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Full Length Research Article

QUANTITATIVE ANALYSIS OF PHARMACEUTICAL EMERGING CONTAMINANTS IN WATER AND FISH SAMPLES OF RIVER KADUNA

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ABSTRACT
The presence of pharmaceutical emerging contaminants (PECs) in natural waters has drawn the attention of many Scientists worldwide. Reports on the presence of these contaminants in different water systems continue to increase over the last few decades. This provokes worry about their potential negative implications to biodiversity and humans and their accumulation over a long period of time due to their ability to biotransform and thus breaks down into metabolites that can be more bioactive than the drug itself. Additionally, they can potentially create physiological effects in humans at small doses. This research involved preliminary investigations on disposal practices of expired drugs by pharmacists within Kaduna Metropolis. Water and fish samples from river Kaduna were collected and prepared for GC-MS analysis to detect the presence of PECs. Preliminary investigations on disposal practices of expired drugs by pharmacists within Kaduna metropolis revealed that about 60% of the pharmacists discard their expired products by either dumping in bin or burning; 20% reported to follow National (NAFDAC) guidelines while about 20% either refused to respond or were not aware about the disposal practices. The GC-MS results obtained for water sample revealed the presence of N-(3-methylbutyl) acetamide (335 g/L), acetic acid (81 g/L) and cyclopentaneundecanoic acid (140 g/L). Whereas in fish sample, 26-nor-5-cholesten-3-beta.-ol-25-one (400 g/kg), 1,3-benzene diol (160 g/kg), cyclopentaneundecanoic acid (170 g/kg) as well as N-(3-methylbutyl) acetamide (40 g/kg) were present. Some of the compounds found in relatively lower concentrations in the water sample were phthalic acid (27 g/L), guanidine (27 g/L), gluconic acid (17 g/L) and silver acetate (0.7 g/L) while in fish sample, hydroxylamine (3 g/kg), 1,5 heptadiene (12 g/kg) and silane (4 g/kg) were present. Most of the compounds detected are either esters, acids and alcoholic compounds. Studies on PECs in Nigeria are either ignored or limited especially in Northern part of the Country despite its occurrence in different locations and different environmental compartments with variations in concentrations. This research will create awareness and expose individuals and stakeholders to the potential negative effects of these contaminants. Pharmaceutical chemicals are very broad which include solvents, water, reactants and others. They are found in different environmental compartments. Many PECs are found in both the water and fish samples. Accumulation of these contaminants over time could be deleterious to life.

Keywords: Pharmaceuticals, emerging contaminants, fish and water sample. PECs, GC-MS.

INTRODUCTION

The socio-economic growth of a society is always determined by human health, food security, livelihood, economic independence, and preserved ecosystem; water quality is intrinsically linked with all these factors. In the late 1970th, a new threat to water quality emerges known as 'Emerging Contaminants (EC) or Contaminants of Emerging Concern (CECs) (Ebele *et al.,* 2020). Contaminants of emerging concern (CECs) are newly synthesized organic compounds or compounds previously not known to cause significant effects. These are compounds that are previously unmonitored or unregulated in the environment and they have the capacity to cause negative effects on human health and ecosystem (Geissen *et al.,* 2015). Most of the CECs came from synthetic chemicals which are crucial for modern societal development, examples include; perfluorinated compounds, water disinfection by-products, gasoline additives, pharmaceuticals, man-made nanomaterials, and UV-filters etc. (Gavrilescu *et al.,* 2015). Others include pesticides, artificial sweeteners, and hormones (UNESCO, 2015; Krettek, 2017). They are increasingly being used in many sectors and thus enter the environment as hazardous wastes and non-biodegradable substances (Murnyak *et al.,* 2009). They are also reported to be persistent and bioactive that are being transferred from one environmental compartment to another and their concentrations in the environment fluctuates widely which is attributed to different doses applied in various regions (James *et al.,* 2014; Gavrilescu *et al.,* 2015; Inam *et al.,* 2015; Krettek, 2017). Studies have shown that pharmaceuticals, personal care products, and endocrine-disrupting compounds are among the prime examples of CEC found in water (Inam *et al.,* 2015; Sui, 2015). In addition, it was discovered that pharmaceuticals ingested by humans are not fully processed by the body (NG, 2018). They often enter into the water system and cause adverse ecological and human effects through urine and feces (Pereira *et al.,* 2015). Pharmaceuticals can enter the surface water through human intake as medication, excretion in municipal wastewater, hospitals, landfills as well as wastes from pharmaceutical industry which are being discharged into a water body directly through the sewer or indirectly by leakage (Fent *et al.,* 2006; Ndidi *et al.,* 2011). It is worth noting that Pharmaceuticals emerging contaminants (PECs) are considered as a special group of CEC due to their potential to create physiological effects in humans in small doses (Ebele *et al.,* 2017). This is because they are designated to be biologically active

