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FOOD SCIENCE & TECHNOLOGY | RESEARCH ARTICLE

Determination of some herbicide residues in sweet potato

Yvonne A. Hormenoo¹, Jacob K. Agbenorhevi^{1*}, Seth O. Ekyem², Kwasi O. Bonsu², Vincent Torve³ and Bright R. Voegborlo³

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*Corresponding author: Jacob K. Agbenorhevi, Department of Food Science and Technology, Kwame Nkrumah University of Science and Technology, Kumasi, Ghana
E-mail: jkagbenorhevi@yahoo.com

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Abstract: The use of herbicides in weed control is on the increase but such chemical residues in crop plants mostly cause health risks. The objective of this work was to determine the level of some herbicide residues in sweet potato. The sweet potatoes were cultivated in randomized complete block design (RCBD) with four replications at the Crops Research Institute Agronomy fields, Kwadaso, Ghana. The different treatments made up of combinations of five pre-emergence herbicides (butachlor [50 g/L—3 L/Ha], imazethapyr [240 g/L—3 L/Ha], metolachlor [333 g/L—4 L/Ha], pendimethalin [500 g/L—3 L/Ha] and terbutryn [167 g/L—4 L/Ha]) and one (1) post-emergence herbicide (propaquizafop [100 g/L—1.2 L/Ha]) were applied. After harvest, samples were randomly selected and extracted using a modified QuEChERS extraction method followed by liquid chromatography-mass spectrometry (LC-MS) to determine the residual levels of the herbicides. The results showed that butachlor, imazethapyr, terbutryn and propaquizafop were not detected in their respective sweet potato samples analysed. However, pendimethalin and metolachlor residues were detected at concentrations of 0.0023 µg/g and 0.0029 µg/g, respectively. The findings suggest that the herbicide residue levels detected were

ABOUT THE AUTHOR

Yvonne A. Hormenoo (MSc) was a Graduate Student in the Department of Food Science and Technology, Kwame Nkrumah University of Science and Technology (KNUST), Kumasi, Ghana.

Jacob K. Agbenorhevi (PhD) is a Senior Lecturer in the Department of Food Science and Technology, KNUST, Kumasi, Ghana.

Seth O. Ekyem (MPhil) is a Research Scientist (Agronomist) in the Horticulture Division of the Council for Scientific and Industrial Research (CSIR)—Crops Research Institute, Kumasi, Ghana.

Kwasi O. Bonsu (MSc) is a Research Scientist (Plant Breeder) in the Horticulture Division of the CSIR—Crops Research Institute, Kumasi, Ghana.

Vincent Torve (BSc) is a Senior Technician in the Department of Chemistry, KNUST, Kumasi, Ghana.

Bright R. Voegborlo (PhD) is an Associate Professor in the Department of Chemistry, KNUST, Kumasi, Ghana.

PUBLIC INTEREST STATEMENT

The cultivation of sweet potato is increasingly encouraged in the developing countries as a major contributor to food and nutrition security. This is however faced with many challenges such as land preparation and weed control. Herbicides are substances which are applied with the intention of killing, controlling or preventing the excessive growth of weeds or unwanted plants. However, the use of herbicides also tends to create health hazards from the herbicide residues. Most of these herbicides are carcinogenic. In this study, some herbicide residues in sweet potato were determined following different treatments. Some herbicides (butachlor, imazethapyr, terbutryn and propaquizafop) were not detected in their respective sweet potato samples analyzed. However, those detected (pendimethalin and metolachlor residues) were lower than the maximum acceptable limit. The findings imply that the application of these herbicides at the recommended rates in sweet potato is safe from the residual point of view. However, further studies would be useful to confirm the present findings.

considerably lower than the maximum acceptable limit (0.05 µg/g) and thus the application of these pre-, and post-emergence herbicides at the recommended rates in sweet potato is safe from the residual point of view.

