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# Assessment of Pesticide Residues in Fish Tissues and Water at Various Time of the Year

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**Abstract**

The present study aimed to assess three pesticide residues (hexachlorobenzene, cypermethrin and chlorpyrifos) in the muscle of common carp (*Cyprinus carpio*), barbus grypus (*Arabibarbus grypus*) and water from Dukan Lake. About 60 samples were collected from different sites on the Dukan Lake in Iraq from different seasons (November 2023 to August 2024). Fish samples were analyzed fresh and grilled for the presence of pesticide residues using the QuEChERS method for extraction and water extraction was done by solid-phase extraction method accompanied by cleanup and detection using GC–MS (gas chromatography-mass spectrometry). The results revealed that the examined pesticides were identified in both types of fish muscles and water during different seasons, except hexachlorobenzene, which was not discovered in either fish type in the spring and in water during the summer. All pesticide residues were less than the maximum residue limit (MRL) in Japan, and just one sample exceeded the maximum residue limit in the European Union. Cooking (grilling) fish samples lowered pesticide residual levels to at least 25% of those measured in raw muscle. The trial findings indicated that eating common carp and barbus grypus and drinking water provides minor health hazards owing to residues of the three studied pesticides.

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## Introduction

One of the many inconsistencies in modern civilization is the use of pesticides to repel pests from plants. Pesticides are a diverse set of substances with established toxicity and catastrophic consequences for humans, animals, and the environment; nonetheless, they are widely employed worldwide in agriculture and health programs (Kumari *et al*., 2014). Pesticides harm a diverse range of non-target creatures, including aquatic invertebrates and fish (Burkepile *et al*., 2000). Pesticides are compounds that can pollute both aquatic and terrestrial habitats. Pesticides sprayed in one location can end up in the aquatic environment via drift, leaching, and drainage (Srivastava *et al*., 2010).

Fish can consume tainted food or directly absorb OCPs from the water. The area where pesticides build up in fish varies depending on the absorption pathway (Monirith *et al*., 2000). Furthermore, the quantity of OCPs in water is reflected in the amount found in the gills, whereas the liver stores all pesticides. Thus, fish may be one of the important bioindicators of pesticide contamination in freshwater environments (Rajendran *et al*., 2005; Ntow, 2005).

Since fish accumulate pollutants from aquatic environment, the habitat of aquatic species, eating them might be seen as one of the main causes of worry. Over time, pesticides bioaccumulate in aquatic creatures' tissue (Jiries *et al*., 2002; Yahia and Elsharkawy, 2014; Hassan *et al*., 2024).

Eating fish may be one of the main ways that people are exposed to environmental pollutants (Storelli, 2008; Yahia and Elsharkawy, 2014; Hassan *et al*., 2025). Aquaculturists may suffer financially due to the buildup of pesticides, which may lower fish quality in hatcheries and jeopardize their survival after release (Storelli, 2008; Botaro *et al*., 2011).

Understanding the dispersion and permanence of pesticides in the environment is necessary for their efficient and successful usage. In order to determine dispersion patterns during application and to assess their environmental fate, a significant portion of this knowledge is gathered through the collection and analysis of soil and water samples (Zidan *et al*., 1997). According to VanCuren (2003) and Maule *et al*. (2007) pesticides can attach themselves to the soil and then reach the aquatic environment by precipitation runoff or dust carried by the air. The current study's objectives are to evaluate the levels of three pesticide residues in fish tissues (common carp and barbus grypus) and water from Dukan Lake at various times of the year and investigate how heat treatment affects these levels.

## Materials and methods

### Ethics Approval

The scientific ethical committee of Animal Production Department, College of Agriculture, University of Tikrit, approved this study and give the Ethical Number 3/7/1517 (27/11/2023), all applicable national and international guidelines for the care and use of animals were followed.

### Sample collection

Water and muscle samples were collected from different sites on Dukan Lake in Iraq from different seasons (November 2023 to August 2024). The study and sample collection were conducted in Sulaimani, Iraq. The absence of the investigated pesticides was verified by testing the blank samples. Analyzed blank samples that were discovered to be devoid of target analytes were utilized as blanks and to create standard curves that matched the matrix. The remaining fraction was spiked and utilized for recovery, standard calibration, and sensitivity investigations after an aliquot of the samples was used for the selectivity research.

