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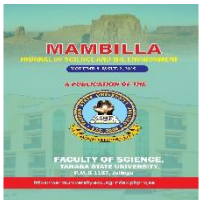

EVALUATION OF HERBICIDE RESIDUE LEVELS AND ASSOCIATED PHYSICOCHEMICAL PROPERTIES OF SOILS IN SELECTED AGRICULTURALLY INTENSIVE ZONES OF TARABA STATE, NIGERIA.

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ABSTRACT

Herbicide application is a common practice in intensive agriculture, yet its environmental persistence raises significant concerns regarding soil quality and ecosystem health. This study evaluates the levels of herbicide residues and associated physicochemical properties of soil in agriculturally intensive zones of Taraba state, Nigeria, specifically within Ardo-kola, Jalingo and Yorro Local Government Areas. Soil samples were collected and analyzed using Gas Chromatography-Mass Spectrophotometer (GC-MS) to detect and quantify residues of selected herbicides, including glyphosate and 2,4-Dichlorophenoxyacetic (2,4-D) as well as their degradation products. Concurrently, key soil parameters- pH, total organic carbon (TOC), particle size distribution and cation exchange capacity (CEC) were determined to assess their influence on herbicide persistence. Glyphosate was detected in significant concentration, averaging 1.68 mg/kg, while 2, 4-D was not detected in its parent form, however, its degradation product 2, 4- chlorophenol, was present at an average concentration of 0.65 mg/kg. Variations in residue distribution appeared correlated with organic carbon content and soil texture. The finding underscores the persistence and transformation of herbicides in soil with potential implication of groundwater contamination, crop safety, and human health. The study recommends the adoption of stricter regulatory controls and sustainable agricultural practices to mitigate herbicide- related environmental risks.

1. Introduction

Herbicides are chemicals used to manipulate or control undesirable vegetation. Herbicide application occurs most frequently in row-crop farming, where they are applied before or during planting to maximize crop productivity by minimizing other vegetation. Use of herbicides is a common way of eradicating unwanted plant growth. According to (Zakari *et al.,* 2020), herbicides are substances or cultured biological organisms used to kill or suppress the growth of unwanted plants and vegetation's selectively or non-selectively. There are several herbicides in the market; however, there is a perceived trade-off between effectiveness and sustainability. One of the most widely commercialized herbicides in the world is glyphosate. It is considered "virtually ideal" because of its broad spectrum and its supposedly low toxicity (Duke & Powles 2008). Herbicides are in great demand in the domestic market and are widely used in paddy rice fields. It is usually mixed with other pesticides, such as chlomethoxynil, bensulfuron-methyl, oxadiazon, or dymron to make a mixture to be both pre-and post-emergence herbicides as well as to inhibit both annual and perennial weeds from growing (Shobha, 2014). The potential effects of herbicides are strongly influenced by their toxic mode of action and their method of application. Herbicides can act by inhibiting cell division, photosynthesis or amino acid production or by mimicking natural plant growth hormones, causing deformities (Ross and Childs 1996). Application methods include spraying unto foliage, applying to soils and applying directly to aquatic systems.

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The use of pesticide in farming activities has significantly provided more hope and assurance for sustainable food security in most countries of the world. However, the residues of the pesticides could be a threat to human beings and other non-target organisms after they have performed their primary roles. It is reported that only 1 % of sprayed pesticides are effective, while the 99 % of the pesticides applied are often released to non-target soils, water bodies, and atmosphere and finally absorbed by almost every organism. The intensive application of herbicides has resulted in the contamination of the atmosphere, ground/waste water, land and agricultural products which includes; wheat, corn, fruit, vegetables. It also results in the direct pollution of food and food products as well as the biological system.

This study evaluates the levels of herbicide residues and associated physicochemical properties of soil in agriculturally intensive zones of Taraba state, Nigeria, specifically within Ardo-kola, Jalingo and Yorro Local Government Areas.

2. Materials and Methods

2.1 Study Area

The study areas were selected based on intensive agricultural activities of the areas: Ardo-Kola (8.8333° N, 11.3667° E), Yorro (8.8833° N, 11.5667° E) and Jalingo (8.8935° N, 11.3596° E) Local Government Areas of Taraba State, Nigeria. The sampling stations were selected because of their proximity to farming activities and the use of herbicides based on observations. The people living in these areas are predominantly farmers.

