**Assessing the Profile and Human Health Risk of Organochlorine Residues in Packaged Herbal Tea in Abuja, Nigeria**

**ABSTRACT**

**Aim:** The organochlorine insecticide residues in herbal teas in the Federal Capital Territory (FCT) Abuja were determined, and their human health risk was estimated.

**Methodology:** Twelve different herbal tea products were procured from Abuja market stores.The herbal tea samples were extracted and cleaned-up according to QuEChERS-AOAC 2000 method and organochlorine pesticides (OCPs) quantification were performed using Gas chromatography Mass Spectrometer (GC-MS). Assessment of health risks were achieved by computing the Estimated Daily intake (EDI) and Hazard Quotient (HQ) and values obtained were benched marked with the Acceptable Daily Intake (ADI) of 20 OCPs and 100 threshold limits respectively. **Result:** The lowest percentage recovery was 95.2% in δ-HCH, confirming the suitability and appropriateness of the extraction procedure and the GC-MS analyses employed.All the tea samples were contaminated with the 20OCs insecticide except for endosulfan sulphate in the Lipton tea. Among the tea samples, heptachlor concentration was the highest (1.641 mg kg-1), followed by Aldrin (0.802 mg kg-1), a cyclodiene that is among the banned insecticides worldwide. The concentration of alpha-BHC was notably higher in Mango, Lem-Gin, and Highland teas, reaching up to 0.356 mg kg-1, against the EU MRL of 0.005 mg kg-1. Delta. The HRI of residues of Alpha-BHC, p,p’-DDE, p,p’-DDD, Endosulfan II, p,p’-DDT, endosulfan sulphate, methoxychlor and endrin ketone HQ in the herbal teas were far above the threshold limit of 100. The OCPs that exhibited the lowest HRI were Endosulfan sulphate (7.337) and Methoxychlor (9.805). **Conclusion:** This study revealed significant contamination of the herbal tea with banned OCs and results from health risk assessment suggest that no health risk as a result of long-time consumption of the herbal products. Nevertheless, stricter regulations and routine monitoring by the regulator are essential to control OCs levels in the beverage.

*Keywords: Food safety, Contaminants, Occurrence, Tea, Abuja-Nigeria.*

1. **INTRODUCTION**

The production and consumption of herbal supplements are fast-growing in Nigeria. Unfortunately, the raw materials are often sourced from localities or countries with inadequate Good Crop Production Practices (GCPPs) and quality control regarding pesticide application on the farm and during storage. Herbal teas, crafted from various plant blends and packaged in convenient containers and bags, offer aromatic and refreshing beverages and therapeutic and medicinal benefits (Zohora *et al*., 2022; Aguilar-Pérez *et al*., 2023).

Organochlorine (OC) residue in food commodities has been implicated in endocrine disruption, reproductive issues, developmental abnormalities, cancer, nervous system, kidney failure, cognitive impairments, and other long-term health problems (Lentini P, Zanoli *et al*., 2017) reported multi-residue organochlorine pesticide and health risk assessment in edible vegetables (Fernandes *et al*., 2023), while Akande *et al*. 2020 also evaluated organophosphate insecticide in post-harvest cowpea in Gwagwalada Abuja. Idowu *et al*. (2022) examined organochlorine residue in cocoa pods and beans from Nigeria while Fagbohun *et al*. (2024) reported several organochlorine residues in cowpea grains from Abuja, Nigeria.

Strict monitoring, adherence to safety standards and sustainable practices are essential to mitigate these concerns and ensure the safety of the food supply (Hoque *et al*., 2022). Regular testing, sustainable farming practices, and public awareness ensure that the food supply remains safe and free from harmful contaminants (Fagbohun *et al*., 2023). There is a paucity of current information on the level of OCs insecticide residue in retailed packaged herbal tea in the FCT, Abuja. To provide safe food and ensure food security, there is a need to determine the pesticide residues in food products (Kolakowski *et al*., 2020). Thus, the study aimed to assess the level of OC residue in marketed packaged herbal teas in Abuja, Nigeria and determine its health risk to the consumers in the study area.

1. **MATERIALS AND METHODS**

**2.1 Sample Collection**

Various herbal tea products were procured from market stores in the Gwagwalada and Kuje area Councils of the FCT, Abuja. The types of tea beverages were packaged as Green, Guava, Moringa, Beetroot, Mango, Lipton, Top Tea, Mint, High land, Fat reduction, and Eyebright teas. The collected samples were coded and swiftly transported to the Chemistry Advanced Research Centre, Sheda Science and Technology Complex (SHESTCO), Abuja. They were then kept in a -20 oC freezer pending analytical determination.

**2.2 Reagents and Materials**

The chemicals used include organochlorine pesticide (OCPs) standard, acetonitrile, acetone, and methanol; all solvents are 99.90 % HPLC grade and purchased from Sigma-Aldrich USA. Besides, Sodium sulphate (Na₂SO4), Magnesium sulphate anhydrous fine powder (MgSO4), graphitized carbon black (GCB), primary, secondary amine (PSA), disodium hydrogen citrate sesquihydrate (C6H6Na2O71.5H2O), trisodium citrate dehydrate (C6H2Na3O7.2H2O), sodium chloride (NaCl), Solid phase extraction tubes (SPE), ceramic discs, were all purchased from Bioccomma Limited, Hong Kong.

**2.3 Sample Extraction and Clean-up**

The Quick, Easy, Cheap, Efficient, Rapid, and Safe (QuEChERS) method and dispersive liquid-liquid micro-extraction (DLLME) were used for sample extraction as previously described by AOAC Official method, 2007-01 (Huertas-Pérez *et al*., 2024). Ten grams of finely pulverized sub-sample was transferred into a polypropylene centrifuge tube (50 mL) and 10 mL water was added for hydration and vortexed for two minutes. Followed by the addition of 15mL acetonitrile, and the mixture was again vortexed vigorously for 5 mins. Further, 0.5 g disodium hydrogen citrate sesquihydrate, 1g trisodium citrate dihydrate, 4 g anhydrous magnesium sulphate, and 1 g sodium chloride were added, and the mixture was immediately vortexed again for another five minutes, then centrifuged at 4500rpm for 5 min. At this stage, an optional low-temperature clean step was performed before dispersive-SPE for the most complex matrices, such as herbal tea. For this, an aliquot of the supernatant was transferred into a glass test tube and stored in a freezer for five minutes (−20 oC). The extract was then separated from the precipitates by simple decantation. An aliquot of the extract was transferred into a solid-phase extraction tube containing 100 mg anhydrous magnesium sulphate, 75 mg GCB, and 20 mg PSA per mL acetonitrile extract. The extract was eluted into a GC vial by gravity method and acidified by adding 15µL of 5 % (v/v) formic acid in acetonitrile per mL of extract and analyzed for OCPs using GC-MS.

