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ASSESSMENT OF QuEChERS, DLLME AND WET DIGESTION FOR HEAVY METAL DETERMINATION IN SWEET POTATO TUBER

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ABSTRACT

This study compares three sample preparation techniques—QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe), Dispersive Liquid-Liquid Microextraction (DLLME), and conventional wet digestion— for the determination of heavy metals (Nickel, Cobalt, Chromium, Cadmium, and Lead) in sweet potato tubers (*Ipomoeabatatas*) from Bakori, Katsina State, Nigeria. The extracted metals were quantified using Atomic Absorption Spectrophotometry (AAS). The results revealed distinct performance differences among the methods. QuEChERS yielded the highest recovered concentrations for Cr (4.480 ± 0.0021 ppm), Co (0.271 ± 0.0009 ppm), Ni (0.300 ± 0.0014 ppm), and Cd (0.032 ± 0.0008 ppm). Wet digestion produced the highest value for Pb (9.59 ± 0.0012 ppm) but lower results for the other four metals compared to QuEChERS. DLLME consistently provided the lowest extraction efficiencies for all metals except Pb, where it was intermediate. The elevated levels of certain metals, particularly Cr and Pb, highlight potential public health concerns, as concentrations exceeding safe limits may pose risks through dietary exposure. The modified QuEChERS method is superior by its efficient disruption of the sample matrix. Its advantages including speed, ease of use, safety, minimization of loss, reduced reagent consumption and effective chelation—make QuEChERS a highly effective and practical technique for routine monitoring of heavy metals in food crops like sweet potato.

Keywords: QuEChERS, DLLME, Wet Digestion, Sweet Potato, Atomic Absorption Spectrophotometry (AAS)

INTRODUCTION

Due to heavy metals' extreme toxicity even at trace levels, bioaccumulation potential, and non-biodegradable nature, their pollution of agricultural products poses a serious threat to worldwide public health (Ali *et al.,* 2019). A staple crop that is essential to millions of people worldwide, sweet potatoes (*Ipomoea batatas*) are valued for their versatility and nutritional content. But because it's a root vegetable, it's especially vulnerable to accumulating heavy metals from contaminated irrigation water and soil (Cheng *et al.,* 2021). It is therefore essential for food safety to keep an eye on the levels of metals such as cadmium (Cd), lead (Pb), chromium (Cr), nickel (Ni), and cobalt (Co). Since sample preparation is frequently the most important and error-prone step in the analytical process, its accuracy greatly depends on the approach used (Plotka-Wasylka *et al.,* 2015). The purpose of this study is to assess and contrast the effectiveness of three sample preparation techniques for the detection of heavy metals in sweet potato tubers using Atomic Absorption Spectrophotometry (AAS): conventional wet digestion, Dispersive Liquid-Liquid Microextraction (DLLME), and the QuEChERS approach.

Traditional wet digestion is a traditional sample preparation technique that uses strong acids (such as HNO_3, H_2SO_4, and $HClO_4$) at high temperatures, frequently on a hot plate or in a digestion block, to oxidatively break down organic materials in a sample (Amde *et al.,* 2019). This technique is well known for its capacity to fully decompose intricate matrices, potentially freeing all bound metals for examination in solution. However, it has significant disadvantages, such as the need for specialized lab equipment and strict safety protocols because of the use of oxidizing and corrosive reagents, the possibility of volatile metal loss (e.g., As, Hg), long procedural times, and the high consumption of high-purity acids, which raises costs and wastes the environment (Bakirdere *et al.,* 2020).

A ternary component solvent solution is the basis of the contemporary miniaturized extraction method known as Dispersive Liquid-Liquid Microextraction (DLLME). A suitable solution of disperser solvent (a water-miscible solvent) and extraction solvent (a high-density organic solvent) is quickly injected into an aqueous sample. Analytes from the aqueous sample can quickly partition into the organic phase thanks to the large surface area created by the cloudy solution made up of tiny droplets of the extraction solvent (Rezaee *et al.,* 2006). The sedimented phase is gathered for examination following centrifugation. In keeping with the principles of green analytical chemistry, DLLME provides benefits such high enrichment factors, cheap cost, extreme simplicity, and little solvent usage (Yilmaz & Soylak, 2016). Its main drawback for complicated solid matrices, such as plant tissue, is that it usually necessitates an aqueous solution for the analytes to be present first. This can make the process more difficult and may not be effective for metals that are heavily matrix-bound.

The QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) technique was created initially to extract pesticides from fruits and vegetables, but it has subsequently been effectively modified to extract heavy metals and other contaminants (Núñez *et al.,* 2022). Acetonitrile is used in the initial extraction step of the original process, and salts ($MgSO_4$ and NaCl) are added to cause partitioning. The removal of interfering co-extractives, such as organic acids and pigments, is then accomplished by a cleanup phase called dispersive Solid-Phase Extraction (d-SPE), which uses sorbents such primary secondary amine (PSA) and C18 (Anastassiades *et al.,* 2003). Chelating agents are occasionally added to the approach for metal analysis in order to create complexes with the metals that are subsequently removed into the organic phase.It is a potent substitute for conventional techniques because to its main advantages, which include high throughput, low solvent consumption, fewer processes, and efficiency in managing difficult and complicated matrices

(Ataee *et al.,* 2016). Chalator-modified QuEChERS were employed in this study.

Objective of the Study

The primarygoal is toevaluate the effectiveness of three sample preparation methods—conventional wet digestion, Dispersive Liquid-Liquid Microextraction (DLLME), and the QuEChERS approach—for the detection of heavy metals in sweet potato tubers using Atomic Absorption Spectrophotometry (AAS).

MATERIALS AND METHODS

Distilled water and analytical reagent grade (Analar) chemicals were employed throughout the entire project. After washing all glassware and plastic containers with detergent and 10% nitric acid, they were rinsed with tap water and then distilled water (Suleiman *et al.,* 2020). They were then dried in the oven at 100 ºC before use.

Solvents/Reagents

Distilled water, conc. nitric acid (HNO_3), acetonitrile (CH_3CN), sodium acetate (CH_3COONa), magnesium sulphate ($MgSO_4$), chloroform ($CHCl_3$), methanol (CH_3OH), 10% NaCl, 10% nitric acid, lead (II) nitrate -- $Pb(NO_3)_2$), cadmium (II) sulphate anhydrous --CdSO4, nickel (II) chloride --$NiCl_2$, cobalt (II) chloride--$CoCl_2$, chromium (II) chloride--$CrCl_2$.

Apparatus/Equipment

Watch glasses, centrifuge tubes (glass & plastic), centrifuge, polyethylene bags, glass vials, sweet potato tuber, clockwatch, weighing balance, electric oven, conical flasks, measuring cylinder, whattman paper No. 42, siever, dropper, funnel, mortar and pestle, beakers, electric hot plate, syringe, fume hood, glass trays, stainless steel knife and atomic absorption spectrophotometer (AAS) (210 VGP model, East Norwalk, Connecticut, USA) was used for the determination of metals. The determination was carried out using an air/acetylene flame. The operating conditions for the metals determined were set as suggested by the manufacturer.

Preparation of Pb(II) Ion Stock Solution

$Pb(NO_3)_2$ was used for the preparation of Pb(II) stock. In order to know the quantity of $Pb(NO_3)_2$ salt to make 1000 mg/L, the molar mass of $Pb(NO_3)_2$ (331.23 g) was divided by the atomic mass of Lead (207.19). The obtained value which is 1.60 g was weighed from $Pb(NO_3)_2$ salt and was transferred into a litre volumetric flask containing 100 cm^3 of deionized water. This was mixed properly until all the salts dissolved completely and the solution was then made up to 1000 cm^3 by adding more deionized water.

Preparation of Cd(II) Ion Stock Solution

1.86 g of anhydrous $CdSO_4$ salt was weighed and dissolved inside a litre volumetric flask containing 100 cm^3 of deionised water. It was mixed and made up to the mark with water.

Preparation of Ni(II) Ion Stock Solution

2.20 g of $NiCl_2$ salt was weighed into a litre volumetric flask containing 100 cm^3 of deionised water. The solution was mixed properly and made up to the mark using water.