### Chemical and apparatus

The standards for pesticides were; hexachlorobenzene (HCb) (98%), cypermethrin (CMT) (94%), and chlorpyrifos (CPF) (96%), Merck Ltd. provided acetonitrile (ACN) HPLC grade, acetic acid (99.9%), primary secondary amine (PSA) with a particle size of 40 µm, octadecylsilane (C18, 50 µm), sodium chloride (NaCl), anhydrous magnesium sulphate (MgSO4), and syringe filters (0.45 µm). The injector, interface, and ion source were heated to 250 °C, and split less injection (1.0 min) was carried out using helium as the carrier gas at a rate of flow of 5 mL/min. The oven temperature was set at 120 and raised at a rate of 20°C per minute.

The injector's temperature was adjusted to 250 °C, and a splitless injection (1.0 min) was done using helium as the carrier gas at a flow rate of 5 mL/min. The oven temperature was set to 130 °C and raised at a rate of 10 °C/min for 2 minutes from 130 to 200 °C. The temperature was then raised from 32 °C/min to 270 °C for 3 minutes and kept for 4 minutes. The injection volume was 50 µL using a split-less injection mode (Sartarelli et al., 2012).

**Gas chromatography-mass spectrometry (GC-MS)**

Shimadzu gas chromatograph 17 A with an ECD (electron capture detection), the primary compound was separated. Column: Supelco Analytical Co., UK, supplied capillary columns, 30 m DB-5, having an internal diameter of 0.25 mm and a thickness of 0.1 µm. ECD electron-capture detector was used for detection (Sartarelli *et al*., 2012).

**Preparation of fish samples and extraction**

The extraction and cleanup processes were carried out using the original QuEChERS (rapid, easy, inexpensive, effective, robust, and safe) technique (Anastassiades *et al*., 2003) and (Lehotay *et al*., 2005): Before being used, fish samples were thawed overnight at 4 °C. Two grams of blank fish muscle samples were mixed and fortified with 20 µl of each of the following multistandard solutions: 75, 50, 25 and 5mg/kg (containing the three pesticides for 30 minutes). A 50 mL Falcon tube was filled with the combined meat samples. After adding 1.6 g of anhydrous $MgSO_4$, 0.4 g of NaCl, and 4 mL of ACN (containing 1% acetic acid), the mixture was stirred for one minute in a vortex mixer. To separate the phases, the mixture was centrifuged for three minutes at 3000 rpm (liquid-liquid partition). The organic solvent ACN-corresponding supernatant was put into a tube with 150 mg of $MgSO_4$, 70 mg of $C_{18}$ and 70 mg of the adsorbent PSA. After a 30-second handshake, the tube was centrifuged for one minute at 4000 rpm. To eliminate the extra coloring components, the supernatant was filtered using a syringe filter (0.45 µm). The filtered supernatant was kept at 4 °C after being evaporated at 45 °C under a nitrogen stream. Under ideal circumstances, the solutions were moved to an auto sampler vial and injected into the GC-ECD.

The following equation was used to quantify the residues of the three studied pesticides:

$$Concentartion\ calculation\ \left(\frac{\mu g}{kg}\right) = \frac{Area\ of\ sample}{Area\ of\ standard} * concetration\ of\ standard * Dilution\ factor$$

**Table 1. Retention times (minutes) and area µvolt for three studied pesticides**

| Seq. | Type of pesticide | Retention time minute | Area µvolt |
|---|---|---|---|
| **1** | Hexachlorobenzene | 3.032 | 1006356 |
| **2** | Chlorpyrifos | 4.43 | 913628 |
| **3** | Cypermethrin | 5.66 | 1042639 |

**Method of validation**

The globally recognized (SANTE, 2021) criteria-selectivity, recovery percentages, linearity, and sensitivity were followed in the validation process for this investigation. Five separate fish muscle sample extracts were injected into a GC-ECD detector to assess the method's selectivity. Each analyte's retention time window of interest was examined for the presence of interfering peaks above a signal-to-noise ratio of 3. Analytical standards (100 µg $kg^{-1}$) were injected into the GC-ECD apparatus individually in order to determine the target retention periods of the analytes. To enhance analytical validation and verify the range of retention time reproducibility, multi-standard solutions were introduced into the GC-ECD.

**Recovery of fish muscle**

By injecting multi-standard solutions containing three analytes at concentrations of 5, 25, 50 and 75 µg/kg for control matrices, recovery was assessed by comparing the obtained concentrations with the same concentrations of the pesticides prepared in the extraction solvent and analysing all spiked levels. To achieve the best technique reliability during screening, standard solutions were injected into blank matrices since each analyte in meat has a distinct maximum residue limit (MRL) (Nilsen, 2010).

**Recovery studies for water samples**

Distilled water samples were spiked with known amounts of pesticide and the spiked concentration sample and three blank samples were processed through the analytical method.

**Limits of detection (LOD) and quantitation (LOQ)**

Limits of detection and quantitation were determined based on a signalto-noise ratio, and concentrations showing peak intensity of signal-to-noise ratio of 3 and 10 were designated as LOD and LOQ, respectively (SANTE, 2021).