**2.4 Gas Chromatography-Mass Spectrophotometry**

The concentrations of OCPs in the sample extracts were determined using an Agilent HP-5-60 to 325 oC GC column (30 m × 320 m x 0.25 m film thickness) attached to a gas chromatograph (6890N Agilent Technologies) and a mass selective detector (Agilent 5975B) (GC-MS). The volume of the sample injected in the splitless mode was 1µL. The initial oven temperature was maintained at 100 oC for 2 minutes, then increased to 180 oC at a rate of 15 oC/minute, ramped up to 300 oC at a rate of 3 oC/minute, and held for 9 minutes. The carrier gas was helium with a 0.8 mL/min flow rate. The operation mode of the mass spectrometer was electron impact ionization with the use of automatic gain control. The storage window was programmed at full scan mode in the range of m/z 200–500, and the selected ion monitoring (SIM) mode was employed in acquiring data by Agilent Chemstation software.

**2.5 Quality control Measures**

All the glass wares used were washed and rinsed with Milli-Q water and acetone before use. The calibration curves were obtained by injecting eight different concentrations of the pesticide standards in a 4-300 ng ml-1 range. The linear regression's correlation coefficient (r2) values obtained from the plot of known concentrations of OCPs against their peak areas ranged between 0.9997 and 0.9999. The concentrations of OCPs in the samples were quantified using the external calibration method according to AOAC (2007).

The limit of determination (LODs) of the OCPs compound ranged from 0.03 to 0.20 ng g-1. Limits of detection (LOD) and limits of quantification (LOQ) of the method were measured by spiked serial dilution of working standards prepared for calibration curves. This was calculated by considering a value of 3 and 10 times of background noise, respectively. LOD was determined by considering it to be 3 times the signal-to-noise ratio, while LOQ was determined to be 10 times the signal-to-noise ratio. This means that LOD and LOQ were determined as the lowest concentrations, yielding a signal-to-noise (S/N) ratio of 3 and 10, respectively (Liao *et al*., 2018; Barriga-Vélez *et al.,* 2023).

The accuracy of the developed method was evaluated by measuring the extraction recovery of the spiked tea as the herbal tea sample was spiked with a solution containing a mixture of the 20 OCPs pesticide standards. A pesticide standard was spiked into a laboratory blank sample to give 0.25 mg g-1, and recovery was based on 4 replicates. The spiked samples were left for 1 hour before extraction to allow the insecticide residue to partition into the matrices, and the percentage recovery was obtained as shown in equation 1 according to (Quesada *et al*., 2023).

C:\Users\TSANJO~1\AppData\Local\Temp\ksohtml8948\wps6.jpg Eqn i

**2.6 Human Health Risk Assessment**

An assessment of health risks was performed by comparing the mean levels of contaminants with international guidelines. However, these assessments do not consider consumption rates and eating habits. The estimated daily intake (EDI) of the 20 pesticides (alpha-BHC, beta-BHC, gamma-BHC, heptachlor, delta-BHC, aldrin, heptachlor epoxide, gamma chlordane, alpha-chlordane, endosulfan I, p,p'-DDE, dieldrin, endrin, p,p'-DDD, endosulfan II, p, p'-DDT (dichlorodiphenyltrichloroethane), endrin aldehyde, endosulfan sulphate methoxychlor, endrin ketone) were determined based on their mean concentration in the herbal tea and the daily intake in grams.

The average annual consumption rate of herbal tea in Nigeria is approximately 0.6 kg/person/year (Bhawan, 2022). The food supply value was divided by the number of days in the year (365 days). The result obtained was an intake of 0.0016 kg/capita/day), which is the food ingestion rate (FIR) of herbal teas in Nigeria. Equation 1 was used for the calculation of the EDI (Antoine *et al*., 2017).

……..Eqn ii

where:

C is the dry weight concentration of the organochlorine pesticides residue in the herbal tea variety in mg kg-1, FIR is the daily FIR in kg/day, and BW is the reference body weight of an adult human at 60 kg.

The Health Risk Index (HRI) or Hazardous Quotient value is a dimensionless index that represents the potential health risk associated with a long-term exposure to a particular contaminant, as stated in equation iii (Elgueta *et al*., 2017).

………….Eqn iii

The Acceptable Daily Intake (ADI) is the quantity of a definite chemical that a human can ingest daily for the life span of the human without substantial health risks (Verger *et al.,* 2013).

**2.7 Statistical analysis**

Elements of descriptive statistics of samples generated included mean, range, minimum, maximum, and standard deviations, which were analyzed using MS Excel and SPSS version 21. The concentration of organochlorine insecticide residues in herbal samples were graphically represented and compared with the Maximum Residual Limit (MRL) recommended by the European Union (Authority *et al*., 2022).

**3. RESULTS**

**3.1 Recoveries of Organochlorine Compounds**

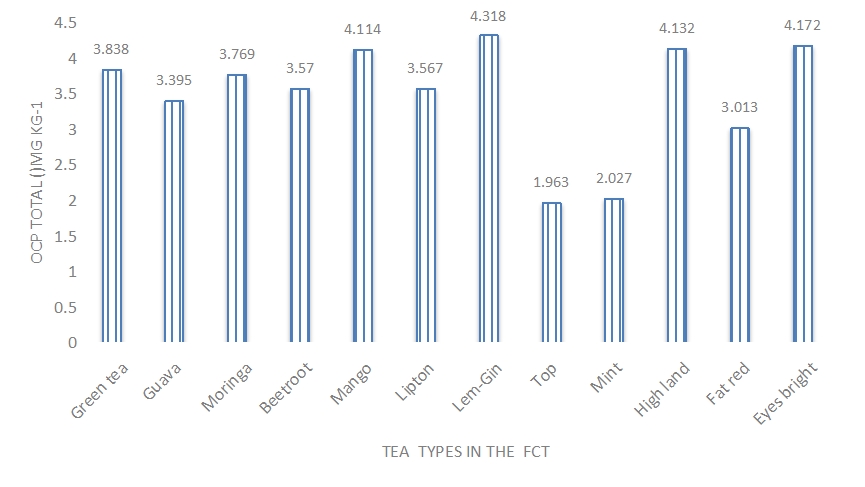
Percentage recoveries of each organochlorine compound for the percentage recoveries as presented in the Table 1 ranged from 96.04 % (Alpha-Chlordane) to 99.24 % (Endosulfan II) confirming the suitability and appropriateness of the extraction procedure and the GC-MS analyses employed. The Fortification concentration used was 25.00 µg/kg.