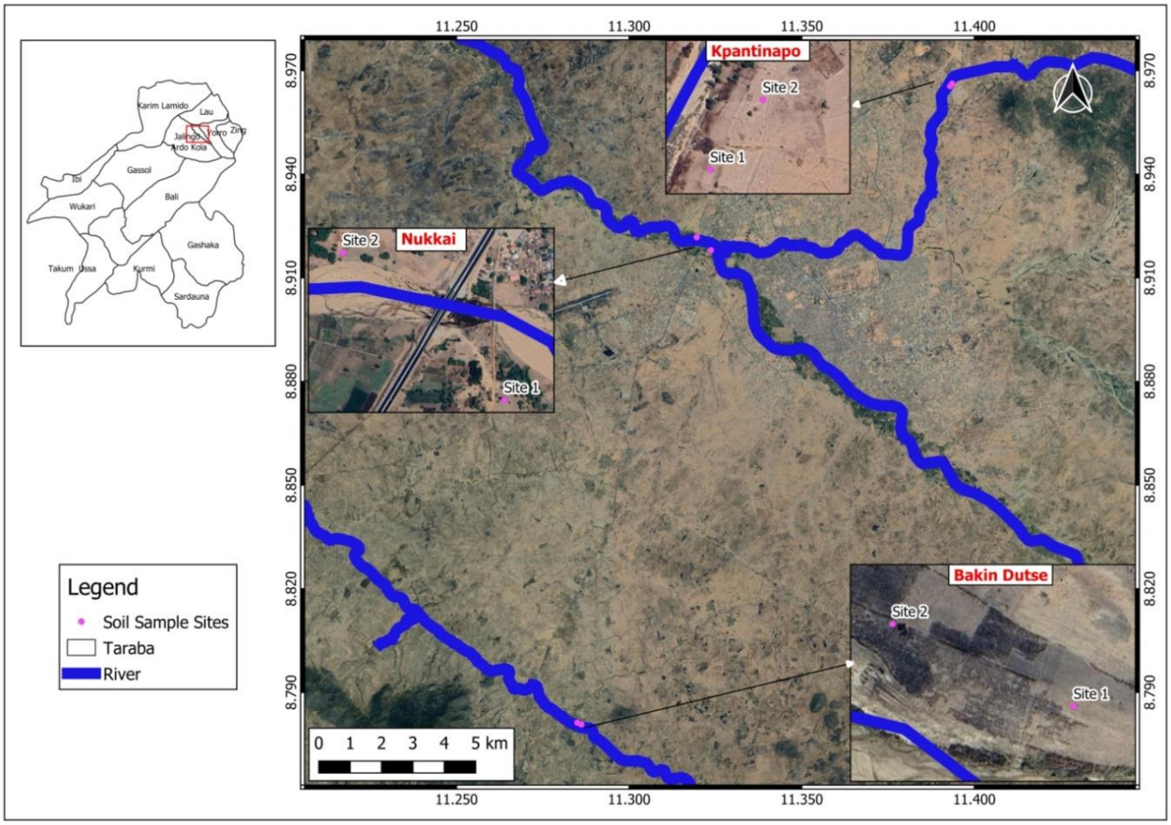

Figure 1: Map of Taraba showing sampling sites deployed in the study

2.2 Sample Collection

For each sampling station, an area of 70 × 70 cm was delineated into 100 grids of 7 × 7 cm and 33 grids were randomly selected for sample collection. Samples were collected with steel auger at 0-15 cm (topsoil) and 15-30 cm (subsoil) height respectively. The collected samples were thoroughly mixed to form a composite sample.

The soil samples were collected at two different stations per location. Samples were transported to the laboratory under temperature-controlled conditions to prevent pesticides degradation.

2.3 Preparation of soil samples

In the laboratory, soil samples were weighed and dried at 40 °C until constant dry weight were obtained. The samples were homogenised using Agatha mortar and pestle, pulverised through 0.5 mm mesh sieve and stored at -4 °C until further analysis. The soil samples were then extracted following method adopted by Darko *et al.* (2007) with some slight modifications. About 10 g of soil sample was weighed and transferred into extraction thimble that was washed with n-hexane and acetone and oven dried. The sample was extracted using 100 mL of n-hexane - acetone mixture 4:1 v/v for eight hours using soxhlet extractor. The extract was evaporated to dryness using a rotary evaporator at 45 °C. Each extract was dissolved in 10 mL n-hexane and subjected to clean-up procedure.

