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Research Article

Assessing the Profile of Glyphosate Residues in Maize Grains Sold in the Federal Capital Territory Abuja, Nigeria

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ABSTRACT

The profile of glyphosate (GLY) residues in maize grains sold in the Federal Capital Territory Abuja (FCT), Nigeria, was assessed. Twenty-six maize grain samples of yellow and white morphotypes were collected from some selected markets within the FCT, Abuja. Analytical methods on the pulverized maize grains included solvent extraction using (acetonitrile/water (55:45), clean-up of pesticide residues as described by the Association of Official Analytical Chemists Quick Easy Cheap Efficient Rugged and Safe method (AOAC-QuEChERS) and quantification done by high performance liquid chromatography (HPLC) with UV detector. The results showed that the mean concentration of glyphosate in the maize grains ranged below the detection limit (BDL) to 24.30±0.002 mg/kg with a mean glyphosate concentration of 3.474 mg/kg was detected. Three samples representing 11.54% of the samples collected from Gwagwalada and Nyanya markets violated the World Health Organization (WHO)/Food Agricultural Organization (FAO) Codex Alimentarius Commission standards of 5.0 mg/kg values recorded for Acceptable Daily Intake (ADI) for all samples were within an acceptable limit of 0.1 mg/kg and it was also observed that all values recorded for Estimated Daily Intake (EDI) were lower than ADI which suggest that the maize grains is toxicologically safe for the consumer. The values obtained for Health risk index indicated that they are all <1, suggesting that the consumer populations were not at risk. The relatively high concentration of glyphosate residue in maize grains from Gwagwalada market which was above regulatory standard is of great concern and needs to be further investigated and mitigated.

Keywords: Maize grains, contamination, residues, herbicides, health hazards, human exposure, Nigeria

1. INTRODUCTION

Maize grains are one of the major staples produced in North Central Nigeria where the Federal Capital Territory (FCT) is located. The use of herbicides is highly merited to improve crop yield and quality by reducing or inhibiting the growth of weeds. Herbicide plays a fundamental role in reducing crop yield losses and stabilizing the supply of crop produce all year round (Salazar & Rand, 2020; Larsen et al., 2021). Currently, glyphosate-based herbicide is the most used herbicide worldwide (Nerozzi et al., 2020). Glyphosate is used for the management of perennial weeds such as crowfoot grass (*Dactyloctenium aegyptium*), wire grass (*Sporobolus diander* and *Sacciolepsis africana)*, as well as by acting as a harvesting aid accelerating crop dry down (Anjorin et al., 2020). N-phosphonomethyl glycine, also known as glyphosate (GLY), is an organophosphorous herbicide used to eradicate a variety of undesirable weeds (Kalofiri et al., 2021). Hundreds of GBHs are currently being sold under various labels in over 100 countries worldwide (Antier et al., 2020). Since glyphosate-tolerant crops like corn, soybeans, and cotton were introduced, its use has dramatically expanded. GLY is sold under various brand names, including Roundup® (Nerozzi et al., 2020; Novotny, 2020). Globally, fields are currently treated with more than 1.4 billion pounds of glyphosate per year (Beckie et al., 2020).

Guidelines established by the US Environmental Protection Agency (EPA) specify tolerance limits for the presence of glyphosate in food products and produce, such as maize grains (5 mg/kg), cowpea (15 mg/kg) (Shaw, 2021). As a result, the sale of GBHs is strictly controlled and maximum residue limits (MRLs) have been set for glyphosate residues in food (Antier et al., 2020). According to FAO (2002), the MRL is an index that represents the highest concentration (expressed in mg/kg) of the herbicide residue that is legally permitted or accepted in food or animal feed after the use of pesticides. Nevertheless, the environment and public health are negatively impacted by the herbicide's hazardous ingredients (Davoren & Schiestl, 2018, Rani et al., 2021). There are concerns regarding glyphosate potential toxicity and potential long-term adverse effects on human health such Parkinson's disease, endocrine disorders, liver cancer, and cardiovascular diseases (Fagbohun et al., 2023). Other reported damages of GLY are male infertility (Jarrel et al., 2020) and the mammalian immune system (Peilex & Pelletier, 2020).