even at low doses and can also bio transform and thus, break down into metabolites which can be more bioactive than the drug itself (Anderson *et al.,* 2013; De Oliveira *et al.,* 2020). Pharmaceutical chemicals are very broad which include solvents (56%), water (32%), reactants (7%) and others (5%) (Cushman-Roisin & Cremonini, 2021).

In recent time, the presence of CEC in natural waters has drawn the attention of many scientists around the world because of the potential negative effects they have on aquatic ecosystem and human health (Krettek, 2017; Pereira *et al.,* 2017); which provokes worry about their potential negative implications to biodiversity and humans (Osuoha *et al.,* 2023). It is for this reason that UNESCO-IHP in 2018 provided a platform for the discussion on the impacts of water contaminants (CEC included).

Researchers in other part of the World such as US (Raghav *et al.,* 2013); Canada (Sauve & Desrosiers, 2014); Romania and Denmark (Gavrilescu *et al.,* 2015); Netherland (Geissen *et al.,* 2015); China (Sui, 2015); Spain (Eric *et al.,* 2017); Swedish (Krettek, 2017) as well as Ukraine (Vystavna *et al.,* 2017) have since explored the presence of PECs in the environment; but Africa is still lagging behind with about 60% of the studies on PEC carried out in South Africa, then Tunisia 14%, Zimbabwe 6%, Cameroon 5% followed by Nigeria with only 3% (Medikizeka *et al.,* 2020). Albeit (Ebele *et al.,* 2020); and (Bao *et al.,* 2015) have argued that the lack of economic prosperity in the developing countries is the major barrier to the studies on CEC but that is not the case in Nigeria being the largest economy in the Continent (Medikizeka *et al.,* 2020). The reason could be due to ignorance on the presence and effect of PEC or negligence (Ndidi *et al.,* 2011). There is the need for more studies in Nigeria having abundant natural water sources and being one of the worst hit Countries in Africa with cases of water related diseases (Alfarra, 2010; Galadima *et al.,* 2011) and yet little attention is paid to water quality. Even though (Ladan, 2012) has reported that the Nigerian environmental regulations on pharmaceuticals was created in 2009 but the enforcement of the regulation has been the major challenge. Therefore, the aim of this research is to quantitatively analyse the presence of PECs in water and fish samples of river Kaduna.

MATERIALS AND METHODS

Preliminary investigations

A preliminary investigation was carried out using questionnaires to determine how pharmacists/chemists discard their expired drugs within Kaduna metropolis. About fifty (50) pharmacists were interviewed and their responses were recorded.

Water Sampling and Preparation

Water samples were collected from three (3) different locations in river Kaduna and mix together to make a single (1) composite sample. The locations are Anguwan Dosa, Malali and Dan bushiya as indicated on the map of the study area. The collected sample was transported to the laboratory and analysed with 48 hours of collection. The sample was prepared according to the method developed by James *et al.* (2017) with slight modification. It was filtered using a 0.45 µm filter paper to remove particles and the filtrate was collected in a clean and oven dried container. In order to extract the target analyte, solid phase extraction was carried out; about 5mls of the filtered water sample and 5mLs of 10% methanol were measured and poured into the C18 cartridge. The sample (500mL) was then loaded into the cartridge at a rate of 10mL/min. 10mLs of 10% methanol was used as a wash solvent and subsequently, 5mL of 99.8% methanol was poured into the cartridge slowly at a rate of 2mL/min. The analyte fraction was collected and then taken for gas chromatography- mass spectrometry (GC-MS) analysis (Model: Agilent 19091S-433UI).