2.4 Clean-up Procedure

A column of about 15 cm (length) × 1 cm (internal diameter) was packed with about 10 g activated silica gel prepared in a slurry form in n-hexane. About 2 g of anhydrous sodium sulphate was placed at the top of the column to absorb any water in the sample or the solvent. The column was pre-eluted with 20 mL of n-hexane without the exposure of the sodium sulphate layer to air. The reduced extract was placed in the column and allowed to sink below the sodium sulphate layer. Elution was done with 2 × 10 mL portions of the extracting solvent. The eluate was then collected, dried with anhydrous sodium sulphate and then evaporated to dryness under a stream of analytical grade nitrogen (99.999 %).

2.5 Analysis of Soil Sample

The dried eluate was analyzed using an Agilent 6890 gas chromatography system coupled with a mass spectrophotometer (GC-MS) following AOAC (1990) guidelines. This setup enabled the detection of herbicide residues in the soil samples, even at trace concentrations, overcoming matrix interferences that other detectors might miss.

3. Results and Discussion

The results of the physicochemical analysis of soils from the sampling stations are presented in Tables 1, 2 and 3.

Table 1: Results of analysis of levels of herbicide residues and physicochemical parameters of samples obtained from Nukkai sampling site in Jalingo Local Government Area

	Sampling Station			
Parameter	N1	SN1	TN2	SN2
2,4-Dichlorophenoxyacetic Acid (mg/kg)	ND	ND	ND	ND
2,4-Dichlorophenol (mg/kg)	1.20±0.00	1.01±0.00	1.305±0.00	1.012±0.00
Glyphosate (mg/kg)	2.426±0.00	ND	2.473±0.00	ND
Glycine (mg/kg)	ND	ND	ND	ND
AMPA (mg/kg)	ND	ND	ND	ND
pH	6.000±0.00	5.800±0.00	5.200±0.00	5.500±0.00
CEC (cmolkg^{-1} soil)	7.910±0.00	6.910±0.00	7.550±0.00	8.710±0.00
TOC (%)	1.560±0.00	1.130±0.00	1.610±0.00	1.390±0.00
TOM (%)	2.690±0.00	1.950±0.00	2.780±0.00	2.400±0.00
Particle Size				
Clay	1.080±0.00	1.010±0.00	1.090±0.00	1.060±0.00
Silt	0.020±0.00	0.010±0.00	0.030±0.00	0.000±0.00
Sand	98.900±0.00	98.980±0.00	98.880±0.00	98.940±0.00

Key:TN = topsoil Nukkai; SN = subsoil Nukkai; AMPA= aminomethylphosphonic acid; ND = not detected;
CEC = cation exchange capacity; TOC= total organic carbon; TOM= total organic matter.

Table 2: Results of analysis of levels of herbicide residues and physicochemical parameters of samples obtained from Bakin-Dutse sampling site in Ardo-Kola Local Government Area

Parameter	Sample Station			
	TB1	SB1	TB2	SB2
2,4-Dichlorophenoxyacetic Acid (mg/kg)	ND	ND	ND	ND
2,4-Dichlorophenol (mg/kg)	0.425±0.00	ND	0.425±0.00	ND
Glyphosate (mg/kg)	2.895±0.00	ND	2.895±0.00	ND
Glycine (mg/kg)	ND	ND	ND	ND
AMPA (mg/kg)	ND	ND	ND	ND
pH	5.600±0.00	5.500±0.00	5.600±0.00	5.700±0.00
CEC (cmolkg^{-1} soil)	7.520±0.00	11.100±0.00	7.890±0.00	8.850±0.00
TOC (%)	1.730±0.00	1.520±0.00	1.370±0.00	1.520±0.00
TOM (%)	2.980±0.00	2.620±0.00	2.360±0.00	2.620±0.00
Particle Size:				
Clay	1.070±0.00	1.070±0.00	1.050±0.00	1.060±0.00
Silt	0.020±0.00	0.020±0.00	0.020±0.00	0.010±0.00
Sand	98.910±0.00	98.910±0.00	98.930±0.00	98.930±0.00

Key:TB = topsoil Bakin-Dutse; SB = subsoil Bakin-Dutse; AMPA= aminomethylphosphonic acid; ND = not detected; CEC = cation exchange capcity; TOC = total organic carbon; TOM = total organic matter