The presence of glyphosate residues above 5 mg/kg MRLs in maize grains might pose a serious threat to human as stated earlier. To provide safe food and ensure food security, there is a need to determine the hazardous compounds in food sources such GLY (Kolakowski et al., 2020; Soares et al., 2021). The use of GLY in pre-planting weed killers is a prevalence among FCT farmers. Herbicide residues have been reported in food grains such as maize previously (Zhang et al., 2020). However, there is a dearth of information on GLY residues in maize in FCT. Therefore, quantification of GLY residues in maize grains sold in FCT market is one way of determining the level of risk and potential health hazards to human's population due to consumption of GLY contaminated maize grains. The residual concentration of GLY in maize grains will be used to estimate human risk assessment using acceptable daily intake (ADI), estimated daily intake (EDI), and health index (HI) with a view to ascertain the health status of the consumer in the study area.

2. MATERIALS AND METHODS

2.1. The study location

The area of study is the FCT which is the administrative capital of Nigeria. The study area lies between Lat. 8.25° N and 9.21° N and Long. 6.45° E and 7.39° E and with an estimated population of 1.8 million. It has a total area of 713 Km^2 (NBS, 2020). The territory's borders are Kaduna State to the North, Kogi State to the South, Nasarawa State to the East, and Niger State to the West. The FCT has six area councils; Kuje, Abaji, Bwari, Gwagwalada, Kwali, and Abuja Municipal Area Council (AMAC) Maize grains were sampled from some selected markets within the 6 area councils.

2.2. Sample collection

Maize grain samples were collected from retail markets located within the six local councils which include, Abaji, Kwali, Sheda, Gwagwalda, Teaching Hospital, Bwari, Lugbe, Karmo, Kado, Garki village, Nyanyan and Kuje markets. In each market, 5 samples of yellow and white maize were randomly collected from each of the markets and bulked together as a composite sample, giving a total of 25 samples in all the markets considered and the samples were transferred swiftly to the laboratory and kept in a -20ºC refrigerator pending analytical determination. The sample's code and name are as follows: AYM: Abaji Yellow Maize, AWM: Abaji White Maize, KWYM: Kwali Yellow Maize : KWWM: Kwali white Maize, SYM: Sheda Yellow Maize, SWM: Sheda White Maize, GWM: Gwagwalada White Maize, GYM: Gwagwalada Yellow Maize, THSWM: Teaching Hospital White Maize, THSYM: Teaching Hospital Yellow Maize, BWM: Bwari White Maize, BYM: Bwari Yellow Maize, LYM: Lugbe Yellow Maize, LWM: Lugbe White Maize, KYM: Karimu Yellow Maize, KWM: Karimu White Maize, FMYM: Fish Market Yellow Maize, FMWM: Fish Market White Maize, UWM: Utako White Maize, UYM: Utako Yellow Maize, G'WM: Garki White Maize, G'YM: Garki Yellow Maize, NWM: Nyanya White Maize, NYM: Nyanya Yellow Maize, WYM: Wuse Yellow Maize, and WWM: Wuse White Maize.

2.3. Chemicals and materials

In this study, the chemicals used were GLY standard, formic acid, acetonitrile, acetone, and methanol, all solvents are 99.90% HPLC grade and purchased from Sigma-Aldrich USA. Solid phase extraction tube (SPE tubes), preloaded column tubes with graphitized carbon black (GCB), primary secondary amine (PSA), and ceramic disc were purchased from Bioccomma Limited Hong Kong.