**Table 1: Percentage Recovery of OCPs in Herbal Tea from Abuja, Nigeria**

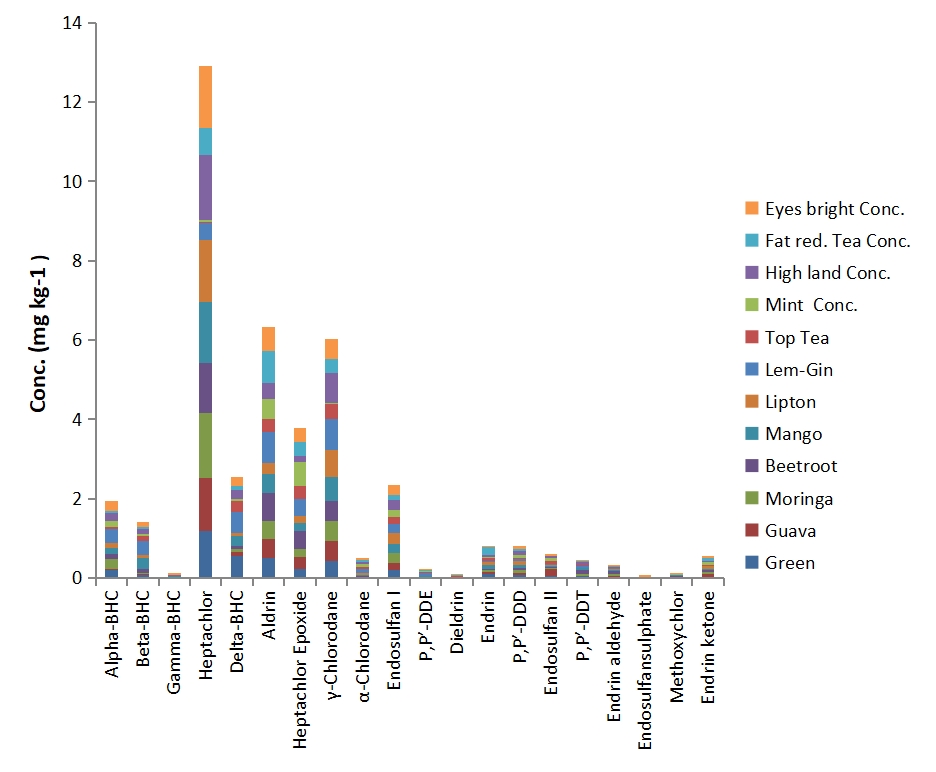
|  |  |  |
| --- | --- | --- |
| **Compound** | **Residue concentration (µg/kg)** | **% Recovery** |
| Alpha-BHC | 24.12 | 96.48 |
| Beta-BHC | 24.23 | 96.92 |
| Gamma-BHC | 24.30 | 97.20 |
| Delta-BHC | 24.47 | 97.88 |
| Heptachlor | 24.42 | 97.68 |
| Heptachlor Epoxide | 24.53 | 98.12 |
| Gamma- Chlordane | 24.58 | 98.32 |
| Alpha-Chlordane | 24.01 | 96.04 |
| Endosulfan I | 24.64 | 98.56 |
| Endosulfan II | 24.81 | 99.24 |
| Endosulfan sulphate | 24.70 | 98.80 |
| p p'-DDE | 24.67 | 98.68 |
| p p'-DDD | 24.76 | 99.04 |
| p p'-DDT | 24.58 | 98.32 |
| Aldrin | 24.50 | 98.00 |
| Dieldrin | 24.67 | 98.68 |
| Endrin | 24.69 | 98.76 |
| Endrin aldehyde | 24.40 | 97.60 |
| Endrin ketone | 24.76 | 99.04 |
| Methoxychlor | 24.75 | 99.00 |

**3.2 Profile of Organochlorine residues in Herbal teas in Abuja, Nigeria**

Figure 1 depicts the total OCP residues found in the tea samples from the FCT, Abuja. The highest combine OCPs residue was in lemon ginger tea (4.318 mg kg-1). This was closely followed by the residue in Eyebright (4.172 mg kg-1). The least range of the OCP residue was found in Top tea (1.963 mg kg-1) and Mint tea (2.027 mg kg-1), respectively. Also Figure 2 shows the concentration of organochlorine residue in all the herbal tea brands from FCT, Abuja, where heptachlor residue had the highest occurrence in the herbal teas.



**Figure 1: Cumulative Organochlorine Residue per Herbal Tea Brand from the FCT, Abuja**



**Figure 2: Profile of Organochlorine Residues in 10 Herbal Tea brands from FCT, Abuja**

Among the tea samples, heptachlor concentration was found to be the highest (1.641 mg kg-1), followed by Aldrin (0.802 mg kg-1), a cyclodiene that is among the banned insecticides worldwide (Table 2). The lowest residual concentrations in the herbal teas were Gamma BHC and Endosulfan sulphate (0.001 mg kg-1), respectively. The mean concentration was also highest in the Heptachlor, and the lowest in the endosulfan sulphate.

**Table 2: Maximum, Minimum and Mean Concentration of 20 organochlorine residue in Herbal teas in Abuja Nigeria**

|  |  |  |  |
| --- | --- | --- | --- |
| **Tea/OCP (mg kg-1 )** | **Max** | **Min** | **Mean** |
| Alpha-BHC | 0.356 | 0.004 | 0.161 |
| Beta-BHC | 0.368 | 0.010 | 0.214 |
| Gamma-BHC | 0.020 | 0.001 | 0.015 |
| Heptachlor | **1.641** | 0.020 | 1.832 |
| Delta-BHC | 0.567 | 0.010 | 0.378 |
| Aldrin | 0.802 | 0.050 | 0.901 |
| Heptachlor Epoxide | 0.589 | 0.020 | 0.546 |
| Gamma-Chlordane | 0.772 | 0.010 | 0.854 |
| α-Chlordane | 0.101 | 0.010 | 0.075 |
| Endosulfan I | 0.261 | 0.005 | 0.333 |
| p,p’-DDE | 0.092 | 0.003 | 0.037 |
| Dieldrin | 0.050 | 0.002 | 0.017 |
| Endrin | 0.214 | 0.010 | 0.127 |
| p,p’-DDD | 0.099 | 0.010 | 0.117 |
| Endosulfan II | 0.159 | 0.012 | 0.094 |
| p,p’-DDT | 0.086 | 0.004 | 0.068 |
| Endrin aldehyde | 0.060 | 0.006 | 0.052 |
| Endosulfan sulphate | 0.050 | 0.001 | 0.014 |
| Methoxychlor | 0.021 | 0.002 | 0.016 |
| Endrin ketone | 0.084 | 0 .011 | 0.082 |

