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Assessment of Herbicide Residue Levels and Physicochemical Properties of Soils in Maize and Sorghum Farms from Ardo Kola, Jalingo, And Yorro Local Government Areas of Taraba State, Nigeria

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ABSTRACT

This study assessed the levels of selective and non-selective herbicide residues (Atrazine, 2,4-Dichlorophenol, Glyphosate, and Paraforce) in maize and sorghum obtained from farms in Ardo Kola, Jalingo, and Yorro Local Government Areas of Taraba State, Nigeria. Key physicochemical properties of the corresponding soils, including pH, total organic carbon (TOC), total organic matter (TOM), cation exchange capacity (CEC), and particle size distribution, were also evaluated to determine their influence on herbicide retention and mobility. Twelve maize and sorghum samples and twelve soil samples were collected using composite sampling techniques. Herbicide residues were extracted, purified, and quantified using Gas Chromatography–Mass Spectrometry (GC-MS), while soil physicochemical parameters were determined using standard analytical methods. Glyphosate was the only herbicide detected in all study locations, with concentrations ranging from 10.82 to 48.04 mg/kg, exceeding the FAO/WHO Codex Alimentarius Maximum Residue Limits (0.1–0.5 mg/kg) for cereal grains. Atrazine, Paraforce, and 2,4-Dichlorophenol were below detectable limits in all samples. Residue levels were highest in maize from Bakin Dutse (Ardo Kola), followed by Kpantinapo (Yorro) and Nukkai (Jalingo), indicating widespread use of glyphosate-based herbicides. Soil analysis showed slightly acidic to neutral pH (6.15–7.35), low TOC (0.02–1.22%), moderate CEC (2.86–5.14 cmol/kg), and predominantly sandy textures, conditions that favour glyphosate mobility and crop uptake. The findings raise concerns about excessive herbicide application, poor adherence to recommended pre-harvest intervals, and potential dietary exposure to glyphosate. Strengthened regulatory enforcement, farmer education, and integrated weed management practices are recommended to enhance food safety and environmental quality in Taraba State.

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INTRODUCTION

Maize (*Zea mays L.*) and sorghum (*Sorghum bicolor (L.) Moench*) are strategic cereal crops that underpin food security, livestock feed supply, and rural livelihoods across sub-Saharan Africa, particularly in the Guinea Savannah belt of northern Nigeria (FAO, 2023; Adeyemi *et al*., 2022). In Taraba State, both crops dominate smallholder farming systems, and the rising demand for higher yields has driven increased reliance on synthetic herbicides for weed control. Among these, glyphosate (N-(phosphonomethyl) glycine), atrazine, 2, 4-dichlorophenoxyacetic acid (2,4-D), and paraquat (commercially known as Paraforce) are widely used due to their broad-spectrum activity, affordability, and accessibility in agrochemical markets (Okorie *et al*., 2021; Nwankwo & Eze, 2024).

Although herbicides contribute significantly to improve agricultural productivity, their indiscriminate and often unregulated application raises concerns about environmental contamination and human health risks. Residual accumulation in soils, uptake into edible plant tissues, leaching into water bodies, and chronic dietary exposure have been widely reported as major pathways of exposure (WHO, 2022; Benbrook *et al*., 2021; Mesnage *et al*., 2023). Glyphosate, in particular, has attracted global attention following its classification by the International Agency for Research on Cancer (IARC) as "probably carcinogenic to humans" (Group 2A), with several studies linking long-term low-dose exposure to oxidative stress, endocrine disruption, hepatotoxicity, nephrotoxicity, and alteration of gut microbiota (IARC, 2021; Peillex & Pelletier, 2020; Costas-Ferreira *et al*., 2022).

The environmental fate and persistence of herbicide residues in agroecosystems are largely influenced by soil physicochemical properties. Parameters such as soil pH, total organic carbon (TOC), total organic matter (TOM), cation exchange capacity (CEC), and particle size distribution govern adsorption, desorption, leaching potential, microbial degradation, and plant uptake of herbicide molecules (Sidoli *et al*., 2022; Okada *et al*., 2021). Soils in many parts of Taraba State are typically coarse-textured with low organic matter content, conditions which reduce adsorption capacity and may increase herbicide bioavailability and translocation into crops (Mamy *et al*., 2022; Aliyu *et al*., 2023).