**Linearity**

The linearity test in fish muscle was performed by injecting five matrix-matched standards for calibration purposes. Calibration curves and individual stock solutions of HCB, CPF, and CMN were made in Pyrex glass vials with acetonitrile concentrations of 100 ug $kg^{-1}$ and kept at -20 °C in dark amber bottles. Diluting the stock solution in acetonitrile yielded working standard solutions (WSS) with concentrations ranging from 5-75 ug $kg^{-1}$. Solutions were injected into extracted blank samples to achieve concentrations of 0.10 for each chemical produced, and linearity was assessed using calibration curve coefficients ($r^2$) (SANTE, 2021).

**Solid-phase extraction (SPE)**

Solid-phase extraction (SPE) was used to remove pesticide residues from water samples (Kapsi *et al*., 2020):

1. Before analysis, the water sample was vacuum-filtered via GF/B (1 µm glass fibre filters; Whatman, UK) to eliminate any suspended solid and prevent any potential interference.
2. Ten milliliters of acetone, ten milliliters of ethyl acetate, ten milliliters of methanol, and ten milliliters of deionised water were used to prepare the C18 discs.
3. Using a vacuum manifold that keeps the pressure differential between the disk's input and output constant, the 1000 mL of water samples were run through the SPE discs before the disc dried up at a flow rate of around 10 mL/min.
4. Rinse the discs with 2 x 5 mL of deionised water once the entire sample has percolated.
5. To get rid of any remaining water, the discs were vacuum-dried for ten minutes.
6. Nine milliliters of ethyl acetate/dichloromethane (85:15) were used to elute the analytes drop by drop at a rate of one milliliter per minute.
7. The excess anhydrous sodium sulphate was used to dry the final.
8. A mild stream of nitrogen caused the methanol extracts to evaporate until they were completely dry.
9. Before chromatographic analysis, reconstitute in 0.1 mL methanol and store at -20 °C.

**Fat percentage**

Fat in fish samples was estimated based on the AOAC method (2002) using a Soxhlet fat extraction apparatus in the presence of hexane as an organic solvent, by heating fish meat samples for 16 hours, and fat was extracted according to the following equation:

$$Fat\ percentage = \frac{\text{weight of flask before extraction \_ weight of flask after extraction}}{\text{weight of sample}} \times 100$$

**Fish length**

Fish specimen's total, fork and standard lengths were measured and documented using the SAP method. To be in line with the measure used by the majority of US fisheries scientists, total length should be applied (EPA, 2000). The subsequent techniques for measuring fish length using a measuring board were adopted from the United States Geological Survey (2002).
Calculating total length:

1. Position the fish so that its head is on the observer's left, its mouth was closed, and its body was on the right board.
2. Press the nose, or front end, up against the stop of the measurement board from mouth to caudal fin.
3. Determine the overall length by measuring the distance between the closed mouth and the caudal fin's extreme tip while its lobes were compressed dorsoventrally, or unsprayed.
4. To the closest millimeter, note the entire length.

**Heat treatment**

Heat treatment (grilling at 180 °C) was carried out using a Binder oven, made in Germany, for the muscles of studied fish for 30 minutes (Al-Joumaa, 2010).

**Results and Discussions**

**Linearity range, regression equation**

Table 2 shows five calibration curves (5-75 µg $kg^{-1}$) for the chemicals evaluated. The integrated peak area was plotted against the concentration. To ensure the linearity of calibration graphs, the correlation coefficient ($R^2$) for each chemical was computed. The calibration data of the pesticides investigated revealed high linearity for the response of the ECD detector, with $R^2$ values ranging from 0.9874 to 0.9996.

**Table 2. Linearity range, regression equation coefficient**

| Pesticides | Linearity range (µg $kg^{-1}$) | Regression equation | $R^2$ | LOD (µg $kg^{-1}$) | LOQ (µg $kg^{-1}$) |
|---|---|---|---|---|---|
| Hexachlorobenzene HCB | 5-75 | Y=9877 X+15800 | 0.9993 | 14 | 42 |
| Chlorpyrifos CPF | 5-75 | Y= 9563 X+15062 | 0.9874 | 6.0 | 18 |
| Cypermethrin CMN | 5-75 | Y=10339 X+869.3 | 0.9996 | 14 | 42 |

**LODs, LOQs and Sensitivity**

As shown in Table 2, the LOD of studied pesticides ranged from 6 to 14 µg $kg^{-1}$ in fish muscles, and the LOQ ranged from 18 to 42 µg $kg^{-1}$ in fish muscle samples (Table 2).