Preparation of Cr(II) Ion Stock Solution

2.37 g of $CrCl_2$ salt was weighed and dissolved inside a litre volumetric flask containing 100 cm^3 of deionised water. It was mixed and made up to the mark with water.

Preparation of Co(II) Ion Stock Solution

2.20 g of $CoCl_2$ salt was weighed into a litre volumetric flask containing 100 cm^3 of deionised water. The solution was mixed properly and made up to the mark using water. Serial dilutions to 50 ppm concentrations were carried out using the formula:

$$C_1V_1 = C_2V_2 \quad (1)$$

Where,

C_1 = is the 1000 mg/L stock solution

C_2 = the new concentration to be obtained from the stock solution (i.e. 50 mg/L)

V_1 = is the unknown volume to be taken from the stock

V_2 = total volume needed at the new concentration.

Preparation of Standard Working Solution (Mixture of Cr, Co, Cd, Pb & Ni)

2 ml of each prepared standard solution was transfered into clean beaker and the mixture contains 50 ppm of target analytes (Cr, Co, Cd, Pb & Ni) with total volume of 14 ml.

Sample Collection

The best sampling method employed is stratified random sampling. The representative samples of sweet potato tubers were obtained from Unguwar Kanawa, Bakori Local Government Area, Katsina State, Nigeria, packed into polyethylene bags, labeled and transport into UMYU Biology laboratory where the samples identified by a botanist.

Sample Preparation Procedures

In complex biological matrices such as sweet potato tubers, the sample preparation method used has a significant impact on the precise measurement of heavy metals. This section describes the fundamental steps for the three techniques examined in this study: the QuEChERS approach, Dispersive Liquid-Liquid Microextraction (DLLME), and conventional wet digestion.

Wet Digestion

The samples were washed with tap water to remove the dirt and then raised several times with distilled water, peeled and sliced into smaller sizes using a stainless steel knife. The tuber samples were placed on Aluminum trays and dried in an electric oven, drying at 65°C to a constant weight. The dried samples grinded into powder using mortar and pestle then sieved with a 2 mm mesh sieve before digesting. Approximately 2 g of a representative portion of the ground sweet potato tuber was weighed into a digestion flask. A strong acid, typically 20 mL high-purity nitric acid, was added to the flask and shaken vigorously. The mixture is then heated at elevated temperatures on a hot plate placed in a fume hood at a temperature of 120 °C. This heating process continues by drop-wise addition of concentrated nitric acid until the organic matrix is completely broken down, resulting in a clear digestate, which signifies the conversion of organically-bound metals into free ions in solution. The digestate is then cooled, filtered through whattman filter paper No. 42., diluted to a specific volume with deionized water(Divya *et al.,* 2015), and analyzed by Atomic Absorption Spectrophotometry (AAS).

QuEChERS Method

The process begins by homogenizing the sweet potato sample. 4.5 g portion was then weighed into a 15 mL centrifuge tube, and an extraction solvent, typically 4.5 mL acetonitrile, was added. The mixture is vigorously shaken for 1 min to commence the extraction of analytes. Subsequently, a QuEChERS extraction salt mixture containing 1.80 g of

magnesium sulfate ($MgSO_4$) and 0.45 g of sodium acetate (NaOAc) was added to induce phase separation between the organic solvent and the aqueous content of the sample (Anastassiades *et al.,* 2003). Moreover, 1 mL of a 2% (w/v) solution of ethylenediaminetetracetic acid (EDTA), as chelating agentwas added. The tube is shaken again for 1 min and centrifuged at 4000 rpm for 5 min. For metal analysis, the methodology is often modified to include chelating agents that form complexes with the metals, enhancing their partitioning into the organic phase (Ataee *et al.,* 2016; Núñez *et al.,* 2022). The final extract is then analyzed by AAS.