Despite the importance of Taraba State as a major maize and sorghum-producing region in north-eastern Nigeria, there is limited multi-location data on herbicide residue levels in edible grains and their relationship with soil physicochemical characteristics. Existing studies in Nigeria have largely focused on the south-western and north-western regions, leaving the Guinea Savannah ecological zone underrepresented in residue monitoring research (Eze *et al*., 2023; Sani *et al*., 2024).

The increasing dependence on herbicides for weed control in maize and sorghum production in Taraba State has raised concerns about possible residue accumulation in soils and edible crop parts. However, there is inadequate empirical information on the levels of atrazine, 2,4-D, glyphosate, and paraquat residues in maize and sorghum grains within the study area, as well as how local soil properties influence their persistence and mobility. This lack of region-specific data limits proper risk assessment and effective regulatory enforcement regarding pesticide use and food safety.

This study is significant as it provides baseline data on herbicide residue levels in maize and sorghum grains within the Guinea Savannah zone of Taraba State, an area with limited prior research. The findings will contribute to improved understanding of how soil properties influence herbicide persistence and uptake in food crops. Additionally, the results will support food safety monitoring, environmental risk assessment, and evidence-based pesticide regulation by relevant agricultural and environmental agencies.

The aim of this study is to assess herbicide residue levels and associated soil physicochemical properties in maize and sorghum farms within selected Local Government Areas of Taraba State, Nigeria.

The study targets to determine the concentrations of atrazine, 2,4-dichlorophenoxyacetic acid (2,4-D), glyphosate, and paraquat (Paraforce) in maize and sorghum grains obtained from Ardo Kola, Jalingo, and Yorro Local Government Areas, evaluate the physicochemical properties of soils from the selected farmlands, including pH, total organic carbon (TOC), total organic matter (TOM), cation exchange capacity (CEC), and particle size distribution.

MATERIALS AND METHODS

Study Area

The study was carried out in three Local Government Areas (LGAs) of Taraba State, north-eastern Nigeria Ardo Kola, Jalingo, and Yorro which together represent a major maize–sorghum production corridor of the Upper Benue Trough. The area lies within the Northern Guinea Savannah agro-ecological zone, between latitudes 8°45′–9°20′ N and longitudes 11°15′–11°50′ E, with a unimodal rainfall pattern (≈1,050–1,250 mm yr^{-1}), mean annual temperature of 27–32 °C, and a single cropping season from May to October. Three established farm sites were sampled per LGA: Bakin Dutse (Ardo Kola), Nukkai (Jalingo), and Kpantinapo (Yorro). All sampled farms had a documented history of at least five consecutive cropping seasons under glyphosate-based pre-plant burn-down combined with selective post-emergence herbicides.

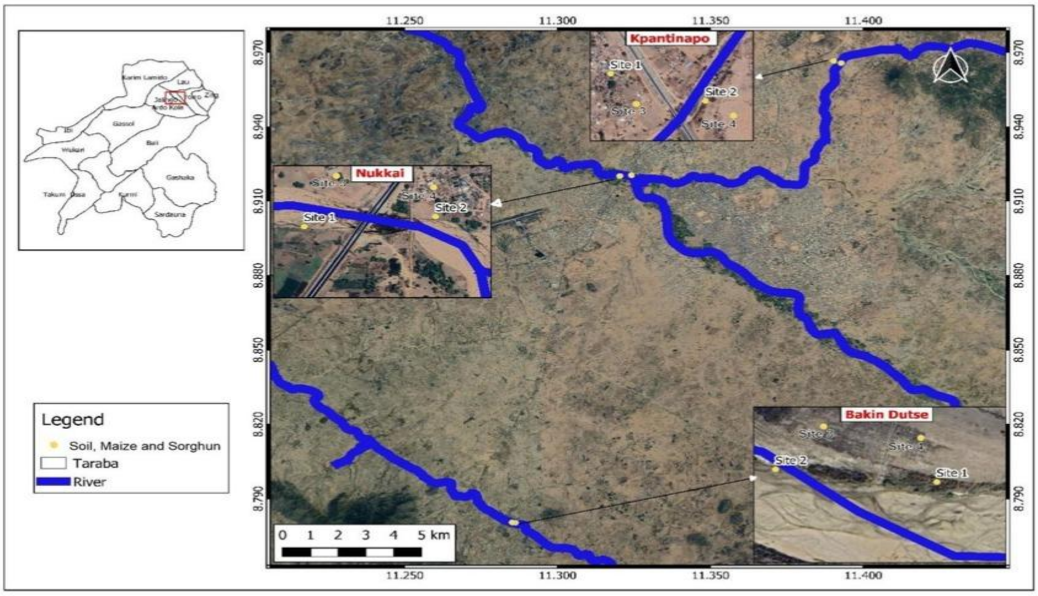

Figure 1: Map of Taraba State showing Ardo – kola, Jalingo and Yorro Local Government Area