**Recovery of fish muscle samples**

The accuracy of the employed analytical method was determined via the calculation of average percentage recoveries for studied pesticides and the percent relative standard deviation (RSD %) of recoveries from fortified blank samples of fish (Table 3). The average percentages of recoveries and the RSD% of recoveries at 75 µg $kg^{-1}$ level of pesticide standards ranged from 99.86 ± 1.3 to 118.46 ± 3.5% and 97.90 ± 3.2% from spiked muscle samples for hexachlorobenzene, chlorpyrifos and cypermethrin, respectively. At 5 µg $kg^{-1}$ level of the reference material, the recovery percentages ranged from 104.6 ± 2.2 to 105.86 ± 4.1% and 99.6 ± 3.2% from spiked muscle samples for hexachlorobenzene, chlorpyrifos and cypermethrin, respectively (Table 3).

The findings demonstrated that the examined pesticide recovery rates were greater than 90% for every tissue sample. These findings are in line with the manual's (SANTE, 2021) permitted recovery rates for pesticide residues. The recovery rate should be between 70 and 120 percent, as numerous researchers have suggested (Stoytcheva, 2011). Recovery rates were in line with research by Fang *et al*., (2024), which revealed that at a spiking level of 50 mg/kg, the tissue recovery rate of cypermethrin was 87.4%. At a dose of 30 µg $kg^{-1}$, the chlorpyrifos insecticide recovered at a rate of 81.32 ± 2.876% in normal carp tissues (Qayoom *et al*., 2024). For fish (*Heteroclarias sp*.), the recovery rate of the pesticide hexachlorobenzene at a spiked level was 11 (ng $g^{-1}$) (107.5%±3.8) (Lourencetti and Ricci, 2020).

**Table 3. Recovery of pesticides at different concentration spiked to blank compared with pure standard at 100 µg $kg^{-1}$ for fish tissue samples from Dukan Lake**

| Concentratio (n µg $kg^{-1}$) | HCB | | CPF | | CMN | |
|---|---|---|---|---|---|---|
| Added | Found | Recovery (%) | Found | Recovery (%) | Found | Recovery (%) |
| 75.0 | 74.899 | 99.86 | 88.1 | 118.46 | 73.43 | 97.90 |
| 50.0 | 49.92 | 99.84 | 52.99 | 105.9829 | 26.11 | 104.44 |
| 25.0 | 26.56 | 106.24 | 29.25 | 117.62 | 56.63 | 116.48 |
| 5.0 | 5.23 | 104.6 | 5.29 | 105.86 | 4.98 | 99.6 |

**Recovery percentage for water samples**

As can be seen in Table 4, from the 3 studied pesticides, there was an acceptable recovery percentage in the range from 70 to 120% for spiked levels 5, 25, 50 and 75 µg $kg^{-1}$, respectively.

Chloropyrifos level from water in El-Behera was 2.25 ± 0.44, in El- Qalyubia was 11.46 ±1.4 and in El-Dakahlia was 7.10 ± 2.49 ppb in El-Gharbia was not detected (Nazeeh *et al.,* 2024). The level of chlorpyrifos residues in water samples from river Challawa was 35 to 0.78 µg $kg^{-1}$, while the concentrations of cypermethrin ranged from 0.01 to 0.09 mg $L^{-1}$ (Akan *et al.,* 2014).

**Table 4. Recovery of pesticides at different concentrations spiked to blank compared with pure standard at 100 µg $L^{-1}$ for water samples from Dukan Lake**

| Concentratio (n µg/kg) | HCB | | CPF | | CMN | |
|---|---|---|---|---|---|---|
| Added | Found | Recovery % | Found | Recovery % | Found | Recovery% |
| 75.0 | 77.96 | 103.94 | 76.98 | 102.64 | 74.23 | 98.97 |
| 50.0 | 50.57 | 101.14 | 51.73 | 103.46 | 57.13 | 114.26 |
| 25.0 | 26.56 | 106.24 | 29.25 | 117.62 | 25.56 | 102.24 |
| 5.0 | 4.7 | 94 | 5.29 | 105.86 | 4.85 | 97 |

**Fish length**

The length (total, standard, and fork) of 40 samples of common carp and barbus grypus was summarized in Table 5. Total lengths for common carp samples ranged from 30.5±0.40 cm to 33.83±1.30 cm, while total lengths for barbus grypus samples ranged from 36.5±0.23 to 40±2.16 cm.