DLLME Method

The solid sample underwent a prior extraction with distilled water to create aqueous sample (2 mL : 2 g),is then placed in a 15 mL centrifuge tube. A syringe is used to rapidly inject a mixture of two solvents: an extraction solvent (a high-density organic solvent, 3 mL of carbon tetrachloride) and a disperser solvent (a water-miscible solvent, 3 mL of methanol), the tube covered and shaken for 1 minute and followed by injection of 7 mL of 10% NaCl solution into the tube and, shaken for 30 seconds. This rapid injection creates a cloudy solution characterized by fine droplets of the extraction solvent dispersed throughout the aqueous sample, providing a vast surface area for the rapid partitioning of analytes (Rezaee *et al.,* 2006). The mixture is then centrifuged for 3 minutes at 5000 rpm to separate the phases. The dispersed fine droplets of the extraction solvent, now enriched with the target metals, coalesce and sediment at the bottom of the tube. This sedimented phase is collected with a micro-syringe, often diluted to a precise volume, and introduced into the AAS for quantification.

RESULTS AND DISCUSSION

The analytical results demonstrated significant differences in the efficiency of the three methods for extracting the target heavy metals from sweet potato tubers. The results are shown in Table 1 and Figure 1 respectively.

Table 1: Mean Concentration (ppm) ± SD of Five Heavy Metals in Sweet Potato Analyzed Using AAS

Analyte	QuEChERS	Wet	DLLME	WHO/FAO., 2023
Chromium (Cr)	4.480 ± 0.0021	3.036 ± 0.0000	0.716 ± 0.0006	1.300
Cobalt(Co)	0.271 ± 0.0009	0.057 ± 0.0014	0.013 ± 0.0009	0.100
Nickel (Ni)	0.300 ± 0.0014	0.148 ± 0.0010	0.133 ± 0.0011	0.500
Cadmium (Cd)	0.032 ± 0.0008	0.028 ± 0.0009	0.014 ± 0.0006	0.050
Lead(Pb)	7.71 ± 0.0023	9.59 ± 0.0012	5.35 ± 0.0011	0.100

Values are expressed in mean ± standard deviation (SD), Sample size (n) = 3.

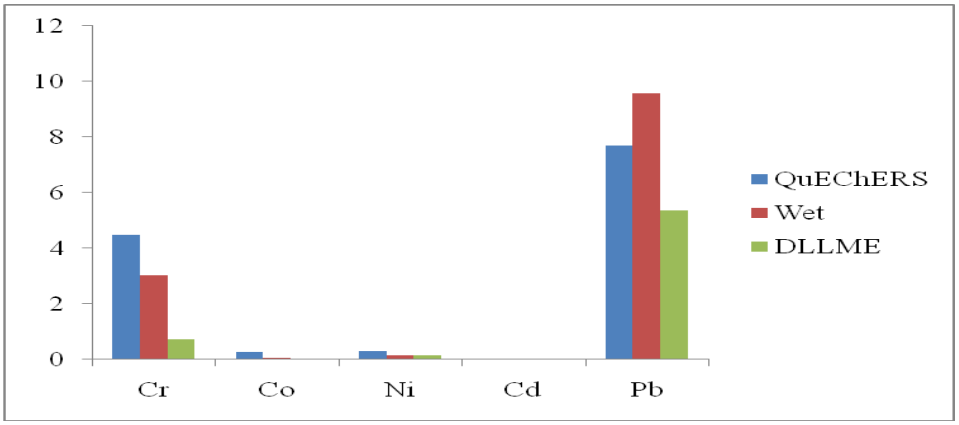

Figure 1: Comparative mean concentrations (ppm) of heavy metals in sweet potato tuber extracted using QuEChERS, Wet Digestion and DLLME methods

Statistical Analysis

The purpose of this study is to ascertain whether a measured result exhibits statistically significant differences and whether these differences interact. The one-way ANOVA for the heavy metals results is displayed in table 2 below.