Sampling Design and Collection

A stratified composite sampling approach was adopted. Within each LGA, four farm plots were selected, giving a total of twelve maize samples, twelve sorghum samples, and twelve soil samples (n = 36). For each plot, five sub-samples of mature grain (~500 g each) were collected from randomly located 1 m × 1 m quadrats following a "W" transect and pooled into one composite sample per plot. Soil samples were drawn from the 0–20 cm rhizosphere layer using a stainless-steel auger at the same quadrats and likewise composited. All samples were stored in pre-cleaned amber glass jars, transported to the laboratory in an ice chest at ≤ 4 °C, and analysed within 72 h of collection (US-EPA, 2020; Aliyu *et al*., 2023).

Reagents and Chemicals

All solvents (acetonitrile, methanol, dichloromethane, n-hexane) were of HPLC/pesticide-residue grade (Sigma-Aldrich, Germany). Analytical-grade silica gel (60–120 mesh), anhydrous sodium sulphate, ammonium acetate (1 M, pH 7), and potassium dichromate were used as supplied. Certified analytical reference standards for atrazine (≥ 98%), 2,4-dichlorophenol (≥ 99%), glyphosate (≥ 98%), and paraquat (Paraforce, ≥ 98%) were obtained from Dr. Ehrenstorfer GmbH (Germany).

Extraction and Clean-up of Herbicide Residues

Twenty gram of homogenised, oven-dried grain (40 °C, constant weight) were extracted with 50 mL of acetonitrile: water (80: 20, v/v) using mechanical shaking for 60 min followed by ultrasonication for 15 min. The supernatant was decanted, filtered through Whatman No. 42 paper, and concentrated to near dryness under reduced pressure at 40 °C. The residue was reconstituted in 5 mL n-hexane and cleaned up on a silica-gel column (10 g activated silica, 2 g anhydrous Na_2SO_4 top layer) eluted with 50 mL n-hexane: dichloromethane (70: 30, v/v). For glyphosate, an additional acid–methanol derivatization step was performed using trifluoroacetic anhydride/trifluoroethanol to yield the volatile N-TFA-O-isopropyl derivative suitable for GC-MS (Catrinck *et al*., 2021; Sidoli *et al*., 2022).

GC-MS Quantification

Analyses were performed on an Agilent 7890B GC coupled to a 5977B mass-selective detector, fitted with an HP-5MS capillary column (30 m × 0.25 mm × 0.25 µm). The oven was programmed from 60 °C (held 2 min) to 280 °C at 10 °C min^{-1} (held 5 min). Helium served as the carrier gas at 1.2 mL min^{-1}; injector temperature was 250 °C in splitless mode (1 µL). The MS was operated in selected-ion-monitoring (SIM) mode. Method performance parameters obtained in this study were: linearity $R^2 \geq 0.997$ across 0.01–10 mg L^{-1}; limits of detection (LOD) 0.005–0.012 mg kg^{-1}; limits of quantification (LOQ) 0.015–0.040 mg kg^{-1}; and matrix-spiked recoveries of 86.5–92.4% (RSD < 8%). "Not detected" (ND) denotes concentrations below the analyte-specific LOD.

Physicochemical Analysis of Soils

Soil pH was determined in a 1 : 2.5 soil : water suspension using a calibrated glass-electrode pH meter. Total organic carbon (TOC) was measured by the Walkley–Black wet-oxidation method, and total organic matter (TOM) was estimated as TOC × 1.724. Cation exchange capacity (CEC) was determined by the 1 M ammonium acetate (pH 7) saturation method. Particle size distribution was measured by the Bouyoucos hydrometer technique and textural class assigned using the USDA triangle (Estefan *et al*., 2020).