**Table 5. Mean of fish length (cm ± SE) samples were collected from Dukan Lake during different seasons**

| Seasons | Common carp | | | Barbus grypus | | |
|---|---|---|---|---|---|---|
| | **Total length** (cm) | **Standard length** (cm) | **Fork length** (cm) | **Total length** (cm) | **Standard length** (cm) | **Fork length** (cm) |
| Autumn | 31.5 ±0.47 | 28.16 ±0.59 | 26 ±0.47 | 40 ±2.16 | 37.16 ±2.29 | 34.66±1.9 |
| Winter | 30.5 ±0.40 | 27.33 ±0.36 | 24.83 ±0.36 | 39.16 ±0.49 | 36.66 ±0.72 | 34.5±0.62 |
| Spring | 30.33±1.20 | 27.5±1.04 | 25.33±0.93 | 36.5 ±0.23 | 32.66±0.27 | 31 ±0.0 |
| Summer | 33.83±1.30 | 30.63±1.35 | 28.16±1.17 | 38.5±1.80 | 35±1.89 | 32.6±2.04 |

**Fish weight and fat percentage**

Table 6 shows the weight of studied fish, weight of common carp from Dukan Lake ranged from 426±11.75 g to 523±7.55 g for different seasons, weight of barbus grypus ranged from 429±2.83g to 526±5.34 g for different seasons. The mean content of muscular fat ranged from 2.764±0.90 to 4.736 ±0.44% for common carp, while fat ranges for barbus grypus were between 2.354±1.01% to 3.701± 1.14 %. The lowest mean content of fat in the muscles of common carp and barbus grypus was in winter while the highest value in autumn.

The findings of Yeganeh *et al*. (2012) (1.5%-5.1%), Čirković *et al*. (2012) (2.42% fat), Afkhami *et al*., (2011) (3.53% fat), Čirković *et al*. (2011) (11.73% fat), and Marcu *et al*. (2010) (5.07% fat) were all consistent with the results of our investigation into the chemical composition of common carp. Regarding the fat content in the muscle of shabout fish in Dukan Lake that ranged from 2.354± 1.01% to 3.372±0.74 % for different seasons, there was a significant difference among winter season with other seasons, fat percentage in winter recorded lower percentage. During sexual maturation, so the final stages of gonadal growth are dependent upon the mobilization and re-allocation of endogenous reserves. In the White Sea bream (*Diplodus sargus*), muscular lipid was highly mobilized during the spawning period, presumably in support of the reproductive effort (Pérez *et al*., 2007).

**Table 6. Mean of fish weight (g ± SE) and fat % of muscle samples were collected from Dukan Lake during different seasons**

| Seasons | Common carp | | Barbus grypus | |
|---|---|---|---|---|
| | Weight (g) | Fat % | Weight (g) | Fat % |
| Autumn | 523 ±7.55 | 4.736 ±0.44 | 473 ±6.34 | 3.571 ±0.74 |
| Winter | 439 ±7.79 | 3.277±0.90 | 526± 5.34 | 3.893±1.01 |
| Spring | 426±11.75 | 2.154±0.31 | 429± 2.553 | 2.593± 0.12 |
| Summer | 442±12.45 | 5.135 ±0.70 | 436±11.68 | 4.224± 1.14 |

**Pesticide residues from fish muscles**

The residue concentrations of pesticides were detected in all seasons except hexachlorobenzene, which was not detected in spring from both fish species (Table 7). Barbus grypus exhibited the highest number of chlorpyrifos pesticide (7.679±2.55 µg $kg^{-1}$) in spring, however, the lowest value was detected in summer from common carp which was (0.488±0.39 µg $kg^{-1}$). Significant differences among seasons and types of were seen ($p \leq 0.05$).

The mean concentrations of chlorpyrifos in the muscle tissues of *S. melaanothern* were 0.0001±0.00002 mg $kg^{-1}$ in Etsii Lagoon and 0.0003±0.00003 mg $kg^{-1}$ in Fosu Lagoon, Ghana. The levels of pesticide residues found in the fish samples from Etsii and Fosu lagoons were all lower than those from their corresponding water samples (Essumang *et al*., 2009). The buildup of chlorpyrifos at such high concentrations in the muscles of carp juveniles may be due to the chemical's structural composition (Qayoom *et al*., 2024). Three chlorine atoms are present in the 2-ortho and 1-meta positions of the benzene ring. Consequently, the molecule is more like a compound of the organochlorine class than an organophosphate group. To put it another way, chlorpyrifos tends to accumulate in fish tissue at higher quantities than any other pesticide in its class and is more lipophilic than other organophosphates.