Subjects: Horticulture; Agriculture and Food; Food Chemistry; Food Analysis

Keywords: herbicide; residue; sweet potato

1. Introduction

Sweet potato (*Ipomoea batatas* L.) is a very vital food and industrial crop, cultivated globally and its production is increasing rapidly in many countries in the sub-Saharan Africa (Korada et al., 2010; Milind et al., 2015; Ofori et al., 2009; Ssali et al., 2021). Saharan-Africa has about 13.37 million hectares of land cultivated with sweet potato, thus making it the third most important root crop after cassava and yam (Amengor et al.; 2016). Sweet potato is widely cultivated in Africa partly due to its ease of cultivation, high ability to tolerate drought, low requirement for fertilizers, flexible planting and harvesting periods (Odebode et al., 2008). The white or yellow-fleshed sweet potato are the commonly grown varieties in most parts of Africa including Ghana (Kapinga et al., 2001). The orange-fleshed cultivars, in particular, have been reported to possess a high content of bio-available precursors of vitamin A (β-carotene) and its cultivation is therefore encouraged in the developing countries due to their prominence in combating vitamin A deficiency (Laurie et al., 2015). Furthermore, properties such as anti-carcinogenic, cardiovascular disease-preventing and its high nutrient content has resulted in its recognition as a health food (Njintang et al., 2016). However, sweet potato cultivation in Ghana is faced with many challenges such as land preparation and weed control (Ennin et al., 2007).

Weeds influence agricultural activities by competing with crops for available soil nutrients, air, water, sunlight and space, and also harbouring other invasive pests (Wyss & Müller-Schärer, 2001). Degras (2003) reported that 57% of food crops in some parts of Africa are lost due to the presence of weeds, hence the need to effectively apply herbicides.

Herbicides are described as a subtype of pesticides which are applied with the intention of killing, controlling or preventing the excessive growth of weeds or unwanted plants. The control of weeds with herbicides in modern agriculture has become indispensable due to the acute shortage of farm labourers (Ponnusamy et al., 2015; Sanyal & Shrestha, 2008). The application of herbicides has indeed become the main strategy for weed control for both agricultural and non-agricultural purposes in Ghana. It has been reported that herbicides make up 44% of pesticides used in Ghana (Ntow et al., 2006). However, excessive use of herbicides may result in phytotoxicity to food crops, residual effects on susceptible crops and adverse effects on non-target organisms. It may ultimately cause severe health hazards to humans and animals due to the accumulation of residues in the crops, soil, surface and groundwater (Das & Mondal, 2014; Ponnusamy et al., 2015).

The use of herbicides in some parts of Africa is generally on the increase (Grabowski & Jayne, 2016). It has also been estimated that about 25% of pesticides produced globally are used by farmers in developing countries and the population suffers deaths from pesticide poisoning (WHO, 2008). Herbicides are produced under very strict regulations as a means of reducing their negative impact on human health and on the environment. However, reports indicate that, when these herbicides are applied, only about 1% is effective whereas the remaining 99% exist as residues in the surroundings thus posing serious threats to human health, the environment, wildlife and other non-target organisms (Eskenzi et al., 2008; Zhang et al., 2011). Residue of herbicides found in crops is inevitable even if applied as instructed by the manufacturers; this has therefore attracted attention from the sweet potato value chain as this could be a great menace to human health and the environment (Damalas & Eleftherohorinos, 2011; Darko & Acquah, 2007). Some farmers, however, use these chemicals indiscriminately since they are more concerned about minimizing the destructive effects of weeds on

their crops and obtaining optimum yield. Some farmers apply herbicides to the target weeds without paying due attention to instructions stated on the labels with respect to the recommended rates of application and the right ways of disposing off excess herbicides after application. This ultimately leads to the presence of more herbicide residues (Adomako & Akyeampong, 2016).

A number of research work have been carried out on pesticide residues especially in fruits and vegetables. However, very little has been done with regard to herbicide residues in roots and tubers, especially sweet potato. There is therefore the need to ascertain the levels of herbicide residues in sweet potatoes and also adopt appropriate measures for controlling the presence of these residues so as to reduce the health risks posed on consumers. The objective of this study, therefore, was to determine the levels of selected herbicide residues in sweet potato roots.

2. Materials and methods

2.1. Experimental layout, field preparation and treatments

The study was conducted at the Crops Research Institute Agronomy fields (with agrometeorological station number 65,442) at Kwadaso-Kumasi in the Ashanti Region of Ghana. The agrometeorological station is on latitude 06° 43' N, longitude 01° 36' W and altitude of 286.3 m. The Global Positioning System (GPS) of the agronomy field is N 06° 40' 755"; W 01° 40' 501". The soil type at the agronomy field was sandy loam. The agronomy field had mean annual rainfall of 3.64 mm, mean annual wind run of 116.1 km/h and mean annual sun shine 4.35 h. The mean annual relative humidity at 0900 hours and 1500 hours was 81.0% and 56.7%, respectively, whereas the annual atmospheric temperature at 0900 hours and 1500 hours was 27.7°C and 27.9°C, respectively. Mean annual soil temperature was 27.6°C (at 5 cm), 27.8°C (at 10 cm), 28.5°C (at 20 cm) and 29.2°C (at 30 cm) at 0900 hours whereas at 1500 hours, the annual soil temperature was 31.4°C (at 5 cm), 27.8°C (at 10 cm), 29.1°C (at 20 cm) and 29.2°C (at 30 cm).