2.4. Sample preparation procedure

A modified QuEChERS method was used for the preparation of sample extracts (AOAC Official method, 2007-01). Ten grams (10 g) of fine ground subsample was placed in a centrifuge tube (50 mL) and hydrated with 10 mL water. Followed by the addition of 15 mL acetonitrile and the mixture was vortexed vigorously for 5 min. Furthermore, 0.5 g disodium hydrogencitrate sesquihydrate, 1 g trisodium citrate dihydrate, 4 g anhydrous magnesium sulphate, and 1 g sodium chloride were added. The mixture was immediately vortexed again for another five minutes, then centrifuged at 4500 rpm for 5 min. At this stage, low-temperature clean step was performed. For this, an aliquot of the supernatant was transferred into a clear glass test tube and stored for at least 2 h in a freezer (- 20˚C). The extract was decanted and transferred to a centrifuge tube containing 100 mg anhydrous magnesium sulphate, 75 mg C18, and 20 mg PSA per mL acetonitrile extract. The tube was again vortexed for 0.5 min and centrifuged at 4500 rpm for 2 min. An aliquot of the supernatant was transferred into glass test tubes and acidified by adding 15 µL of 5% (v/v) formic acid in acetonitrile per mL of extract.

2.5. Preparation of glyphosate standard solution

Stock solution containing 1000 mg/L of GLY was prepared by accurately weighing 10 mg of GLY standard in a 5 mL beaker and dissolved in 5 mL acetonitrile and later, transferred quantitatively into a 10 mL standard volumetric flasks and make-up to the mark with acetonitrile to prepare 1000 ppm. Working standard solutions (ranging from 5-40

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mg/mL) were prepared using the dilution method. All solutions were stored under refrigeration below - 4ºC pending analysis (Halim et al., 2013).

2.6. Instrumentation

Chromatographic analyses were performed using CECIL 3500 High-Performance Liquid Chromatography (HPLC) equipped with a binary pump and UV-visible wavelength detector (VWD), all purchased from CECIL, England. The chromatographic separation of the target analytes was performed based on previous methods (Bedassa et al., 2015) with minor modifications. An isocratic elution with a binary mobile phase comprising 45% water (solvent A) and 55% acetonitrile (solvent B) was used throughout the analysis. Before the subsequent sample/extract injection, the HPLC column was washed by adjusting the mobile phase composition to 5% water (solvent A) and 95% acetonitrile (solvent B) for 15 min and then was conditioned with the mobile phase (55% acetonitrile and 45% water) for an additional 20 minutes. Analysis was performed with the mobile phase flow rate of 0.3 mL/min, column temperature set at 30ºC, injection volume 10 µL, and monitoring wavelength of 254 nm. Chromatograms of each of the samples and data acquisition were affected by power stream Adept CECIL 4900.

2.7. Analytical method validation

The calibration curves were obtained by injecting five different concentrations of the GLY herbicide standards in a range of 5-40 mg/mL (Santilio et al., 2019). The limit of detection (LOD) and limits of quantification (LOQ) were determined as signal to noise ratio 1:3 and 1:10, respectively.

2.8. Recovery studies

Two samples of pulverized maize grains weighing 10 g each were selected. One sample was spiked with 10 mg/kg of GLY standard and the mixture was properly mixed by a vortex mixer. The other sample was left as a control (unspiked) sample and two samples were extracted, purified, and evaluated as described in the previous section. The recoveries of the GLY were calculated from the concentration of the analytes that were detected in the spiked samples using the formulae below (Liao et al., 2018; Akande et al., 2020).