The data presented in Table 3 show that all the tea samples were contaminated with the 20OCPs insecticide except for endosulfan sulphate in the Lipton tea. The OCPs were classified as benzene hexachloride, cyclodienes, chlorodane, endosulfans, pp’ and its isomers, and methoxychlor. European Union (EU) Maximum Residue Limits (MRLs) in the Table, provides insights into the degree to which these pesticide residues exceed international safety standards. Ten of the 12 tea brands, violated the EU MRLs. The concentration of alpha-BHC were notably higher in Mango, Lem-Gin, and Highland teas, reaching up to 0.356 mg kg-1, against the EU MRL of 0.005 mg kg-1. Delta BHC had the highest concentration in the green tea, and lowest in the Gamma-BHC in Lipton. In all the BHC residues detected.

Cyclodiene compounds, including aldrin, heptachlor, heptachlor epoxide, dieldrin, endrin, endrin aldehyde, and endrin ketone were found with varied and relatively high concentrations that exceeded the EU MRLs. Aldrin residual concentration was highest in fat reduction tea (0.802 mg kg-1). This was followed by lemon ginger tea (0.765 mg kg-1) and then Beetroot tea (0.707 mg kg-1), Lipton tea had the lowest residual OCPs concentration (0.286 mg kg-1). Among the cyclodines, dieldrin, appeared to have the lowest concentration range (0.012 mg kg-1) in Top tea to (0.002 mg kg-1) in mint tea in this study.

All the tea samples analyzed were contaminated with heptachlor and its metabolite. The residual concentration of heptachlor epoxide in the tea samples was higher than the EU MRLs of 0.0001 mg kg-1. Moringa tea had the highest contamination residue of heptachlor1.641 mg kg-1), followed by highland tea (1.639 mg kg-1) and Eyebright tea (1.576 mg kg-1), while mint tea (0.040 mg kg-1) had the lowest OCPs residue of heptachlor. Heptachlor epoxide, a metabolite of heptachlor, displayed a consistently elevated concentration across the samples such as mint (0.589 mg kg-1), beetroot (0.442 mg kg-1) and Lemgin (0.438 mg kg-1).

Endrin and its metabolites, including endrin aldehyde and ketone exceeded their respective MRLs. Zhang *et al.* (Xie *et al*., 2019) reported a mean Endrin residue of 0.5 mg kg-1 indry weight in herbal teas. The values recorded for Alpha and Gamma chlordane in the sample teas were also higher than the EU MRLs. The two alpha and gamma chlordane residual concentrations ranged between 0.010-0.101 mgkg-1and 0.01 - 0.772 mg kg-1, respectively. In lemon green tea, the highest concentration of Gamma-chlordane (0.772 mg kg-1) occurred, while the lowest concentration residue (0.043 mg kg-1) was found in mint tea. Also, the highest concentration of Alpha-chlordane (0.101 mg kg-1) was found in lemon ginger, while the least residual concentration (0.010 mg kg-1) was found in guava tea.

All the herbal teas were contaminated with endosulfan sulphate except Lipton tea. These values were within the EU limit except in highland tea (0.008 mg kg-1). The values obtained for endosulfan I ranged between 0.005 mg kg-1 (beetroot) to 0.261 mg kg-1 (Lipton). Guava had the highest Endrin ketone residue (0.084 mg kg-1). For Endrin aldehyde, residual concentrations in the herbal tea ranged from 0.006 mg kg-1 in (highland tea) to 0.06 mg kg-1 (beetroot tea).

The pp’ DDE, a metabolite of pp’ DDT, was contaminated in the range of 0.003 to - 0.092 mgkg-1. The pp’ DDE residue in all the tea samples was below EU's MRLs except for in Lemon gin tea at 0.092 mg kg. The concentration of pp’ DDD in all the samples was above the EU MRL except in the Top tea (0.010 mg kg-1). All the tea samples were contaminated with pp’DDT with the maximal concentration of 0.086 mg kg-1 in beetroot. Methoxychlor insecticide, was also detected in concentrations above the EU limit of 0.005 mg kg-1 in teas like Guava (0.011 mg kg-1), Moringa (0.017 mg kg-1) and Beetroot (0.013 mg kg-1), methoxychlor concentration ranged between 0.002 -0.021 mg kg-1. The concentration of methoxychlor in Mango tea, Highland tea, Fat reduction tea and Eyes bright tea were all below the EU standard.