According to Zhao *et al*. (2011), who examined 30 fish tissue samples from a local fish market, one muscle sample tested positive for cypermethrin at 5.4 µg $kg^{-1}$ and one liver sample tested positive at 7.2 µg $kg^{-1}$ from crucian carp in China. Cypermethrin was discovered in seven of the 18 Canadian farmed salmon samples (39%), but not in any wild domestic salmon. Cypermethrin values in the positive samples varied between 0.3 and 6.5 ng $g^{-1}$. It was not found in any imported or domestically manufactured fish product (Rawn *et al*., 2010). Mahmoob *et al*. (2015) studied the concentration of cypermethrin pesticide in the muscle tissues of different weight groups of *Catla catla* in dry and wet seasons. The dry season concentration ranged from 0.29±0.06 to 3.45±0.10 ng $g^{-1}$, while the wet season's concentration ranged from undetectable to 2.85±0.36 ng/g.

Research has documented variations in HCB concentrations across quite narrow geographical areas. Because the East and West Coasts get air masses from various portions of North America, it has been demonstrated that the levels of HCB in Greenland polar cod are much greater on the East Coast than on the West Coast (Cleemann *et al*., 2000). According to Muir *et al*. (1990), burbot livers in high-latitude locations in Canada have greater quantities of HCB, which may indicate that cold condensation is taking place. Fish and shellfish in the Belgian North Sea had lower levels of HCB the further they were from Antwerp, suggesting a higher amount of exposure upstream, according to Voorspoels *et al*. (2004). Some of the Organochlorine pesticides were higher in the wet season in the water, gills and muscle compared to dry season. This could be due to increased farming activities in the wet season, since farming is one of the primary occupations of the people that live around the water body (Abubakar *et al*., 2024).

**Table 7. Detected pesticide residues (µg $kg^{-1}$ ± SE) in common carp and barbus grypus muscles in Dukan Lake during different seasons**

| Seasons | Common carp | | | Barbus grypus | | |
|---|---|---|---|---|---|---|
| | HCB | CPF | CMN | HCB | CPF | CMN |
| Autumn | 1.170±0.85 ab | 1.070±0.5 b | 3.533±0.50 ab | 2.087±0.96 ab | 3.180±0.45 ab | 4.033±0.19 ab |
| Winter | 4.761±0.87 ab | 2.871±1.09 ab | 3.267±1.41 ab | 5.815±1.82 ab | 1.894±0.82 ab | 1.397±0.34 ab |
| Spring | ND | 4.415±1.23 ab | 1.959±0.37 ab | ND | 7.679±2.55 a | 2.381±0.97 ab |
| Summer | 0.488±0.39 b | 0.806±0.38 b | 0.662±0.54 b | 2.657±1.07 ab | 2.629±0.71 ab | 1.356±0.55 ab |

ND= Not detected

**Pesticide residues from water**

The results of the determination of pesticides in the surface water samples collected from Dukan Lake were summarized in Table 8. Pesticides were detected in all seasons, except hexachlorobenzene was not detected in summer. There were significant differences ($p \leq 0.05$) among seasons and types of pesticides, the highest residues were in autumn for chlorpyrifos 2.793±0.97 µg $L^{-1}$ while the lowest average was detected in spring, which was 0.277±0.13 µg $L^{-1}$.

**Table 8. Detected pesticide residues (µg $L^{-1}$ ± SE) in water from Dukan Lake during different seasons**

| Seasons | Hexachlorobenzene (µg $L^{-1}$) | Chlorpyrifos (µg $L^{-1}$) | Cypermethrin (µg $L^{-1}$) |
|---|---|---|---|
| Autumn | 0.790±0.21 abc | 2.793±0.97 a | 2.170±0.22 ab |
| Winter | 2.291±0.46 ab | 1.099±0.48 abc | 0.478±0.23 bc |
| Spring | 0.277±0.13 bc | 1.025±0.42 abc | 0.445±0.24 bc |
| Summer | ND | 0.543±0.34 bc | 1.222±0.99 abc |

ND=Not detected.

**Heat treatment**

The effect of heat treatment on pesticide residues is presented in (Table 9). Grilled temperature reduced the pesticide residues by different percentages in both fish species and ranged from 26.01% to 43.39 % for three studied pesticides. Zabik *et al*. (1995) observed that heat treatment of Chinook salmon (Oncorhynchus tshawytscha) and common carp (*Cyprinus carpio*) reduced HCB, dieldrin, and total DDT residues by 30-41%. Hot smoking caused the greatest (over 50%) loss of HCB, total DDT, and chlordane complex in the muscle tissue of lake trout (Salvelinus namaycush namaycush) when exposed to various heat treatment procedures. In comparison, boiling or grilling were less successful, reducing pesticide concentration by 12-38 % on average (Zabik *et al*., 1996).