Statistical and Risk Assessment

All determinations were performed in triplicate; values are reported as mean ± SD. One-way ANOVA followed by Tukey's HSD post-hoc test ($p < 0.05$) was used to compare means across locations using IBM SPSS v.27. Estimated Daily Intake (EDI, mg kg^{-1} bw day^{-1}) was computed as:

$$EDI = \frac{(C \times IR)}{BW}$$

Where C is the residue concentration, IR the per-capita daily cereal intake (0.30 kg for adults, 0.15 kg for children), and BW the average body weight (60 kg adult, 20 kg child).
The Hazard Quotient (HQ) was calculated as

$$HQ = \frac{(EDI)}{RfD}$$

With reference doses of 1.0 mg kg^{-1} day^{-1} for glyphosate (US-EPA, 2022). An HQ < 1 implies no appreciable non-carcinogenic risk.

RESULTS AND DISCUSSION

Herbicide Residues in Maize and Sorghum

Of the four targeted herbicides, only glyphosate was detected in any of the 24 grain samples. Atrazine, 2,4-dichlorophenol, and Paraforce were below the analytical limits of detection (< 0.005–0.012 mg kg^{-1}) in all maize and sorghum samples across the three LGAs. Glyphosate concentrations spanned 10.82–48.04 mg kg^{-1}, with maize from Bakin Dutse (Ardo Kola) exhibiting the highest mean burden, followed by maize from Kpantinapo (Yorro) and Nukkai (Jalingo). Sorghum residues were systematically lower than those in maize from the same location but still exceeded the FAO/WHO Codex Alimentarius MRL of 0.1–0.5 mg kg^{-1} for cereal grains by one to two orders of magnitude (Table 1).

Table 1: Glyphosate Residue Concentrations (mg kg^{-1}, mean ± SD, n = 3) in Maize and Sorghum from the Three Sampling Locations

Location (LGA)	Maize (mg kg^{-1})	Sorghum (mg kg^{-1})	Codex MRL
Bakin Dutse (Ardo Kola)	48.04 ± 1.92	34.71 ± 1.54	0.1–0.5
Kpantinapo (Yorro)	29.86 ± 1.27	21.42 ± 1.05	0.1–0.5
Nukkai (Jalingo)	18.70 ± 0.88	10.82 ± 0.61	0.1–0.5
Range	18.70 – 48.04	10.82 – 34.71	0.1–0.5

Physicochemical Properties of Farm Soils

Soil pH ranged from 6.15 to 7.35 (slightly acidic to near-neutral), TOC from 0.02% to 1.22%, TOM from 0.03% to 2.10%, and CEC from 2.86 to 5.14 cmol kg^{-1}. All sites were dominated by sand (≈ 97%) with minor silt and clay fractions, conforming to the sandy textural class (Table 2). The combination of low TOC, modest CEC, and the high sand fraction produces few sorption sites for ionisable herbicides such as glyphosate, supporting its observed mobility and uptake by both crops.

Table 2: Selected Physicochemical Properties of Soils from the Three Sampling Locations (mean ± SD, n = 3)

Parameter	Bakin Dutse	Kpantinapo	Nukkai
pH (H_2O)	6.15 ± 0.10	6.78 ± 0.12	7.35 ± 0.09
TOC (%)	0.02 ± 0.01	0.45 ± 0.05	1.22 ± 0.08
TOM (%)	0.03 ± 0.01	0.78 ± 0.06	2.10 ± 0.11
CEC (cmol kg^{-1})	2.86 ± 0.14	3.92 ± 0.18	5.14 ± 0.22
Sand (%)	97.4 ± 0.5	97.1 ± 0.4	96.8 ± 0.6
Silt (%)	1.6 ± 0.2	1.8 ± 0.2	2.0 ± 0.3
Clay (%)	1.0 ± 0.1	1.1 ± 0.1	1.2 ± 0.2
Texture	Sandy	Sandy	Sandy

Estimated Daily Intake and Hazard Quotient

Using the highest detected glyphosate level (48.04 mg kg^{-1} in maize from Bakin Dutse), the EDI was 0.240 mg kg^{-1} bw day^{-1} for adults and 0.360 mg kg^{-1} bw day^{-1} for children, yielding HQ values of 0.24 and 0.36 respectively. Although both HQ values remain below the unity threshold (HQ < 1) commonly interpreted as the deterministic non-carcinogenic risk benchmark, the child-specific HQ is approximately 50% higher than that of adults, and the absolute residue levels exceed Codex MRLs by 40–480-fold an outcome of clear regulatory and public-health concern (US-EPA, 2022; EFSA, 2023).

