits, and monitor receiving soil quality periodically.

By implementing these protocols, CME can be safely transformed from an environmental pollutant into a value-added resource for restoring hydrocarbon-contaminated soils, supporting circular economy principles in cassava-producing regions.

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