Table 3: Results of analysis of levels of herbicide residues and physicochemical parameters of samples obtained from Kpantinapo sampling site in Yorro Local Government Area

Parameter	Sample Station			
	TK1	SK1	TK2	SK2
2,4-Dichlorophenoxyacetic Acid (mg/kg)	ND	ND	ND	ND
2,4-Dichlorophenol (mg/kg)	1.188±0.00	ND	1.188±0.00	ND
Glyphosate (mg/kg)	2.222±0.00	2.317±0.00	2.535±0.00	2.410±0.00
Glycine (mg/kg)	ND	ND	ND	ND
AMPA (mg/kg)	ND	ND	ND	ND
pH	5.500±0.00	4.900±0.00	4.900±0.00	5.900±0.00
CEC (cmolkg^{-1} soil)	10.800±0.00	7.410±0.00	6.240±0.00	9.690±0.00
TOC (%)	0.740±0.00	0.650±0.00	0.890±0.00	0.650±0.00
TOM (%)	1.280±0.00	1.120±0.00	1.530±0.00	1.120±0.00
Particle Size:				
Clay	1.030±0.00	1.050±0.00	1.060±0.00	1.040±0.00
Silt	0.010±0.00	0.020±0.00	0.010±0.00	0.010±0.00
Sand	98.960±0.00	98.930±0.00	98.930±0.00	98.950±0.00

Key: TK= topsoil Kpantinapo; SK= subsoil Kpantinapo; AMPA= Aminomethylphosphonic Acid; ND = not Detected; CEC = cation exchange capacity; TOC = total organic carbon;
TOM = total organic matter

The soils deployed in this work from all the sampling sites are considered as sandy (≥ 98.9 %) with very small amounts of clays (1.0 -1.09 %) and silts (0.00 - 0.02 %). From Tables 1, 2 and 3 above, 2,4-Dichlorophenoxyacetic acid was not found in all the samples from the different sites. This may be due to rapid degradation as 2,4-D is known to degrade quickly (half-life of 2-4 weeks) in aerobic soils because of the microbial activities (like *Pseudomonas* and *Sphingomonas*) or photodegradation as sunlight can accelerate 2,4-D break down, especially in tropical climates like Taraba State. Glyphosate, (N-(phosphonomethyl)glycine) was consistently detected in the topsoils in the range of 2.222-2.895 mg/kg and absent in Nukkai and Bakin-Dutse subsoils. It was present in the range of 2.317 - 2.410 mg/L in Kpantinapo subsoils. The soil pH across the topsoils of the three sampling locations ranged from 4.9 - 6.0, indicating moderately to slightly acidic conditions.

Cation exchange capacity (CEC) is a soil property that allows it to bind positively charged ions cations such as Ca^{2+}, Mg^{2+} and K^{+}. It is also a measure that expresses the ability of the soil to adsorb cations, and corresponding with plants capability of absorbing nutrients through the roots in ionic form (Jones and Kathrin, 2016). The sandy soils used in this study have low CEC of 6.24 - 11.10 cmolkg^{-1}soil because of the low clay contents (1.03 - 1.08 %) and low total organic matters (1.12 - 2.98 %). This low CEC enhances glyphosate adsorption and retention on the soil particles especially at a low pH range of 4.9 - 6.0, which was the pH range of the soils used in the study. This is reflected in the levels of glyphosate recorded for the topsoils in all the sampling sites. Hazelton and Murphy (2007) reported that low CEC of 6 - 12 cmolkg^{-1}soil favours adsorption of glyphosate and supports our findings. The absence of glyphosate in the subsoils of Nukkai and Bakin-Dutse reveals that the glyphosate was strongly bound to the soil particles of the topsoils. pH significantly influences herbicide behaviour, nutrient availability and microbial activities. Enhanced adsorption of glyphosate in acidic soils is well documented in literature (Piccolo *et al.,* 1994). Lower pH generally favours glyphosate adsorption because the molecule becomes less negatively charged and more attracted to the soil surface, according to ScienceDirect.com corroborating our findings.