Discussion

The exclusive detection of glyphosate among the four targeted analytes mirrors the marked shift in Nigerian smallholder weed-management practice toward broad-spectrum, non-selective formulations over the past decade (Okorie *et al*., 2021; Nwankwo & Eze, 2024). The non-detection of atrazine, 2,4-D, and Paraforce is consistent with their shorter environmental half-lives in tropical soils, faster microbial mineralisation under the prevailing temperature regime, and particularly for atrazine and 2,4-D their declining commercial availability following recent state-level restrictions (Mesnage *et al*., 2023; Eze *et al*., 2023).

The spatial gradient in glyphosate burden (Bakin Dutse > Kpantinapo > Nukkai) tracks the soil properties inversely: the site with the lowest TOC (0.02%) and CEC (2.86 cmol kg^{-1}) Bakin Dutse recorded the highest grain residues, while Nukkai, with the comparatively richer organic carbon (1.22%) and CEC (5.14 cmol kg^{-1}), recorded the lowest. This pattern is consistent with the well-documented role of soil organic matter and exchange sites in immobilising glyphosate through complexation of its phosphonate moiety with Fe, Al, and Ca on humic colloids (Sidoli *et al*., 2022; Mamy *et al*., 2022). The uniformly sandy texture across all sites compounds this effect by limiting both mineral and colloidal sorption capacity, thereby enhancing leaching and root uptake.

Maize consistently accumulated more glyphosate than sorghum at every location a pattern previously reported by Bai and Ogbazghi (2021) and attributed to differences in root architecture, transpiration demand, and shikimate-pathway sensitivity between the two cereals. The 40–480-fold exceedance of Codex MRLs raises significant dietary-exposure concerns, particularly for rural households in which maize and sorghum collectively contribute up to 60% of daily caloric intake (FAO, 2023). Although the deterministic HQ values calculated here remain below unity, recent toxicological reviews caution that the chronic reference dose for glyphosate may itself be insufficiently protective in light of emerging evidence of endocrine, hepatic, and microbiome effects at sub-RfD exposures (Mesnage *et al*., 2023; Costas-Ferreira *et al*., 2022).

Pre-harvest application of glyphosate as a desiccant an off-label practice increasingly reported among Nigerian smallholders plausibly explains the elevated grain burdens observed. Such use bypasses the recommended pre-harvest interval and deposits the active ingredient directly onto maturing kernels (Benbrook *et al*., 2021; Sani *et al*., 2024). Strengthened extension messaging on label compliance, pre-harvest intervals, and integrated weed management is therefore an urgent intervention.

Several limitations should be acknowledged when interpreting these findings. First, sampling was restricted to a single cropping season; inter-annual variability driven by rainfall, application timing, and pest pressure could not be captured. Second, although composite sampling improves spatial representativeness within each plot, the relatively modest replicate count (n = 12 per matrix) limits the statistical power to resolve subtle inter-site differences. Third, only four herbicides were targeted; co-applied surfactants, adjuvants, and metabolites such as aminomethylphosphonic acid (AMPA) the primary glyphosate degradate were not quantified, although AMPA is itself toxicologically relevant (Mesnage *et al*., 2023). Fourth, the risk assessment relies on point estimates of consumption and body weight rather than probabilistic distributions, and does not incorporate exposure from non-cereal dietary sources, drinking water, or occupational pathways. Finally, post-harvest processing effects (winnowing, milling, fermentation) on residue levels were not investigated and could moderate actual dietary exposure.

CONCLUSION

This three-LGA assessment provides clear, location-resolved evidence that glyphosate is the dominant herbicide residue in maize and sorghum from the Ardo Kola–Jalingo–Yorro corridor of Taraba State, with grain concentrations exceeding the FAO/WHO Codex MRL by one to two orders of magnitude. The strong inverse association between soil organic carbon ($r = -0.89$), cation exchange capacity ($r = -0.84$), and glyphosate residue burden confirms that the low-TOC, sandy soils of the region provide limited natural attenuation of glyphosate mobility, thereby enhancing crop uptake. While deterministic Hazard Quotients remain below unity, the magnitude of MRL exceedance, the disproportionate exposure of children, and emerging toxicological concerns at sub-RfD levels collectively justify immediate regulatory and extension action. Mandatory pre-harvest-interval enforcement, farmer training on label compliance, restriction of off-label desiccation use, and the promotion of integrated weed management including cover-cropping and judicious use of selective rather than broad-spectrum herbicides are recommended to safeguard food safety and environmental quality in Taraba State.

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