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **OCP**  **(mg kg-1)**  **Subclass** | **Green Tea** | **Guava** | **Moringa** | **Beetroot** | **Mango** | **Lipton** | **Lem-Gin** | **Top tea** | **Mint** | **High land** | **Fat red** | **Eyes bright** | **EU MRL (mg kg-1)** |
| **Benzene Hexachloride** | | | | | | | | | | | | | |
| Alpha-BHC | 0.219 | 0.004 | 0.259 | 0.123 | 0.160 | 0.120 | 0.356 | 0.047 | 0.151 | 0.195 | 0.058 | 0.242 | 0.005 |
| Beta-BHC | 0.076 | 0.038 | 0.028 | 0.099 | 0.267 | 0.066 | 0.368 | 0.110 | 0.061 | 0.119 | 0.062 | 0.125 | 0.003 |
| Gamma-BHC | 0.009 | 0.006 | 0.015 | 0.012 | 0.004 | 0.001 | 0.006 | 0.004 | 0.006 | 0.009 | 0.02 | 0.008 | 0.0003 |
| Delta-BHC | 0.567 | 0.102 | 0.067 | 0.064 | 0.265 | 0.082 | 0.517 | 0.274 | 0.065 | 0.222 | 0.098 | 0.222 | 0.005 |
| **Cyclodienes** | | | | | | | | | | | | | |
| Dieldrin | 0.005 | 0.004 | 0.005 | 0.006 | 0.006 | 0.003 | 0.005 | 0.012 | 0.002 | 0.008 | 0.012 | 0.006 | 0.0001 |
| Endrin | 0.094 | 0.061 | 0.058 | 0.025 | 0.106 | 0.058 | 0.025 | 0.085 | 0.01 | 0.052 | 0.214 | 0.033 | 0.0002 |
| Aldrin | 0.502 | 0.487 | 0.453 | 0.707 | 0.478 | 0.286 | 0.765 | 0.331 | 0.518 | 0.397 | 0.802 | 0.605 | 0.0001 |
| Endrin aldehyde | 0.025 | 0.033 | 0.051 | 0.060 | 0.028 | 0.031 | 0.030 | 0.016 | 0.011 | 0.006 | 0.015 | 0.026 | 0.0002 |
| Endrin ketone | 0.032 | 0.084 | 0.044 | 0.059 | 0.013 | 0.063 | 0.011 | 0.033 | 0.068 | 0.025 | 0.078 | 0.041 | 0.0002 |
| Heptachlor | 1.189 | 1.345 | 1.641 | 1.252 | 1.524 | 1.567 | 0.432 | 0.025 | 0.04 | 1.639 | 0.681 | 1.576 | 0.0001 |
| Heptachlor Epoxide | 0.239 | 0.298 | 0.203 | 0.442 | 0.212 | 0.161 | 0.438 | 0.336 | 0.589 | 0.173 | 0.332 | 0.369 | 0.0001 |
| **Chlordane** | | | | | | | | | | | | | |
| γ-Chlordane | 0.436 | 0.498 | 0.512 | 0.493 | 0.623 | 0.674 | 0.772 | 0.372 | 0.043 | 0.737 | 0.362 | 0.495 | 0.0005 |
| α-Chlordane | 0.014 | 0.010 | 0.014 | 0.023 | 0.025 | 0.055 | 0.101 | 0.044 | 0.068 | 0.053 | 0.06 | 0.039 | 0.0005 |
| **Endosulfan** | | | | | | | | | | | | | |
| Endosulfan I | 0.214 | 0.180 | 0.233 | 0.005 | 0.234 | 0.261 | 0.246 | 0.162 | 0.18 | 0.256 | 0.129 | 0.244 | 0.005 |
| Endosulfan II | 0.064 | 0.159 | 0.036 | 0.032 | 0.020 | 0.027 | 0.014 | 0.075 | 0.07 | 0.052 | 0.012 | 0.036 | 0.008 |
| Endosulfan sulphate | 0.005 | 0.004 | 0.001 | 0.006 | 0.002 | BDL | 0.001 | 0.004 | 0.001 | 0.008 | 0.006 | 0.006 | 0.006 |
| **DDT and its isomers** | | | | | | | | | | | | | |
| pp’- DDE | 0.010 | 0.012 | 0.009 | 0.008 | 0.008 | 0.003 | 0.092 | 0.016 | 0.02 | 0.012 | 0.012 | 0.008 | 0.02 |
| pp’-DDD | 0.083 | 0.053 | 0.069 | 0.055 | 0.083 | 0.086 | 0.067 | 0.010 | 0.086 | 0.099 | 0.042 | 0.072 | 0.02 |
| pp’-DDT | 0.053 | 0.006 | 0.054 | 0.086 | 0.054 | 0.013 | 0.052 | 0.004 | 0.023 | 0.068 | 0.013 | 0.017 | 0.02 |
| **Methoxychlor** | **0.003** | 0.011 | 0.017 | 0.013 | **0.002** | 0.010 | 0.021 | **0.003** | 0.015 | **0.002** | **0.005** | **0.002** | 0.005 |
| **Sum** | **3.838** | **3.395** | **3.769** | **3.57** | **4.114** | **3.567** | **4.318** | **1.963** | **2.027** | **4.132** | **3.013** | **4.172** | - |

**Table 3: Concentration Profile of Organochlorine Residues in the Herbal Tea brands from FCT, Abuja compared with EU MRL**

**BDL**= Below the Detection

**3.3 Health Risk Assessment of 20 Organochlorine Residue in Herbal teas in Abuja Nigeria**

Table 4 depicts the long-time risk exposure of consumption of contaminated herbal teas with OCPs insecticides. The residues of Alpha-BHC, p,p’-DDE, p,p’-DDD, endosulfan II, p,p’-DDT, endosulfan sulphate, methoxychlor and endrin ketone EDI in the herbal teas were all lower than their respective ADIs as their HRI values were far above the threshold limit of 100. The OCPs that exhibited the lowest HRI were endosulfan sulphate and methoxychlor withHRI of 9.805 and 7.337 respectively. The EDI of other 12 OCPs were higher than their respective ADIs indicating their long-time risk exposure.

**Table 4: Estimated Daily Intake, Acceptable Daily Intake and Health Risk Index of Organochlorine Residue in Herbal teas in Abuja Nigeria**

|  |  |  |  |
| --- | --- | --- | --- |
| **OCP in teas** | **EDI ((mg kg-1 )** | **ADI** | **HQ** |
| Alpha-BHC | 0.00495 | 0.005 | 99.179 |
| Beta-BHC | 0.00659 | 0.003 | **219.897** |
| Gamma-BHC | 0.00047 | 0.0003 | **157.265** |
| Heptachlor | 0.05637 | 0.0001 | **56375.385** |
| Delta-BHC | 0.01163 | 0.005 | **232.492** |
| Aldrin | 0.02772 | 0.0001 | **27721.026** |
| Heptachlor Epoxide | 0.01681 | 0.0001 | **16806.154** |
| Gamma-Chlordane | 0.02629 | 0.0005 | **5257.846** |
| α-Chlordane | 0.00230 | 0.0005 | **460.718** |
| Endosulfan I | 0.01025 | 0.005 | **205.087** |
| p,p’-DDE | 0.00115 | 0.020 | 5.764 |
| Dieldrin | 0.00051 | 0.0001 | **508.718** |
| Endrin | 0.00391 | 0.0002 | **1954.872** |
| p,p’-DDD | 0.00361 | 0.020 | 18.041 |
| Endosulfan II | 0.00288 | 0.008 | 35.974 |
| p,p’-DDT | 0.00209 | 0.020 | 10.482 |
| Endrin aldehyde | 0.00158 | 0.200 | 125.9 |
| Endosulfan sulphate | 0.00044 | 0.006 | 7.337 |
| Methoxychlor | 0.00049 | 0.005 | 9.805 |
| Endrin ketone | 0.00254 | 0.200 | 78.895 |

This study assessed the Health Risk Assessment (HRA) of 20 organochlorine insecticide residues in herbal teas marketed in Abuja. Heptachlor exhibited the highest HRI (56375.385) against the threshold limit of 100. Aldrin followed this with an HRI of 27721.026. The third OCP with high HRI was heptachlor epoxide (16806.154). From this study, Beta-BHC, Delta-BHC, deldrin, and Alpha-chlordane all had moderate to high-risk OCPs, p,p’-DDE and p,p’-DDD, endosulfan I and II had relatively lower relative risk Endrin and endrin ketone had an HRI of 1,954.872 while endrin ketone showed an HRI of 1,267.692, indicating high risk from both compounds.