Four (4) treatments were involved in the weed management strategies investigated. Table 1 shows the treatment combinations employed.

The experiment was laid out in a Randomized Complete Block Design (RCBD) with four replications. Four (4) ridges per plot were spaced 60 cm apart. Each ridge measured 6 m long and was planted to 12 stands at 50 cm apart. Plots measured 6 m × 2.4 m were laid out with 1.2 m border between plots.

Field preparation involved ploughing, harrowing to fine tilt and ridging. Pre-emergence herbicides, Activus 500 EC [Pendimethalin (500 g/L—3 L/Ha)]; Terbulor 500 EC [Metolachlor (333 g/L) +

Table 1. Experimental treatments

Treatment	Herbicides
Treatment 1	<i>Pre-emergence herbicide</i> : Activus 500 EC [Pendimethalin (500 g/L—3 L/Ha)] + <i>Post-emergence herbicide</i> : Agil 100 EC [Propaquizafop (100 g/L—2 L/Ha)]
Treatment 2	<i>Pre-emergence herbicide</i> : Terbulor 500 EC [Metolachlor (333 g/L) + Terbutryn (167 g/L)—(4 L/Ha)] + <i>Post-emergence herbicide</i> : Agil 100 EC [Propaquizafop (100 g/L—1.2 L/Ha)]
Treatment 3	<i>Pre-emergence herbicide</i> : Butaplus 50 EC [Butachlor (50 g/L—3 L/Ha)] + <i>Post-Emergence herbicide</i> : Agil 100 EC [Propaquizafop (100 g/L—1.2 L/Ha)]
Treatment 4	<i>Pre-emergence herbicide</i> : Vezir 240 SL (Imazethapyr 240 g/L—3 L/Ha) + <i>Post-emergence herbicide</i> : Agil 100 EC [Propaquizafop (100 g/L—1.2 L/Ha)]

Terbutryn (167 g/L)—(4 L/Ha)]; Butaplus 50 EC [Butachlor (50 g/L—3 L/Ha)] and Vezir 240 SL (Imazethapyr 240 g/L—3 L/Ha), were first applied to their respective plots. One week after the application of pre-emergence herbicides, healthy vine cuttings were planted at 30 cm intra row spacing. Post-emergence herbicide, Agil 100 EC [Propaquizafop (100 g/L—1.2 L/Ha)], was applied 4 weeks after planting. At this time, weed seeds that escaped the pre-emergence herbicides or were dormant or have newly been introduced to the field would have germinated, emerged and established. At maturity (16 weeks after planting), sweet potato roots were harvested from the two inner rows of each plot. Twelve roots from each plot were randomly picked and bulked on treatment lines. Twelve out of the 48 roots per treatment were again randomly picked, bagged, labelled and stored in a refrigerator for herbicide residue analyses.

2.2. Determination of herbicide residues

Butachlor, Imazethapyr, Metolachlor, Pendimethalin, Propaquizafop and Terbutryn herbicide reference standards (purity >94%) were obtained from Dr Ehrenstorfer GmbH (Augsburg, Germany). Acetonitrile (HPLC grade) and acetic acid (analytical grade) were obtained all from VWR Prolabo, France. All other chemicals used were analytical grade reagents.