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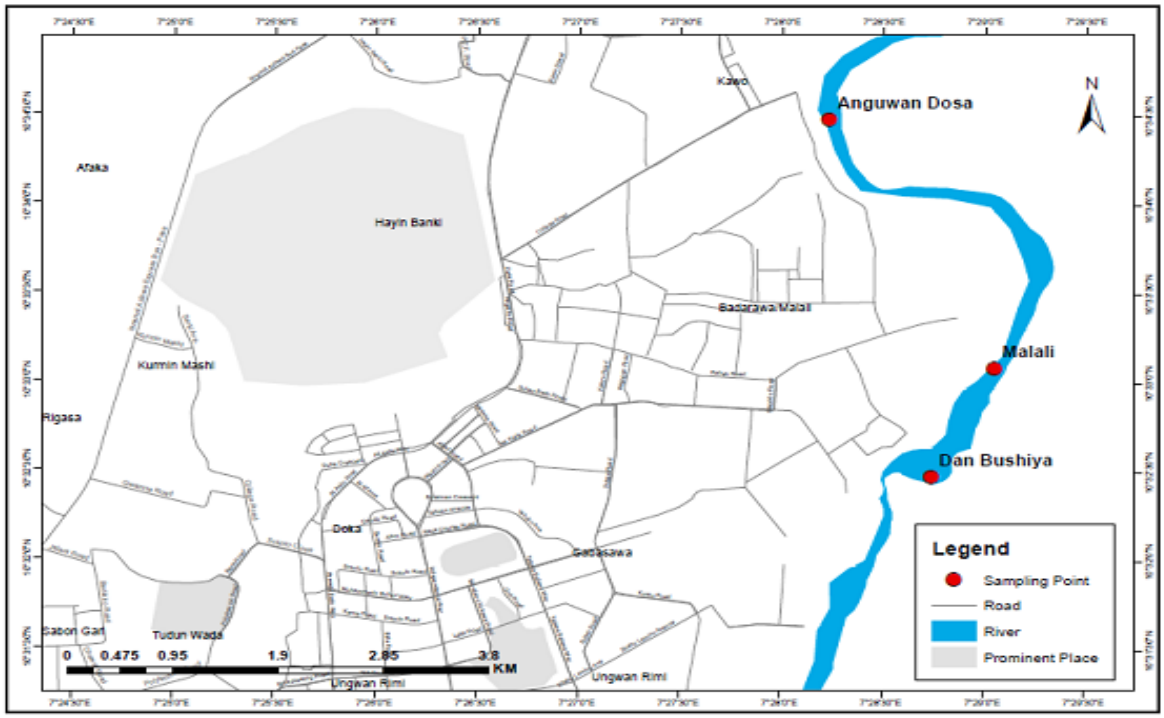

Figure 1: Map of the Study Area showing the sampling Locations

Fish Sampling and Preparation

Fish sample (Tilapia fish) was collected from river Kaduna by Fishermen. From each location, one fish was randomly selected to make a composite sample of three (3) sub-samples; it was immediately transferred to a water-full container and transported to the laboratory where it was frozen. The procedure according to (Mallauchos *et al.,* 2020) was adopted for the sample preparation. The frozen fish was cut immediately into pieces using knife, fins and inedible bones was removed and the remaining part was ground using blender to obtain a frozen powder.

About 0.5g of the sample was weighed into a centrifuge tube and dissolved in 500 µL methanol/water (1.5:0.5 v/v, HPLC grade), 50 µL of internal standard was added homogenized for 2 min at 20 Hz. Then, 200 µL of chloroform was added, and the homogenization repeated for 10 mins. The sample was centrifuged for 2 min at 12,000 rpm and separation was observed. The aqueous fraction was transferred in a glass autosampler vial. The sample was taken for gas chromatography- mass spectrometry (GC-MS) analysis and was analysed within 48 hours..