1. **DISCUSSION**

The detection of OCP residues in herbal tea samples indicated significant contamination of several classes, many of which exceeded EU standards. This contamination might be due to using OCPs during cultivation or in storage (Yigit and Velioglu, 2020; Taiwo, 2019). These residues raise concerns about potential health risks for consumers, especially chronic exposure. Similar studies on OCP residues in Nigeria have detected pesticides in herbal teas, medicinal plants, and food products above regulatory limits. For instance, Taiwo (Alani *et al*., 2023) reported high levels of OCPs, including aldrin, endrin, and heptachlor, in agricultural and medicinal plants from Northern Nigeria. Values were often in the 0.05–0.2 mg kg-1 range, similar to the relatively high concentrations of OCP residues found in Abuja herbal teas. Studies by Alani et al. (2023) also highlighted the high prevalence of aldrin, dieldrin, and endosulfan residues above EU standards in various food products across Lagos, Nigeria, with levels reaching 0.100 mg kg-1 - 0.800 mg kg-1. Kumar *et al.(2025)* reported an aldrin residue of 0.06–0.100 mg kg-1dry weight in therapeutic teas*,*

International Research on herbal teas and traditional medicines in India, such as by Chaudhuri *et al.* (2019) , also identified alpha-BHC and endrin contamination at levels comparable to those in Abuja samples. Concentrations reached up to 0.300 mg kg-1, notably in chlordane and BHC isomers, underscoring the global persistence of these compounds. Chlordane’s toxicity and long-term environmental persistence make these findings particularly concerning for public health.

Benzene was detected at a concentration similar to what was obtained in the current study at a range of 0.015 – 0.378 mg kg-1. González-Hernández *et al.* (2022) found Alpha-BHC levels between 0.002 mg kg-1 and 0.032 mg kg-1 in herbal teas, and Zhang *et al*. (2019) reported Beta-BHC values were 0.015 mg kg in black tea. Wang et al. 2019 reported a mean concentration of 0.164 ug/kg dry weight in green tea. These values were all significantly lower than those found in this study. This study’s values reflect a concerning trend of higher pesticide residues, likely due to continued field or storage contamination despite restrictions on their use. Benzene hexachloride is a fumigant pesticide used in the early 1960s and was later banned by WHO for its acute toxicity and as an endocrine disruptor.

Previous research in the EU and North America reports much lower OCPs levels in herbal products, possibly due to stringent regulations. Studies by Emoyan *et al*. (2022) found that most OCPs residues were either non-detectable or within the permissible range (< 0.01 mg kg-1), showing the effectiveness of strict regulatory oversight. This revelation underscores the need for increased monitoring, policy enforcement, and public awareness to mitigate health risks associated with OCP residues.

Aldrin was detected from 0.025 mg kg-1 in Mint tea to 0.802 mg kg-1 (Fat Red), well above the EU limit of 0.0001 mg kg-1. Opuni *et al*. [26] reported dieldrin residues in maize and grains up to 0.011 mg kg-1, and endrin levels ranged from 0.002 mg kg-1 to 0.045 mg kg-1, aligning closely with the lower end of the concentrations found in this study. The elevated levels of aldrin and endrin found in this study reflect the continued contamination from their use. Adenuga *et al*. [27] detected Heptachlor residues of 0.004 - 0.008 mg kg-1 dry weight in Indian herbal teas, and Xie *et al* . [24] reported a mean concentration of 0.012 mg kg-1 dry weight in tea, which is much lower than those found in this study. Thus, there is a potential risk for significant exposure to Heptachlor through herbal tea consumption in Abuja, Nigeria.

Gamma-chlordane with an HRI of 5,257.846, suggests substantial toxicity. Chronic exposure has been linked to immune suppression and carcinogenic potential. The compound’s bio accumulative nature exacerbates long-term risks. Gamma chlordane ranged from 0.043 mg kg-1 (Mint) to 0.772 mg kg-1in Lem-Gin, exceeding the EU limit of 0.0005 mg kg-1 in all samples. Alpha-chlordane was found at levels between 0.010 mg kg-1 (Guava) and 0.101 mg kg-1 (Lem-Gin), surpassing the EU standard of 0.0005 mg kg-1 in all samples. (Tarawneh, 2022) reported γ-Chlordane values of 0.002–0.054 mg kg-1 in herbal tea samples, significantly lower than the concentrations found in this study. Endosulfan Sulphate ranged from 0.001 mg kg-1 - to 0.008 mg kg-1, with most levels being within the EU standard of 0.006 mg kg-1. Yang *et al*. (2022) reported Endosulfan I residues between 0.012–0.045 mg kg-1 in cereals, which aligns with the lower range of concentrations in this study. The detection of Endosulfan I and II at levels exceeding the EU standards in this study

Mukasa *et al.* (2022) detected p,p’-DDT at 0.001–0.045 mg kg-1 in Kenya, which consistent with the low to moderate levels found in this study. The detection of pp’-DDE and other pp’ -DDT metabolites requesting for more constant surveillance study on this beverage in Nigeria. Methoxychlor was detected in concentrations of 0.001 mg kg-1 in Lipton to 0.021 mg kg-1 in Lem-Gin, exceeding the EU limit of 0.005 mg kg-1 in the tea samples. Adenuga *et al.* (2022) found methoxychlor residues at a range of 0.050–0.300 ng/g tea dry weight highlighting the level of methoxychlor contamination in Nigeria’s food as opined in this current study. The ability of this pesticides to act as an endocrine disruptor, methoxychlor contamination in food warrants attention and stricter quality control measures, even at low levels (Lokesha *et al*., 2017) The presence of methoxychlor suggests potential misuse or contamination during production and storage, even though its use has been largely phased out.

With an HRI of 219.897 of Beta-BHC, it indicated significant neurotoxic potential. Prolonged exposure could disrupt hormonal and nervous system functions. Delta-BHC presented an HRI of 232.492, significantly exceeding the acceptable limit. Its bio accumulative nature and endocrine-disrupting effects pose a notable risk to public health (Oshatunberu, 2023).