The sweet potato samples were placed in well-labelled sample bags and transported on ice to the laboratory for onward processing and subsequent herbicide residue analysis. The samples were washed unpeeled and cut into smaller pieces and mixed together. About 100 g of sweet potatoes per treatment was homogenized in a Binatone blender at high speed with

A modified approach of the QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) methodology (Kolberg et al., 2010) was employed to extract the herbicides from the sweet potato samples. Respective weights of 10 g per treatment of the homogenized slurry were taken in 50 mL centrifuge tubes. Ten milliliters of acetonitrile, containing 1% (v/v) of acetic acid, was added to the sample after which the mixture was hand-shaken for a minute. A 3 g of anhydrous MgSO_4 was then added and the tube was immediately hand-shaken for about 20 s. Subsequently, 1.7 g and 1 g of sodium acetate and sodium citrate, respectively, were added and the tube was hand-shaken for another minute to provide well-defined phase separation after 8 min of centrifugation at 4,000 rpm. A 4 mL aliquot of the upper layer was then transferred into a centrifuge tube (15 mL) which contained 0.6 g of anhydrous MgSO_4 and 0.5 g of PSA and adsorbent C18. The tube was closed vigorously hand-shaken for a minute after which it was again centrifuged at 4000 rpm for 8 min. An aliquot of the supernatant was finally transferred into an autosampler vial prior to its injection into the LC-MS system.

Equipment parameters: Agilent 1200 series HPLC coupled to an API 4000 Qtrap mass spectrometer equipped with an electrospray ionization interface. The HPLC separation was carried out using an Atlantis dC18 column (100 mm x 2.1 mm x 5 μm). The mobile phase used was made up of (A) methanol: water (20:80 v/v with 5 mM ammonium formate and 0.15% formic acid) and (B) methanol: water (90:10 v/v with 5 mM ammonium formate); with gradient 0–0.5 min 85% A, 0.5–7 min 85–2% A, 7–15 min 2% A, 15–16 min 2–85% A and 16–20 min 85% A. The mobile-phase flow rate was 0.3 mL min^{-1} and column temperature was maintained at 35°C. The source parameters included nebulizer gas 40 psi; heater gas 60 psi; ion source temperature 550°C; ion spray voltage 5500 V. An aliquot of 10 μL was injected with autosampler. The evaluation of the calibration curves and linearity were carried out based on injections of the standard solutions prepared at the concentrations of 2.0, 5.0, 10.0, 20.0 and 30.0 ng mL^{-1} . The linearity (R^2 value) obtained was ~0.999 for all the standard herbicide compounds analyzed.

For each batch, a matrix blank was also analyzed. Extracts were kept frozen until quantitation was achieved.

3. Results and discussion

The results obtained after the herbicide residue analysis are presented in [Table 2](#).

Table 2. Herbicide residues in sweet potato samples

Treatments	Herbicide residues (µg/g)					
	Butachlor	Imazethapyr	Metolachlor	Pendimethalin	Terbutryn	Propaquizafop
1	ND	ND	ND	0.0023 ± 0.00	ND	ND
2	ND	ND	0.0029 ± 0.00	ND	ND	ND
3	ND	ND	ND	ND	ND	ND
4	ND	ND	ND	ND	ND	ND

ND – Not Detected; Limit of Detection (LOD) = 1 ppb (0.001 µg/g)

The LC-MS results indicated the detection of pre-emergence herbicides; pendimethalin and metolachlor residues in treatments 1 and 2 samples at concentrations 0.0023 $\mu\text{g/g}$ and 0.0029 $\mu\text{g/g}$, respectively (Table 2). All other treatments showed no herbicide residues, i.e. butachlor, imazethapyr, terbutryn and propaquizafop (which was applied post-emergence throughout all the treatments). It is possible that these herbicide compounds got degraded to undetectable levels by the time the sweet potatoes were harvested. According to Howell (2011), biotic and abiotic factors make up the various mechanisms involved in the fate of herbicides in the environment. The biotic factors are basically the interactions with living organisms which include uptake by plants and degradation by microorganisms, whereas the abiotic factors include volatilization and photochemical degradation. Adomako and Akyeampong (2016) further explained that after application, the fate of herbicides to a large extent depends on the ability of the soil microorganisms to cause the degradation of the herbicides and this is ideally through the complete mineralization of the parent compounds into carbon dioxide (CO_2) and also the transfer of the chemicals through some other physical processes. The concentrations of pendimethalin and metolachlor applied were 500 g/L (3 L/Ha) and 333 g/L (4 L/Ha) respectively. Imazethapyr, terbutryn, butachlor and propaquizafop concentrations were also 240 g/L (3 L/Ha), 167 g/L (4 L/Ha), 50 g/L (3 L/Ha) and 100 g/L (1.2 L/Ha), respectively. This indicates that the concentrations of pendimethalin and metolachlor applied were higher compared to the other herbicides used and this may have contributed to the presence of their residues in the sweet potato samples post-harvest. This could also signify that the quantities applied with respect to the remaining four herbicides were just right to avoid the persistence of their residues in the sweet potatoes. Furthermore, this phenomenon may also be attributed to their short-duration nature of persistence in plants.