2.9. Health risk assessment

The Estimated Daily Intakes (EDI) of the GLY residue and food consumption assumption were used to determine long-term health risks to consumers. The food consumption rate for cereals such as maize is quoted to be 0.1062 kg/person/day with an average body weight of 60 kg for an adult (MoFA, 2010).The EDI was obtained as stated in Equation 1.

$$\text{Percent recovery} = \frac{\text{Conc in spike sample} - \text{Conc in the unspike sample}}{\text{Amount added}} \times 100 \qquad \text{Eq. 1}$$

The health risk indices were obtained by dividing the EDI by their corresponding values of acceptable daily intake (ADI) (Akomea-Frempong et al., 2017; FAO/WHO, 2019), assuming an average adult's body weight of 60 kg. When the health risk index >1; the food involved is

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considered a risk to the consumers. When the health index is < 1, the food involved is considered acceptable (Darko & Akoto, 2008).

$$(EDI) = \sum \frac{C \times IR \times EF \times ED}{BW \times AT} \quad \text{Eq. 2}$$

Where C is the concentration of the GLY residue in maize grains in mg/kg, IR is the ingestion rate or consumption rate for an adult (0.1062 kg), EF is exposure frequency (365 days), ED is exposure duration which represents 55.12 years life expectancy rate, BW: Body weight of adults: 60 kg, AT: Average time of exposure. To understand the human health risk factors of contaminated maize, the Joint Food and Agriculture Organization of the United Nations FAO/WHO Codex Alimentarius Commission has set the Acceptable Daily Intake (ADI) of 0.1 mg/kg in maize grains respectively (FAO/WHO, 2011). The Hazard Risk Index (HI) was computed according to the following formula:

$$(HRI) = \frac{EDI}{ADI} \quad \text{Eq. 3}$$

The estimation of non-carcinogenic health hazards from the consumption of maize grains was determined by equation 2 as provided by the United States Environmental Protection Agency (EPA, 2007; Akande et al., 2020). HRI < 1 indicates no potential non-carcinogenic health risk, whereas HRI > 1 indicates non-carcinogenic potential chronic health risk. The hazard index (HRI) was calculated with the formulae in Equation 4.

$$(HI) = \sum HRI \quad \text{Eq. 4}$$

3. RESULTS AND DISCUSSION

Analysis of herbicide residues in food is a key tool for monitoring the levels of human exposure to herbicide residues and determining food quality and safety. The percentage recoveries of the GLY standard were found to be acceptable at 90.01-101%, which indicates that the reproducibility of the method was satisfactory and the calibration curve of the studied analysts shows satisfactory linearity over the selected concentration range with a regression correlation coefficient (r^2) of 0.987332 for GLY. The limits of detection and quantification of GLY standard were 0.011 mg/kg, and 0.022 mg/kg respectively. The result obtained from each sample's chromatogram is as displayed in Table 1. It was revealed that 24 out of the 26 samples, i.e., 92.31%, were contaminated with GLY residues at various levels of concentrations. Glyphosate contamination range is between 0-24.3 mg/kg with a mean residual concentration of 3.474 mg/kg. All maize grains collected from Garki markets (G'YM and G'WM) were below the detection limit. While the least mean concentration (1.00 mg/kg) was recorded for LYM, the highest (24.3 mg/kg) was found in GWM samples. It was revealed that the GWM grains had the highest concentration followed by GYM and NWM had the most contaminated maize grains in the FCT, Abuja. This observation might be as a result of heavy usage of GLY during the maize planting season to control weeds in the zone.

Zhang et al. (2020) reported in their study the range of GLY residues in maize grains in China as 0.0912-0.477 mg/kg, which was lower than the one reported in this study as the highest concentration was 24.3 mg/kg in some of the samples investigated. Moreover, Kaun et al. (2023) reported the range of GLY concentration in fresh maize as 0.04-0.09 mg/kg. Kolakowski et al. (2020) found GLY in popcorn (0.018-0.020 mg/kg), corn (0.092 mg/kg), corn flour (0.0051-0.045 mg/kg) and cowpea (0.0051-8.60 mg/kg). Fagbohun et al. (2023) recorded 0.11-44.33 mg/kg GLY in different varieties of cowpea from FCT, Abuja. Research report on GLY profile in maize grains, wheat, cowpeas, and their products is depicted in Table 2. The grains from GWM, GYM, and NWM with the following respective concentrations, 24.3, 8.52, 5.48 mg/kg which was made up of 11.54% of analyzed samples were contaminated with GLY residue above the 5 mg/kg MRL set by CODEX and the European Union (EU).