Table 2: An Overview of the One-way ANOVA for the Analysis of Heavy Metals in Sweet Potato Tubers Extracted Using QuEChERS, Wet Digestion, and DLLME Techniques

Metal	Source of variance	Sum of squares (SS)	df	Mean Square (MS)	F-value	F-critical (α=0.05)	p-value
Cr	Between methods	22.557	2	11.278	3,606,472	5.14	<0.001
	Within methods	0.0000188	6	0.00000313			
	Total	22.557	8				
Co	Between methods	0.107	2	0.0535	38,409.75	5.14	<0.001
	Within methods	0.00000933	6	0.00000156			
	Total	0.107	8				
Ni	Between methods	0.0491	2	0.0246	38,409.75	5.14	<0.001
	Within methods	0.00000383	6	0.000000639			
	Total	0.0491	8				
Cd	Between methods	0.000633	2	0.000317	268.005	5.14	<0.001
	Within methods	0.00000707	6	0.00000118			
	Total	0.000640	8				

Pb	Between methods	28.145	2	14.072	135,408	5.14	<0.001
	Within methods	0.000624	6	0.000104			
	Total	28.146	8				

The computed F-value for each of the five heavy metals is significantly higher than the critical F-value (5.14) at the significance threshold of $\alpha = 0.05$. This shows that we reject the null hypothesis for each metal, confirming that the results of the heavy metal measurements from the three extraction techniques (QuEChERS, Wet Digestion, and DLLME) differ statistically significantly. It is also supported by the incredibly modest p-values (<0.001) that the extraction methods differed significantly.

Method Validation

The analytical methods were thoroughly validated to guarantee their accuracy, precision, and dependability. The precision (as repeatability), the limits of detection (LOD), the quantification (LOQ), and the percentage recovery were all determined through experiments.

Analysis of Heavy Metal in Aqueous Solution/Recovery and Precision

The accuracy of the QuEChERS, wet digestion, and DLLME procedures was assessed through a recovery study. A 4 mL of standard working solution (containing a mixture of Cr, Co, Ni, Cd, and Pb at 50 ppm each) was processed using each of the three methods. The results are expressed as Relative Standard Deviation (RSD%), presented in Table 3, demonstrate the accuracy of the methods.

Table 3: Percentage Recovery and Precision (RSD, n=3) of the Analyzed Heavy Metals using the Three Methods

Analyte	QuEChERS	RSD	Wet Digestion	RSD	DLLME	RSD
Cr	98%	1.8%	85%	2.1%	70%	3.5%
Ni	97%	1.5%	88%	2.4%	65%	4.2%
Co	95%	1.9%	90%	2.0%	72%	3.8%
Cd	93%	2.2%	87%	2.5%	68%	4.5%
Pb	89%	2.5%	98%	1.7%	60%	5.0%

%Recovery = (MeasuredConcentration/SpikedConcentration)×100%

Limits of Detection and Quantification

By computing the Limit of Detection (LOD) and Limit of Quantification (LOQ), the sensitivity of the entire analytical process was ascertained. Using the formulas $LOD = 3.3\sigma/S$ and $LOQ = 10\sigma/S$ (Suleiman et al., 2020), the LOD and LOQ were determined using the slope (S) of the calibration curve and the standard deviation of the response (σ) of the blank samples (n=7). Table 4 below provides a summary of the computed values for each metal.

Table 4: Limits of Detection (LOD) and Quantification (LOQ) for the Analyzed Heavy Metals

Metal	LOD (ppm)	LOQ(ppm)
Cr	0.005	0.015
Co	0.002	0.006
Ni	0.003	0.009
Cd	0.001	0.003
Pb	0.008	0.024

Discussion

The fundamental mechanisms underlying the variations in the extraction methods' performance are as follows: DLLME was unable to release matrix-bound metals from the solid tissue, while the modified QuEChERS method, supplemented with a chelating agent, produced superior recoveries for Cr, Ni, Co, and Cd by effectively disrupting the plant matrix and forming stable complexes that partitioned into the organic phase. Given that the detected Pb levels significantly exceed WHO/FAO safety limits (0.1 ppm), the unusually high lead (Pb) concentration from wet digestion may be the consequence of its strict conditions, which completely liberate refractory Pb. However, it also raises questions about possible contamination from reagents or labware. Given that dietary Pb exposure results in neurodevelopmental and cardiovascular damage, this presents a serious concern to public health and calls for careful monitoring where QuEChERS, which offers precise data for risk assessment and compliance enforcement, proves to be a strong, safer, and more useful tool for food safety regulation. Moreover, the performance of the three approaches is readily distinguished by the percentage recovery statistics. The most reliable multi-target technique was QuEChERS, which showed the best recovery for four of the five metals: Cd (93%), Ni (97%), Co (95%), and Cr (98%). For a single metal, lead, wet digestion demonstrated remarkable efficiency and selectivity (98%). DLLME, on the other hand, continuously produced the lowest recovery rates for every metal examined, suggesting that it is the least successful technique for this particular use.