Dieldrin's HRI of 508.718 indicated its high health risk. It is a known neurotoxin and carcinogen, with chronic exposure causing liver and kidney damage (Trivedi *et al*., 2025). Alpha-Chlordane exhibited an HRI of 460.718, with a potential for long-term effects on the immune system (Ansari *et al*., 2024). Although less potent than Gamma-chlordane, Apha-chlordane’s cumulative effects cannot be ignored. p,p’-DDE and p,p’-DDD metabolites of DDT had relatively low HRIs (5.764 and 18.041, respectively) compared to other compounds in the sampled teas,

The Health Risk Assessment (HRA) indicates that certain OCPs in herbal teas exceed safety thresholds by several orders of magnitude. Agricultural Organization (FAO), the World Health Organization (WHO), and the CODEX Alimentarius Commission (CODEX) have previously set Maximum Residue Limits (MRLs) and Acceptable Daily Intake (ADI) for insecticide residues on various foods, including herbal tea. These MRLs are based on scientific studies and aim to ensure that insecticide residues in food products remain below the threshold deemed safe for human consumption. Notably, heptachlor, aldrin, heptachlor epoxide, and Gamma-chlordane pose the most immediate and severe health risks, underscoring the urgent need for regulatory intervention. The high HRI values associated with these compounds suggest a critical public health risk, especially in cases of frequent or prolonged consumption.

Cyclodienes such as aldrin and dieldrin have been classified by the International Agency for Research on Cancer (IARC) as possibly carcinogenic to humans (Group 3), with studies suggesting an association with cancers of the liver, pancreas, and brain (IARC, 2018). Recent studies have also shown that cyclodienes can disrupt the endocrine system, potentially affecting thyroid and reproductive hormones. Previous study has documented an association between high levels of dieldrin and heptachlor in pregnant women and altered fetal development outcomes (Soman *et al*., 2024).

Many detected OCPs (e.g., aldrin, dieldrin, endrin) are associated with neurotoxicity and potential carcinogenicity. Chronic exposure to these compounds, even at low concentrations, can lead to irreversible health impacts, including developmental and reproductive toxicity. Compounds like Beta-BHC, Delta-BHC, and endosulfan are endocrine disruptors. Even at low levels, their presence can have far-reaching effects on hormonal regulation, potentially impacting reproductive health, growth, and development. Several OCPs detected (e.g., heptachlor epoxide, endosulfan I and II) can bioaccumulate in human tissue, and because they are lipophilic, this can increase the risk of adverse health effects. Even low-level exposure can lead to harmful concentrations in body tissues over prolonged periods.

1. **CONCLUSION**

This study has significantly revealed the occurrence of organochlorine pesticides in herbal teas from Federal Capital Territory, Abuja markets. Notable OCPs that constantly violated the EU MRLs, includes some cyclodienes like endrin, ketone, heptachlor, aldrin and endrin ketone The occurrence of banned dichlorodiphenyltrichloroethane and benzene hexachlorides suggest inadequate controls on pesticide residues in agricultural inputs in the study area.

The presence of cyclodiene pesticides, chlordane, endosulfan, and DDT-related compounds at such levels may pose long-term health risks to consumers, given their persistence, bioaccumulation potential, and toxicity. Stricter regulations and routine monitoring are essential to control OCPs levels in herbal teas and other consumables. Enforcement of pesticide restrictions and post-market surveillance are critical to protect consumers

**DISCLAIMER (ARTIFICIAL INTELLIGENCE):**

Authors hereby declare that NO generative AI technologies such as Large Language Models (Chat PT, COPILOT, etc) and text to image generators have been used during the writing or editing of this manuscript

**REFERENCES**

Adenuga, A.A., Ore, O.T., Amos, O.D., Onibudo A.O., Ayinuola, O., & Oyekunle, J.A. (2022). Organochlorine pesticides in therapeutic teas and human health risk assessment. *Food Additives & Contaminants*: Part B. 2;15(4):301-9.

Aguilar-Pérez, K.M., Ruiz-Pulido, G., Medina, D.I., Parra-Saldivar, R., Iqbal, H.M., (2023). Insight of nanotechnological processing for nano-fortified functional foods and nutraceutical-opportunities, challenges, and future scope in food for better health. *Critical Reviews in Food Science and Nutrition* 7;63(20):4618-35.

Akande, M.G., Sanni, F.S., & Enefe, N.G., (2020). Human health risk evaluation of organophosphate insecticide residues in post-harvest cowpea in Gwagwalada, Abuja, Nigeria. *Journal of health and pollution.* 1;10(28):201203.

Alani, R.A., Nwude, D.O., Bello, I.I., Okolie, C.J., Akinrinade, O.E., (2023). Levels and Health Risks of Heavy Metals and Organochlorine Pesticide Residues in Soil and Drinking Water of Flood-Prone Residential Area of Lagos, Nigeria. Water, Air, & Soil Pollution. 234(12):783.

Ansari, I., E., l-Kady, M.M., Mahmoud, AED, Arora, C., Verma, A., Rajarathinam, R.K., Mittal, J., (2024). Persistent pesticides: Accumulation, health risk assessment, management and remediation: an overview. *Desalination and Water Treatment*, 100274.

Antoine, J.M., Fung, L.A., & Grant, C.N., (2017). Assessment of the potential health risks associated with the aluminum, arsenic, cadmium and lead content in selected fruits and vegetables grown in Jamaica. *Toxicology reports*. 1;4:181-7.

Authority, E.F., Cabrera, L.C., & Pastor, P.M., (2022). The 2020 European Union report on pesticide residues in food. EFSA Journal. 20(3). Ali U, Syed JH, Malik RN, Katsoyiannis A, Li J, Zhang G, Jones KC. Organochlorine pesticides (OCPs) in South Asian region: a review. *Science of the total environment*. 2014 Apr 1;476:705-

Barriga-Vélez, M. A., Ramírez-Vargas, L. C., Lopez-Barrera, E. A., & Peña-Rincón, C. A. (2023). Potential ecological risk index for metals in a grazing area, Guasca, Cundinamarca. *Revista Facultad de Ingeniería Universidad de Antioquia*, (106), 103-112.

Bhawan, F., Guidance document & standard operating procedures for fixation of maximum residue limits (MRLs) of pesticides in food commodities (2022). India: Food Safety & Standards Authority of India (FSSAI).

Chaudhuri, S., Are Medicine Prices High and Unaffordable after Trips? Evidence from Pharmaceutical Industry in India. Evidence from Pharmaceutical Industry in India (December 2019). Commentary on India’s Economy and Society Series-10. 2019.