According to the EU pesticide database, the maximum residue limits of both pendimethalin and metolachlor in sweet potatoes are 0.05 mg/kg or 0.05 $\mu\text{g/g}$ (European Commission, 2016). It is evident that the residues detected in this study are lower and this therefore signifies that the risk associated with the dietary exposure of these herbicides in sweet potatoes is considered safe to humans and may not pose no adverse health effects as far as food safety is concerned.

Saha et al. (2015) also cultivated peanut samples (*Arachis hypogaea* L.) in experimental fields and reported that at harvest time, no herbicide residues were detected in peanut kernel for pendimethalin and imazethapyr after treatments. This therefore indicated that the residue levels of the selected herbicides were below the maximum residue limits prescribed by European Union as well as other international organizations. A similar study carried out by Sireesha et al. (2011) revealed that the detected residues of pendimethalin in radish tubers were below the maximum residue limit at harvest. Alternatively, Sondhia and Dubey (2006) reported the detection of 0.007 $\mu\text{g/g}$ residues of pendimethalin in green onion at 1.0 kg/ha application rate. Comparatively, the amount of pendimethalin applied in this study was twice that used in the sweet potato research and that possibly explains why the concentration of residues detected was also higher. Furthermore, Sondhia (2013) conducted a field study and analysed the residual effects of pendimethalin applied as pre-emergence at 1.0 kg/ha in cauliflower, radishes and tomato. At harvest, 0.001 $\mu\text{g/g}$, 0.014 $\mu\text{g/g}$ and 0.008 $\mu\text{g/g}$ residues of pendimethalin were detected in cauliflower, radishes and tomato, respectively. In another research by Sondhia (2008), which involved the application of 100 g/ha imazethapyr, the residues detected in soybean grains, straw and soil were 0.102 $\mu\text{g/g}$, 0.301 $\mu\text{g/g}$ and 0.008 $\mu\text{g/g}$, respectively. Despite the fact that the application rate in this research was lower, higher concentrations were detected. This could probably mean that the rate of degradation of the herbicide was low hence the persistence of the residues in the crops and soil. The concentration of the residues in the soil was the least, in this case, and this could be attributed to the degradation by microorganisms in the soil. A recent study by Poonia et al. (2017) also reported that imazethapyr residues in soil were detected below the limit of quantification; however, in the plants, residues persisted to the levels of 0.015 $\mu\text{g/g}$ at harvest of the groundnut samples. The concentration of the residues detected was far below the tolerance limits approved by European Union standards as well as the Indian Food Safety and Standards Authority and hence, risks associated with dietary exposure of these herbicides were considered safe for human

consumption. The authors also reported that the short-duration nature of persistence of the herbicides in soil and plants also confirmed that the herbicides were safe for the environment as well as for rotational crops.

4. Conclusion

Butachlor, imazethapyr, terbutryn and propaquizafop were not detected in their respective sweet potato samples analyzed. However, pendimethalin and metolachlor residues were detected at concentrations of 0.0023 µg/g and 0.0029 µg/g, respectively. The detected herbicide levels were lower than the maximum acceptable limit (0.05 µg/g) implying the sweet potato samples were considerably safe for consumption from the residual point of view. Further studies would be useful to ascertain the effect of herbicide concentration as well as different geographical locations or soil environment on the herbicide residue levels in sweet potato.

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Author details

Yvonne A. Hormenoo¹
Jacob K. Agbenorhevi¹
E-mail: jkagbenorhevi@yahoo.com
ORCID ID: <http://orcid.org/0000-0002-8516-7656>
Seth O. Ekyem²
Kwasi O. Bonsu²
Vincent Torve³
Bright R. Voegborlo³
¹ Department of Food Science and Technology, Kwame Nkrumah University of Science and Technology, Kumasi, Ghana.
² Council for Scientific and Industrial Research, Crops Research Institute, Kumasi, Ghana.
³ Department of Chemistry, Kwame Nkrumah University of Science and Technology, Kumasi, Ghana.

Competing interests

The authors declare no competing interests.

Data availability

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

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