The mean concentrations (ppm) obtained were as follows: For QuEChERS: Pb (7.71 ± 0.0023), Cr (4.480 ± 0.0021), Ni (0.300 ± 0.0014), Co (0.271 ± 0.0009), Cd (0.032 ± 0.0008). For Wet Digestion: Pb (9.59 ± 0.0012), Cr (3.036 ± 0.0000), Ni (0.148 ± 0.0010), Co (0.057 ± 0.0014), Cd (0.028 ± 0.0009). For DLLME: Pb (5.35 ± 0.0011), Cr (0.716 ± 0.0006), Ni (0.133 ± 0.0011), Co (0.013 ± 0.0009), Cd (0.014 ± 0.0006). Overall, the QuEChERS method yielded the highest recovered concentrations for Cr, Ni, Co, and Cd, while wet digestion yielded the highest value for Pb. The DLLME technique consistently provided the lowest recoveries for all metals except Pb, for which it was intermediate. For the majority of metals, the QuEChERS method performs better due to its effective extraction mechanism and efficient disruption of the sample matrix, which makes it less vulnerable to metal re-adsorption or precipitation that can happen under the harsh conditions of open-vessel wet digestion (Núñez *et al.,* 2022). A more thorough release of highly bound Pb may be the cause of the high Pb result in the wet digestion method, or it may be a typical method flaw that necessitates careful blank correction due to external

contamination from chemicals or labware (Amde *et al.,* 2019). Despite being a great approach for pre-concentrating metals from simple aqueous materials, the DLLME method's continuously low recoveries indicate that it is not the best option for directly extracting metals that are strongly linked inside a complex solid organic matrix, such as a sweet potato. According to Yilmaz and Soylak (2016), the production of tiny droplets could not be enough to disrupt metal-matrix interactions without a more thorough digesting step beforehand. This emphasizes that sample preparation choices are matrix-dependent and that techniques need to be verified for every application.

CONCLUSION

This comparative study concludes that, in comparison to conventional wet digestion and DLLME, the modified QuEChERS method is a more efficient and dependable sample preparation methodology for the extraction of heavy metals (Cr, Ni, Co, and Cd) from sweet potato tubers. QuEChERS is a superior modern method for routine food safety monitoring because of its performance and intrinsic benefits of being quicker, easier, safer, and more ecologically friendly due to lower reagent use (Akilu & Lawal, 2025). Wet digestion is still a reliable method, although there are some significant safety and contamination risks. Despite being a potent microextraction method, DLLME doesn't seem to be as well-suited for direct application to intricate solid food matrices in the absence of a previous digesting stage.

The recovery rates for QuEChERS and wet digestion were generally satisfactory (85-98%), falling within the acceptable range for trace metal analysis, thereby confirming the methods' accuracy for this matrix (Núñez et al., 2022). The low RSD values (<5%) for these methods indicate good precision. The higher RSDs and lower recoveries for DLLME highlight its challenges with the complex solid matrix. The LOD and LOQ values confirm that the AAS method, combined with the sample preparation techniques, is sufficiently sensitive to detect and quantify the target heavy metals at the levels found in the sweet potato samples.

RECOMMENDATIONS

The following recommendations are made in light of the study's findings:

i. The QuEChERS method has to be implemented and improved for the multi-residue analysis of heavy metals in different root and tuber crops.
ii. To increase the extraction efficiency for a larger spectrum of metals, including Pb, future research should concentrate on discovering and validating particular chelating agents to add to the QuEChERS process.
iii. In order to reach lower detection limits, laboratories could look into using DLLME as a supplemental pre-concentration and cleanup step following a primary extraction (e.g., using QuEChERS).
iv. To guarantee accuracy and spot possible contamination, especially when utilizing wet digestion, stringent quality control procedures, such as the analysis of certified reference materials and method blanks, must be followed regardless of the approach selected.

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