RESULTS AND DISCUSSION

Source of PECs in River Kaduna

Preliminary investigations on disposal practices of expired drugs by pharmacists within Kaduna Metropolis revealed that about 60% of the pharmacists discard their expired products through either dumping in bin or burning. Twenty (20) percent reported to follow National (NAFDAC) guidelines while about 20% either refused to respond or were not aware about the disposal practices. This study is similar to the study conducted by (Iweh *et al.,* 2019) in Anambra State Nigeria. They reported that only 23.4% complied fully with the National guidelines, 22.1% complied partially and larger percentage of 54.5% did not comply and therefore disposes their wastes through sinks, dumping etc. In a similar vein, thirty four (34) selected Nigerian based pharmaceutical manufacturers and major importers of medicines were interviewed to ascertain their waste management practices, knowledge of waste management policies and subjection to regulatory control. The study indicated that the wastes were poorly managed by 91.2% of the respondents, while 58.8% of the health and safety personnel had little or no modern knowledge of waste management. Furthermore, 73.5% of the respondents claimed that they were aware of the regulatory requirements on waste, but no adherence was observed. Most of the secondary manufacturers (79.4%) discharged wastewater without removal of pharmaceuticals. The researchers highlighted the urgent need on the management of waste which should be planned, documented, implemented and sustained (Ndidi *et al.,* 2011).

GC-MS for Water Sample

The GC-MS results for water sample indicated the presence of N-(3-methylbutyl)acetamide (335 mg/L), cyclopentaneundecanoic acid (140 g/L) and acetic acid (81 g/L). The chemical compounds N-(3-methylbutyl)acetamide also called iso-amylacetamide is a pheromone substance according to (Jimenez *et al.,* 2016). In addition, N-substituted acetamides are reported to have analgesic activity (Verma *et al.,* 2020) like the common drug (paracetamol) which is also a derivative of acetamide with the chemical name N-(4-hydroxyphenyl) acetamide. The presence of *N*-(3-methylbutyl) acetamide in autumn gynes of *Polistes metricus* Say, *Polistes bellicosus* Cresson, and *Polistes dorsalis* (F.), as well as workers of *Polistes aurifer* (Saussure), *P. bellicosus, P. metricus*, and *P. dorsalis* was reported in Florida, Georgia, South Carolina and Washington. It was revealed that *N*-(3- methylbutyl)acetamide attracted male and female *P. aurifer* and *P. metricus*, as well as male *P. dorsalis* and *P. bellicosus.* Thus, *N*-(3-methylbutyl)

acetamide may be a useful lure for trapping paper wasps in pest situations. Additionally, this compound *N*-(3-methylbutyl) acetamide was found to be venom in many female vespid wasps, including numerous *Polistes* species (Elmuquist *et al.,* 2020). Cyclopentaneundecanoic acid is reported to be under investigation according to (Wishart *et al.,* 2006). Acetic acid serves as an antimicrobial agent used to treat susceptible infections of the external auditory canal; it is also used as antibiotic to treat infections caused by bacteria and fungus (Wishart *et al.,* 2006).
Several other harmful compounds are found in the water sample such as; phthalic acid (27 g/L) which is an irritant, guanidine (27 g/L) is a flammable, corrosive and irritant used in manufacture of plastics and explosives, it is also used in the treatment of myasthenia and gluconic acid (17 g/L) is an electrolyte supplement used in total parenteral nutrition, formulation of pharmaceutical cosmetics.

GC-MS for Fish Samples

In fish sample, a cholesterol compound 26-Nor-5-cholesten-3-beta.-ol-25-one has the highest concentration of about 400 g/kg followed by cyclopentaneundecanoic acid (170 g/kg), 1,3-benzene diol (160 g/kg) and N-(3-methylbutyl) acetamide (40 g/kg). The compound 26-Nor-5-cholesten-3-beta-ol-25-one is a steroid compound with antimicrobial, anti-inflammatory, antioxidant, hepatoprotective, hypoglycemic, antipyretic and estrogenic activities (Ahmed *et al.,* 2019). This compound was previously found in Indonesian shallot grown in tidal swamp land (Galingging *et al.,* 2018). Another study by Ahmed *et al.,* (2019) revealed the presence of 26-Nor-5-cholesten-3-beta-ol-25-one in an illicit erection enhancer tablets (Hard on). 1,3-benzene diol is used as antiseptic and disinfectant in topical pharmaceutical products to treat skin disorders such as eczema and acne (Wishart *et al.,* 2006; NCBI, 2023b).