The topsoils had TOM (2.36 - 2.98 %) > 1.5 % across the sampling stations; this enhances glyphosate binding, increases its adsorption and retention to the topsoil particles, reduces its availability for microbial degradation and also decreases their mobility into the subsoils, preventing leaching into groundwater. Slightly acidic soils with appreciable organic matter have been reported to degrade slowly, inhibit microbial activities and show strong glyphosate adsorption (Okada *et al.*, 2022) and in a similar study conducted by Adeyinka *et al.* (2021) reported that high TOM (3.5 - 5.0 %) >1.5 % retained glyphosate effectively. These results agree with the findings of our work for the topsoils. In the subsoils across the sampling sites, glyphosate was only detected at Kpantinapo with the concentrations of: SK1= 2.317 mg/kg at of pH = 4.9 and SK1= 2.41 mg/kg at of pH = 5.9. Among other factors, the presence of glyphosate in SKI could be attributed to its low TOM value of 1.120 that is < 1.5 thereby promoting its leaching into the subsoil of this station. In a similar work by Oluwatosin *et al.* (2023) shows that low-TOM in soils (< 1.5 %) enhances herbicide leaching and supports Kpantinapo's result. Also the findings of Bento *et al.* (2022) who reported that sandy soils (98 % sand) and low pH enhances mobility of the leached glyphosate into the subsoils, further gives credence to the results obtained for Kpantinapo's subsoil. Increase in adsorption is reduced by leaching and may also decrease the effectiveness of herbicides in controlling weeds, particularly if the herbicides are strongly bound to the organic matter. The effect of organic carbon on herbicide degradation and activity can vary depending on the specific herbicide, the type of organic matter, and other soil properties (ScienceDirect.com.).

The highest detected glyphosate level in our work was 2.895 mg/kg and is below the EPA's ecological soil screening level of 15 mg/kg, yet chronic exposure risks warrant monitoring (EPA, 2020).

2,4-Dichlorophenol (2,4-DCP) was detected in all the topsoils deployed in the study with concentration ranging from 0.425 - 1.305 mg/kg and a mean value of 1.03 mg/kg. It was only detected in the subsoil of Nukkai (SN1, SN2), with the concentration of 1.012 mg/kg. This depicts wide spread contamination, possibly due to agricultural use or runoff. The slightly acidic nature of the topsoils (pH = 4.9 - 6.0) is one of the prevailing factors responsible for the observed concentrations of 2, 4-DCP in the topsoils and Li *et al.* (2015) who juxtaposed that acidic conditions slows down its break down strongly supports our observations. Also the persistence of 2,4-Dichlorophenol in the top soils of sampling stations with TOC ≥ 1.56 mg/L suggest its slower degradation in these soils with moderate organic carbon and this assertion strongly agrees with the studies done by Góngora-Echeverría *et al.* (2020) who earlier reported that soils with moderate organic carbon of 1.56 % TOC exhibit such degradation.

It was earlier established in our discussion that sandy soil of low CEC and moderate organic matter of 2.36 - 2.98 % of the topsoils across the sampling locations were used in this study. The adsorption of 2,4-DCP in soils involves both electrostatic interactions (between charged species) and hydrophobic interactions (between the organic molecule and the soil's organic matter). In sandy soils with lower CEC (6 -12 cmolkg^{-1}soil) the reduced number of charges makes electrostatic forces less significant, therefore promotes hydrophobic interactions, which are stronger for the neutral form of 2,4-DCP, to play a more dominant role in the adsorption process.

2,4-DCP was effectively adsorbed by the soil particles of the topsoil in range of 0.425 - 1.305 mg/L across the topsoils of the three sampling locations. It was absent in the subsoils of Bakin-Dutse and Kpantinapo but present the subsoils of Nukkai in the level of 1.012 mg/L each. The TOM of 1-3 % has also been reported in literature for optimal adsorption of 2,4-DCP at a pH range of 4.9 – 6.0. At this pH values, hydrophobic interactions between the neutral 2,4-DCP molecule and the soil organic matter become more prominent, contributing to strong adsorption, according to Research Gate.

2,4-DCP can exist in both neutral and charged forms depending on the pH of the soil. In acidic conditions (pH 4.6 - 6.0), a significant portion of 2,4-DCP will be in its neutral, hydrophobic form and thus more readily adsorbed by the soil. As the pH approaches the pKa of 2,4-DCP (around 7.85), the molecule becomes increasingly ionized, leading to weaker adsorption. The organic matter provides hydrophobic sites for 2,4-DCP to bind to, enhancing adsorption, according to Research Gate. The absence of 2,4-DCP in the subsoils of Bakin-Dutse and Kpantinapo may be that it has been adsorbed totally and retained on the topsoils of these sites. Its presence in the Nukkai subsoils is likely due to other contributing environmental factors. It may be as a result of the slightly low pH that favoured microbial degradation of 2,4-DCP the at topsoils and subsequently leached into the subsoils of this location.