**Table 9. Effect of heat treatment (grilled at 180 °C for 30 minutes) on pesticide residues (µg kg$^{-1}$) in common carp and barbus grypus muscles in Dukan Lake during different seasons**

| Seasons | Types of fish | Type of pesticides | Raw muscle | Heat treated | Reduction rate (%) |
|---|---|---|---|---|---|
| Autumn | C. Carp | Hexachlorobenzene | 3.533±0.50 | 2.493±0.11 | 29.66 |
| | | Chlorpyrifos | 1.070±0.5 | 0.730±0.34 | 31.15 |
| | | Cypermethrin | 1.170±0.85 | 0.809±0.59 | 30.79 |
| | B. grypus | Hexachlorobenzene | 4.033±0.19 | 2.83±1.02 | 33.42 |
| | | Chlorpyrifos | 3.180±0.45 | 2.029±0.23 | 35.03 |
| | | Cypermethrin | 2.087±0.96 | 1.516±0.71 | 27.33 |
| Winter | C. Carp | Hexachlorobenzene | 3.267±1.41 | 2.41±1.01 | 26.34 |
| | | Chlorpyrifos | 2.871±1.09 | 1.950±0.78 | 31.93 |
| | | Cypermethrin | 4.761±0.87 | 3.055±0.48 | 35.82 |
| | B. grypus | Hexachlorobenzene | 1.397±0.34 | 1.033±0.85 | 26.01 |
| | | Chlorpyrifos | 1.894±0.82 | 1.322±0.57 | 32.82 |
| | | Cypermethrin | 5.815±1.82 | 4.117±1.02 | 29.18 |
| Spring | C. Carp | Hexachlorobenzene | 1.959±0.37 | 1.56±0.29 | 20.33 |
| | | Chlorpyrifos | 4.415±1.23 | 1.666±0.37 | 43.39 |
| | | Cypermethrin | ND | ND | _ |
| | B. grypus | Hexachlorobenzene | 2.381±0.97 | 1.555±0.64 | 34.58 |
| | | Chlorpyrifos | 7.679±2.55 | 5.343±1.45 | 30.41 |
| | | Cypermethrin | ND | ND | _ |
| Summer | C. Carp | Hexachlorobenzene | 0.662±0.54 | 0.431±0.35 | 34.87 |
| | | Chlorpyrifos | 0.806±0.38 | 0.533±0.26 | 31.94 |
| | | Cypermethrin | 0.488±0.39 | 0.348±0.28 | 28.50 |
| | B. grypus | Hexachlorobenzene | 1.356±0.55 | 0.792±0.32 | 43.54 |
| | | Chlorpyrifos | 2.629±0.71 | 1.850±0.22 | 30.14 |
| | | Cypermethrin | 2.657±1.07 | 1.546±0.35 | 41.79 |

ND= Not detected.

## Comparing pesticide residues in fish tissues with MRLs

Hexachlorobenzene, chlorpyrifos and cypermethrin were compared with pesticide MRL data for seafood from Japan and the European Union (EU), (Table 10). Japan and European Union have established standards for all three pesticides. Japan has set MRL for hexachlorobenzene, chlorpyrifos and cypermethrin (100, 300, 50 µ kg$^{-1}$), respectively. There were no samples that exceeded the Japan MRL. European Union has established a MRL for pesticides in fish; only one sample exceeded the EU MRL which was in winter and from b. grypus (11.964 µ kg$^{-1}$).

**Table 10. Comparison of pesticide residues from fish muscle with maximum residue limits (MRL) standard of pesticides in Japan and EU (µg kg$^{-1}$)**

| Seasons | Types of fish | Type of pesticides | Range (µg kg$^{-1}$) | MRL EU | MRL Japan |
|---|---|---|---|---|---|
| Autumn | C. Carp | Hexachlorobenzene | 0-6.19 | 10 | 100 |
| | | Chlorpyrifos | 0-2.13 | 10 | 300 |
| | | Cypermethrin | 0-3.25 | 10 | 50 |
| | B. grypus | Hexachlorobenzene | 3.55-4.28 | 10 | 100 |
| | | Chlorpyrifos | 2.08-3.89 | 10 | 300 |
| | | Cypermethrin | 0-4.07 | 10 | 50 |
| | | Hexachlorobenzene | 0-5.23 | 10 | 100 |