Table 1. Glyphosate residue concentration (mg/kg) in maize grain samples from Nigeria Market

S/N	Sample ID	Glyphosate residue Conc. (mg/kg) in maize samples	S/N	Sample ID	Glyphosate residue Conc. (mg/kg) in maize samples
1	AYM	1.147 ±0.012	14	LWM	2.700 ± 0.011
2	AWM	3.510 ± 0.021	15	KYM	4.280 ± 0.031
3	KWYM	3.989 ± 0.023	16	KWM	3.980 ± 0.002
4	KWWM	3.820 ± 0.013	17	FMYM	2.340 ±0.016
5	SYM	1.250 ±0.011	18	FMWM	1.700 ± 0.010
6	SWM	1.100 ± 0.021	19	UWM	1.234 ±0.032
7	GWM	24.30 ± 0.002	20	UYM	2.231 ± 0.045
8	GYM	8.520 ±0.012	21	G'WM	*BDL
9	THSWM	4.740 ±0.020	22	G'YM	*BDL
10	THSYM	2.300 ± 0.032	23	NWM	5.480 ± 0.000
11	BWM	2.000 ± 0.032	24	NYM	3.200 ± 0.0021
12	BYM	1.420 ±0.032	25	WYM	3.420 ± 0.040
13	LYM	1.000 ±0.022	26	WWM	2.200 ± 0.020

*BDL: Below Detection Level

Table 2. Grain glyphosate concentration (mg/kg)

Cereals and cereal-based foods	Glyphosate Concentration (mg/kg)	References
Maize	0.0912-0.477	Zhang et al. (2020)
Fresh maize	0.04-0.09	Kuan et al. (2023)
Corn	0.092	Kolakowski et al. (2020)
Popcorn	0.018-0.029	
Corn flour	0.051- 0.045	
Cowpea	0.11-44.33	Fagbohun et al. (2023)
Wheat	< 0.13	Jan et al. (2018)
	1.06-1.13	
Breakfast cereal	< 0.001–0.291	Zoller et al. (2018)
Soybean	0.1, 1.6, 1.8	USDA (2020)
	1.94	Christopher et al. (2021)
	0.049	
Maize meal	0.093-0.027	Christopher et al. (2021)
Instant maize meal	0.034-0.035	
Corn-soy blend	0.043-0.065	

The health risk assessment of the sampled maize grains is shown in Table 3. The Average Daily Intake (ADI), i.e. 0.1 mg/kg is the amount of GLY active ingredient that can be consumed daily over a lifetime without harm, expressed in mg/kg body weight of the consumer. It was indicated that the EDI of glyphosate ranges from 0 to 6.544×10^{-1} mg/kg.

Herbicide residue in food is usually monitored with reference to Maximum Residue Limits (MRLs) and Average Daily Intake (ADIs). Codex Alimentarius Commission and EU set 5 mg/kg as the Maximum Residual Limit (MRL) of glyphosate in maize in 2006 (FAO/WHO, 2019). Consumer exposure is of concern if the Estimated Dietary intake of a pesticide exceeds the ADI (Maigari et al., 2022). The ADI is the estimated amount of a chemical in food (mg/kg body weight/day) that can be ingested daily over a lifetime without appreciable health risk to the consumer (FAO/CODEX, 2011b). Based on the toxicological evaluation, the calculated EDIs for this study are all below the CODEX/FAO/WHO maximum permissible limit of 0.1 mg/kg for GLY, which suggested that consumers in the study area might not have health risks from consuming the maize samples (FAO/WHO, 2013; Fucic et al., 2021). The results of the Health Risk Index (HRI) also showed that the HRI >1 for KWM (0.6543), GWM (0.4301), and GYM (0.152) while the HI values obtained are less than 1 and according to EPA, of HRI >1 signifies associated risk; meaning the exposed population is likely to expose to adverse health risk while no associated health risk to the consumer within the population if HRI <1 (EPA, 2019, Maggi et al., 2021).