N-(3-methylbutyl) acetamide and cyclopentaneundecanoic acid were found in both water (335g/L and 140g/kg) and fish sample (40g/kg and 170g/L) respectively. This indicated the likelihood of these contaminants in human body through the food chain. Many other compounds are present in a very minute concentrations; accumulation of these contaminants over prolong period of time might have detrimental effect. For example hydroxylamine (3 g/kg), silane (4 g/kg) and 1,5 heptadiene (12 g/kg) are reported to be flammable and dangerous to the health (Wishart *et al.,* 2006). A review carried out by Khan *et al.* (2020) provides a critical understanding of fate and toxicity of pharmaceutical compounds and highlights their vulnerability and occurrence in South Asia. They concluded that aquatic life is more vulnerable to involuntary and continuous exposure of pharmaceutical compounds. Antibiotics, analgesics, and psychiatric drugs were found predominantly in the water environment of the regions.
Analysis on emerging contaminants in aquatic environment in Africa began about a decade ago in Zambia (Sorensen *et al.,* 2015). The study analyzed emerging contaminants in urban ground water in Kabwe and the results obtained were as follows: The insect repellent DEET was found to be 1.8 mg/L, bactericide triclosan (0.03 mg/L), chlorination by-products trihalomethanes (50 mg/L), and the surfactant 2,4,7,9-tetramethyl-5-decyne-4,7-diol (0.6 mg/L). Generally, personal care products, life-style compounds, and pharmaceuticals were not detected in the aquatic environment.
In the same year, the presence of veterinary drugs (Oxytetracycline, Tetracycline and Chloramphenicol) in fish pond was analyzed using Solid phase extraction-High performance liquid chromatography (SPE-HPLC) in Ogun State Nigeria. Chloramphenicol was found to have the highest concentration of 0.60 ng/mL, followed by Oxytetracycline 0.46 ng/mL while Tetracycline was found to be insignificant (James *et al.,* 2014). The authors recommended the establishment of modern wastewater treatment devices which can conveniently remove pharmaceuticals in water before they are discharged into the environment.
A research was also carried out by (Olarinmoye *et* al., 2015) to analyze surface water samples and sewage sludge samples (collected from waste water treatment plants in Lagos) for a range of different pharmaceuticals using liquid chromatography-tandem mass spectrometry (LC-MS/MS). About nine (9) different pharmaceutical substances were detected which include; ibuprofen, sulfamethoxazole, erythromycin, Betasitosterol, chloramphenicol, naproxen, ulfadiazine, trimetoprim and clofibrate. Among all, Diclofenac was found to be present in all samples at concentrations of up to 1100 µg/kg dry weight; this concentration appears to be the highest measured concentration in sludge samples worldwide.

Another research indicated that water samples (tap-water, well-water and river-water) obtained from Olabisi Onabanjo University Teaching Hospital Sagamu, Ogun state contained six pharmaceutically active ingredients. These include paracetamol, ibuprofen, diclofenac, ciprofloxacin, sulphadoxine, amodiaquine in varying low concentrations. The tap-water water samples contained paracetamol, ibuprofen, diclofenac, ciprofloxacin and sulphadoxine in concentrations of 0.306ng/mL, 3.738ng/mL, 0.138ng/mL, 0.44ng/mL and 1.012ng/mL respectively. The well-water samples contained paracetamol, ibuprofen, sulphadoxine and amodiaquine in concentrations of 0.152 ng/mL, 5.078 ng/mL, 1.008 ng/mL and 0.01892 ng/mL while the river-water samples were found to contain paracetamol, ibuprofen and sulphadoxine in concentration 0.192 ng/mL, 3.042 ng/mL and 1.294 ng/mL respectively. It is also reported that sulphadoxine and amodiaquine detected in this research have not been detected elsewhere in the world. They also recommended effective water treatment plants that can conveniently remove pharmaceuticals in water (James *et al.,* 2017).