| | | | | | |
|---|---|---|---|---|---|
| Winter | C. Carp | Chlorpyrifos | 0.287-4.788 | 10 | 300 |
| | | Cypermethrin | 3.458-6.874 | 10 | 50 |
| | B. grypus | Hexachlorobenzene | 0-4.19 | 10 | 100 |
| | | Chlorpyrifos | 0-3.439 | 10 | 300 |
| | | Cypermethrin | 0-11.964 | 10 | 50 |
| Spring | C. Carp | Hexachlorobenzene | 0-5.876 | 10 | 100 |
| | | Chlorpyrifos | 0-8.830 | 10 | 300 |
| | | Cypermethrin | ND | 10 | 50 |
| | B. grypus | Hexachlorobenzene | 0-3.633 | 10 | 100 |
| | | Chlorpyrifos | 0-19.296 | 10 | 300 |
| | | Cypermethrin | ND | 10 | 50 |
| Summer | C. Carp | Hexachlorobenzene | 0-1.986 | 10 | 100 |
| | | Chlorpyrifos | 0-1.627 | 10 | 300 |
| | | Cypermethrin | 0-1.463 | 10 | 50 |
| | B. grypus | Hexachlorobenzene | 0-2.182 | 10 | 100 |
| | | Chlorpyrifos | 0-6.763 | 10 | 300 |
| | | Cypermethrin | 0-7.971 | 10 | 50 |

ND= Not detected, EU=European Union.

**Pesticide residues from water with MRLs**

The residue levels of the detected pesticides in this monitoring were found to be considerably lower than foreign standards and compared with Japan and USA MRLs (Table 11). Japan only established MRL for chlorpyrifos for mineral/packaged drinking water whereas USA established MRL for hexachlorobenzene (30 µg $L^{-1}$) and chlorpyrifos (50 µg $L^{-1}$).

**Table 11. Comparison of pesticide residues from water with maximum residue limits (MRL) standard of pesticides in Japan and USA (µg $L^{-1}$)**

| Seasons | Type of pesticides | Range | MRL USA | MRL Japan |
|---|---|---|---|---|
| Autumn | Hexachlorobenzene | 1.73-2.69 | 30 | - |
| | Chlorpyrifos | 1.35-5.43 | 50 | 30 |
| | Cypermethrin | 0.32-1.21 | - | - |
| Winter | Hexachlorobenzene | 0-0.987 | 30 | - |
| | Chlorpyrifos | 0.18-2.205 | 50 | 30 |
| | Cypermethrin | 1.41-3.298 | - | - |
| Spring | Hexachlorobenzene | 0-1.017 | 30 | - |
| | Chlorpyrifos | 0-1.659 | 50 | 30 |
| | Cypermethrin | 0-0.581 | - | - |
| Summer | Hexachlorobenzene | 0-3.664 | 30 | - |
| | Chlorpyrifos | 0-1.627 | 50 | 30 |
| | Cypermethrin | ND | - | - |

ND= Not detected.

**Correlation matrix (Pearson) among parameters**

Fish muscles from same sampling zone showed no statistical differences ($p \leq 0.05$) in each instance except the correlation among cypermethrin and chlorpyrifos. Correlation analysis revealed a positive correlation among hexachlorobenzene residue concentration, fish weight, fish length and fat percentage (Table 12).

According to Akor *et al*. (2021), lindane and p,p'-DDT were found to be positively correlated with fat in various organs of the sampled fish species, which supports the findings of Dang *et al*. (2016) who discovered that the bioaccumulation of certain types of contaminants in animal tissues is generally proportional to their lipid content. There was a high positive association between lindane and fat in catfish liver and muscle tissue, indicating a direct proportional link; hence, as fat levels grew, so did lindane accumulation in the liver and muscle tissue. This shows that excessive fat content is a significant contributor to lindane buildup in liver and muscle tissue, especially given the high overall prevalence rate of 63.8% in all samples analysed. This is consistent with the literature as stated by Helberg *et al*. (2005).

**Table 12. Correlation matrix (Pearson) among parameters**

| Variables | Fish weight | Fish length | Fat percentage | HCB | CMN | CPF |
|---|---|---|---|---|---|---|
| Fish weight | **1** | | | | | |
| Fish length | 0.391 | **1** | | | | |
| Fat percentage | 0.341 | 0.129 | **1** | | | |
| HCB | 0.193 | 0.151 | 0.127 | **1** | | |
| CMN | -0.050 | -0.062 | -0.001 | 0.128 | **1** | |
| CPF | -0.160 | 0.159 | -0.205 | -0.032 | **0.422** | **1** |

Values in bold are different from 0 with a significance level alpha=0.05.

## Conclusions

This study showed that pesticide residues were found in both fish tissues and water from Dukan Lake. However, the degree of pollution did not pose any significant health hazards to all of the investigated fish species. Nonetheless, there should be a public awareness campaign about the existence of these toxins in the Lake's fish. As a result, farmers in the study region should apply pesticides far enough away from the lake to safeguard aquatic life and the health of humans who consume polluted fish.

## Conflict of interest

The authors confirm that there are no conflicts of interest to declare regarding the publication of this paper.

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