The high concentrations of 2,4-Dichlorophenol in Nukkai and Kpantinapo topsoils exceeded typical degradation rates, suggesting historical accumulation or repeated applications. 2,4-Dichlorophenol often degrades faster in aerobic soils (topsoil), but its persistence in Nukkai and Kpantinapo topsoils may reflect repeated applications or slower microbial activities (ATSDR, 2020) and could harm soil microbiota and aquatic systems if transported via erosion. Its absence in Bakin-Dutse and Kpantinapo subsoils is likely due the slightly acidic nature (pH= 5.5 - 5.7) of these subsoils that promotes its degradation via microbial activities. It is also possible that these soils may have history of repeated herbicide application and harbours microbial population better adapted to degrading 2,4 - DCP. Optimal pH for degradation may vary but generally neutral to slightly acidic pH favours microbial degradation and aligns with the studies of Merini *et al.* (2022) that reported rapid degradation under aerobic conditions.

The TOC values follow similar trends with TOM for effective adsorption of 2,4-DCP in sandy soils of low pH = 4.9 - 6.0. In summary, to enhance 2,4-DCP adsorption in sandy soil, the organic carbon content should be high and the soil pH within the range of 4.6 - 6.0. This will maximize hydrophobic interactions and enhance the adsorption of the neutral form of 2,4-DCP. However, the relationship is complex and can be influenced by other factors like specific herbicide. The reason for the persistence presence of glyphosate in the subsoils of Kpantinapo is not fully clear and could be as result of repeated application of herbicides, accumulation history or other interwoven environmental factors coming into play.

Glycine and aminomethylphosphonicacid (AMPA) (metabolites of glyphosate) were not detected in any of the samples from the sampling sites. This may be due to possible recent application of glyphosate in the farms hence their absence in the samples. Glycine is not a major metabolite of glyphosate. Glyphosate primarily breaks down to AMPA, Silva *et al.* (2018) and Nowack (2002) in their works suggested that the absence of AMPA may be as a result of limited glyphosate degradation possibly due to short degradation time or anaerobic conditions.

It is reported that only 1 % of sprayed pesticides are effective, while the 99 % of the pesticides applied are often released to non-target soils, water bodies, and atmosphere and finally absorbed by almost every organism (Jiang *et al.,* 2011). The fate of the pesticides in the soil environment in respect of pest control efficacy, non-target organism exposure and offsite mobility has become a matter of environmental concern (Hafez and Thiemann, 2003) potentially because of the adverse effects of pesticide chemicals on soil microorganisms (Araújo *et al.,* 2003), which in turn may affect soil fertility (Schuster & Schröder, 1990).

4. Conclusion

This study has demonstrated the presence of herbicide residues in agricultural soils of Ardo-Kola, Jalingo, and Yorro Local Government Areas, with glyphosate being the most prevalent. The variability in herbicide concentration across different locations suggests differential application rates and environmental conditions affecting herbicide persistence.

The analysis of soil physicochemical parameters revealed potential influences on herbicide retention and degradation. Given the potential risks associated with herbicide residues, including soil contamination, water pollution, and health hazards, there is a need for improved management practices and regulatory oversight. This research underscores the importance of continuous monitoring and sustainable herbicide usage to mitigate environmental and health impacts.

5. Recommendations

1. Implement stricter regulations on herbicide application to minimize environmental contamination.

2. Encourage farmers to adopt integrated weed management practices to reduce reliance on chemical herbicides.
3. Promote the use of environmentally friendly and biodegradable herbicides.
4. Conduct regular soil monitoring to assess herbicide residues and their impact on soil health.

5. Educate farmers and agricultural workers on the safe handling and application of herbicides.

6. Enhance public awareness regarding the potential risks associated with herbicide residues in food and water sources.

7. Support research on alternative weed control methods to reduce dependency on chemical herbicides.

8. Improve waste disposal and handling practices to prevent herbicide contamination of water bodies.

9. Establish buffer zones around agricultural areas to reduce herbicide drift and non-target exposure.

10. Encourage sustainable agricultural practices to maintain soil health and prevent long-term environmental degradation.

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