Table 3. Health Risk Assessment of maize grain sold in Abuja, Nigeria

Sample ID	Estimated Dietary Intake (EDI) mg/kg	Hazard Risk Index (HRI)	Health Risk Status	Sample ID	Estimated Dietary Intake (EDI) mg/kg	Hazard Risk Index (HI)	Health Risk Status
AYM	2.030×10^{-3}	2.030×10^{-2}	None	LWM	4.779×10^{-3}	4.779×10^{-2}	None
AWM	6.213×10^{-4}	6.213×10^{-3}	None	KYM	7.576×10^{-3}	7.576×10^{-2}	None
KWYM	7.067×10^{-3}	7.067×10^{-2}	None	KWM	7.045×10^{-3}	7.045×10^{-2}	None
KWWM	6.543×10^{-2}	6.543×10^{-1}	Yes	FMYM	4.134×10^{-4}	4.134×10^{-3}	None
SYM	2.221×10^{-3}	2.221×10^{-2}	None	FMWM	3.003×10^{-3}	3.003×10^{-2}	None
SWM	1.943×10^{-3}	1.943×10^{-2}	None	UWM	2.184×10^{-3}	2.184×10^{-2}	None
GWM	4.301×10^{-2}	4.301×10^{-1}	Yes	UYM	3.949×10^{-3}	3.949×10^{-2}	None
GYM	1.518×10^{-2}	1.518×10^{-1}	Yes	GWM	BDL	BDL	None
THSW	8.389×10^{-3}	8.389×10^{-2}	None	NWM	9.699×10^{-3}	9.699×10^{-2}	None
THYM	4.071×10^{-3}	4.071×10^{-2}	None	NYM	5.664×10^{-3}	5.664×10^{-2}	None
BWM	3.540×10^{-3}	3.540×10^{-2}	None	WYM	6.053×10^{-3}	6.053×10^{-2}	None
BYM	2.513×10^{-3}	2.513×10^{-2}	None	WWM	3.894×10^{-3}	3.894×10^{-2}	None
LYM	1.770×10^{-4}	1.770×10^{-3}	None	-	-	-	-

The result partially agrees with those of Bai et al. (2016), Oyeyiola et al. (2017), and Fedrick et al. (2018) on glyphosate on human health via food contamination both on dietary exposure to GLY herbicide where they obtained HRI <1, and agreed with Akande et al. (2021) who obtained HI greater than 1. There is concern for the population around Kwali and Gwagwalada markets based on toxicological studies and for others that have their HRI less than 1. However, some of the HRI values obtained were very close to the maximum value for the HRI of 1. The effect of the consumed food items on glyphosate residues may be additive or synergistic. This means that even pesticides that were detected at safe levels may eventually pose health hazards to humans due to combined and accumulated effects in the body (Maigari et al., 2022).

4. CONCLUSION

This study revealed glyphosate residue contamination of maize grains from two major markets, Nyanya (NYM) and Gwagwalada (GWM & GYM) to have violated CODEX MLR of 5 mg/kg i.e. 11.547%. The research has provided important information on GLY herbicide residue contamination in maize grains in the FCT, Abuja. Moreover, GWM, GYM, and KWWM have their HRI more than 1, which suggests that the consumer populations are at risk as regards consuming such maize grains. This indicated that most of the maize grains in the FCT Abuja markets are currently safe for human consumption. Farmers are advised to embrace Integrated Pest Management and avoid the application of GLY herbicides. Besides, governments at all levels and non-Governmental Organizations should embark on herbicide usage advocacy with a view to educate and orientate farmers and marketers toward correct application of herbicides such as GLY. Assessment of food products for pesticide residues should be carried out periodically to ensure sufficient data for regulatory bodies and policy makers in Nigeria.

Declaration of Interest
I declare that there is no conflict of interest.

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