Emoyan, O.O., Peretiemo-Clarke, B.O., Tesi, G.O., & Ohwo E., (2022) Occurrence, origin, ecological and human health risks of organochlorine pesticides in soils from selected urban, suburban and rural storm water reservoirs. Soil and Sediment Contamination: *An International Journal*. 17;31(2):152-75.

Elgueta, S., Valenzuela, M., Fuentes, M., Meza, P., Manzur, J. P., Liu, S., ... & Correa, A. (2020). Pesticide residues and health risk assessment in tomatoes and lettuces from farms of metropolitan region Chile. *Molecules*, *25*(2), 355.Fagbohun, A., Dauda, M.S., & Anjorin, T.S., Occurrence and Health Risk Assessment of Organochlorine Residues in Cowpea Grains Marketed in Abuja, Nigeria (2024). *Pesticide Science and Pest Control*. 3(1):2833-0943.

Fagbohun, A. A., Dauda, M., and Anjorin, T.S., (2023). A Review on Global Pesticide Use and Food Contamination: Africa Perspective. *Pollution*, (), -. doi: 10.22059/poll.2023.358732.1899

Fernandes, I.D., Maciel, G.M, Bortolini, D.G,, Pedro, A.C., Rubio, F.T., de Carvalho K.Q., & Haminiuk C.W., The bitter side of teas: pesticide residues and their impact on human health (2023). *Food and Chemical Toxicology.* 22:113955.

Hoque, M.Z., Akhter, N., & Chowdhury, M.S., Consumers’ preferences for the traceability information of seafood safety (2022). *Foods.* 7; 11(12):1675.

Huertas-Pérez, J.F., Baslé, Q., Dubois, M., & Theurillat, X., Multi-residue pesticides determination in complex food matrices by gas chromatography tandem mass spectrometry (2024). Food Chemistry. 15;436:137687.

Idowu, G.A., Aiyesanmi, A.F., & Oyegoke, F.O., Organochlorine pesticide residues in pods and beans of cocoa (*Theobroma cacao* L.) from Ondo State Central District, Nigeria (2022). *Environmental Advances*. 1;7:100162.

Kolakowski, B.M., Miller, L., Murray, A., Leclair, A., Bietlot, H., & van de Riet, J.M., (2020).Analysis of glyphosate residues in foods from the Canadian retail markets between 2015 and 2017. *Journal of agricultural and Food Chemistry*. 8;68(18):5201-11.

Kumar, N., Gupta, K.S., & Soni, R.K., Health risk assessment and toxicity management of organochlorine pesticide: With reference to Aldrin (2025). In Hazardous Chemicals 1 (pp. 43-55). Academic Press.

Lentini, P., Zanoli, L., Granata, A., Signorelli, S.S., Castellino, P., & Dellaquila, R., Kidney and heavy metals-The role of environmental exposure (2017). *Molecular medicine reports*.. 15(5):3413-9.

Liao, Y.; Berthion, J.M.; Colet, I.; Merlo, M.; Nougadère, A.; Hu, R. (2018). Validation and application of analytical method for glyphosate and glufosinate in foods by liquid chromatography-tandem mass spectrometry. J. Chromatogr. A 1549, 31–38

Lokesha, L.V., Sanganal, J.S., Shridhar, N.B., Rao, K.S., Narayanaswamy, H.D., Kumar, V.G., & Gowda, S.Y., (2017). Multi-residue analysis (gc-ecd) of some organochlorine pesticides in commercial broiler meat marketed in bengaluru city. *Journal of Experimental Zoology India*, *20*(2).

Mukasa, P., Wasswa J., Namuyomba P., & Ntambi E., Dichlorodiphenyltrichloroethane (DDT) and its metabolites in house dust, soil and selected food crops from indoor residual sprayed areas of Apac and Oyam Districts, Uganda (2022). African Journal of Pure and Applied Chemistry. 31;16(2):28-39.

Opuni, K.F., Asare-Nkansah S., Osei-Fosu P., Akonnor A., Bekoe S.O., & Dodoo A.N., Monitoring and risk assessment of pesticide residues in selected herbal medicinal products in Ghana (2021). Environmental Monitoring and Assessment. 193(8):470.

Oshatunberu, M., A., (2023). *Evaluation of Pesticide Residues in Grains Sold at Selected Markets of Southwest Nigeria* (Doctoral dissertation, Kwara State University (Nigeria)).

Soman, S., Rex, K. R., & Chakraborty, P. (2024). Environmental occurrence of pesticidal endocrine disrupting chemicals. In *Endocrine-Disrupting Chemicals* (pp. 147-168). Elsevier.

Taiwo, A.M., A review of environmental and health effects of organochlorine pesticide residues in Africa (2019). Chemosphere. 220: 1126-40.

Tarawneh, I.N., Polycyclic aromatic hydrocarbons and some of organochlorine pesticide residues and health risk assessments in commonly consumed teas in Jordan. Polycyclic Aromatic Compounds (2022). 26;42(10):7632-43.

Trivedi, D., Tanwar K., Kakodia A.K., & Jain M., Chlordane: Exposure, biohazard, current research, and precautions (2025). *Hazardous Chemicals*.1:119-35.

Verger, P.J., Risk analysis paradigm and total diet studies (2013). In Total diet studies (pp. 19-25). New York, NY: Springer New York.

Xie, F., Huang Q., Fang F., Chen S., Wang Z, Wang K., Fu X., Zhang B., Effects of tea polyphenols and gluten addition on in vitro wheat starch digestion properties (2019). *International Journal of Biological Macromolecules.* 126:525-30.

Yang, J., Pan, M., Yang, X., Liu, K., Song, Y., & Wang, S. (2022). Effective adsorption and in-situ SERS detection of multi-target pesticides on fruits and vegetables using bead-string like Ag NWs@ ZIF-8 core-shell nanochains. *Food Chemistry*, *395*, 133623.

Yigit, N., & Velioglu, Y.S., Effects of processing and storage on pesticide residues in foods (2020). *Critical Reviews in Food Science and Nutrition*. 60(21):3622-41.

Zhang, H., Qi R., & Mine, Y., The impact of oolong and black tea polyphenols on human health (2019). *Food Bioscience*. 29:55-61.

Zohora, K.F., & Arefin, M.R., (2022). Tea and Tea Product Diversification: A Review. *Turkish Journal of Agriculture-Food Science and Technology*. (12):2334-53.