To the best of our knowledge, higher concentration of pharmaceutical chemicals is observed in this research compared to the previous studies in Nigeria. This could be associated with either stagnation of the river as a result of dry season or inappropriate waste disposal practices. According to Qian *et al.* (2020), the global market for pharmaceutical industries is projected to increase by $ 0.75 trillion in 2023. As such, they emphasized the need to monitor ambient water sources and wastewater for pharmaceutical pollutants in order to safeguard human health and ecosystems.

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Table 1: GC-MS results of PECs in water and fish samples

S/N	Contaminant	Category	Concentration	Uses	Synonyms	Reference
	Water Sample					
1	N-(3-methylbutyl) acetamide	Nitrogen compound	335 g/L	Analgesic and pheromone substance	Iso-amyl acetamide, N-butyl ethanimidite	(Jimenez *et al.*, 2016)
2	Acetic acid	Acid	81 g/L	Antimicrobial and antibiotic	Ethanoate, vinegar and ethanoic acid	(Wishart *et al.*, 2006)
3	Cyclopentaneun decanoic acid	Acid	140 g/L	Under investigation	Dihydrohydnocarpic acid, 11-cyclopentylundecanoic acid	(Wishart *et al.*, 2006; NCBI 2023a)
	Fish Sample					
1	26-Nor-5-cholesten-3-beta-ol-25-one	Lipid (steroid)	400g/kg	Antimicrobial, antiinflammatory, antioxidant, hepatoprotective,hypoglycemic, antipyretic and estrogenic activities	Nil	(Gallingging *et al.*, 2018)
2	1,3-benzene diol	Alcohol	160 g/kg	Antiseptic and disinfectant in topical pharmaceutical products to treat skin disorder	Sodium salt, disodium benzene-1,3-diolate	(Wishart *et al.*, 2006; NCBI 2023a)
3	Cyclopentaneun decanoic acid	Acid	170 g/kg	Under investigation	Dihydrohydnocarpic acid, 11-cyclopentyl undecanoic acid	(Wishart *et al.*, 2006; NCBI, 2023a)
4	N-(3-methylbutyl) acetamide	Nitrogen compound	40 g/kg	Analgesic and pheromone substance	Iso amyl acetamide, N-butyl ethanimidite	(Jimenez *et al.*, 2016)

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Table 2: Sources, Concentrations and Locations of Different Types of Pharmaceutical Emerging Contaminants

S/N	Sample	Contaminants	Conc. Range	Location	Reference
1	Fish pond, waste water and river water	Chlorampenicol Oxytetracycline Tetracycline	0.46-0.60 ng/mL	Ogun, Nigeria	(James *et al.*, 2014)
2	Ground water	DEET Bactericide Triclosan Chlorination by products Surfactants	0.6-50 mg/L	Kabwe, Zambia	(Sorensen *et al.*, 2015)
3	Surface water and sewage sludge	Chlorampenicol Ibuprofen Sulpamethoxazole Erythomycin Betasitosterol Neproxen Ulfadiazine Trimeoprim Clofibrate	0.67-1100µ/kg	Lagos, Nigeria	(Olarinmoye *et al.*, 2015)
4	Well, tap and river water	Diclofenac Paracetamol Ibuprofen Ciprofloxacin Sulphadoxine Amodiaguine	0.01892-5.078 ng/Ml	Ogun, Nigeria	(James *et al.*, 2017)
5	Surface water	Triclosan Ibuprofen Diclofenac	0.10-1.54 µg/L	Gulf of Urab, Colombia	(Pemberthy *et al.*, 2020)
6	Ground water	DEET Bactericide Triclosan Chlorination by products Surfactants	0.6-50mg/L	Kabwe, Zambia	(Sorensen *et al.*, 2015)
7	Surface water Waste water Fish	Over 200 compounds	1-400ng/L (water sample), 0.5-150ng/g (Fish sample)	Albufera Lagoon, Spain	(Eric *et al.*, 2017)
8	Surface water	About twenty one (21) contaminants	-	Mid-eastern Sweden	(Krettek, 2017)
9	Water	Acetaminophen, trimethoprim, oxytetracycline and sulfamethoxazole	1.9-8.0 ng/L	Northern Coast of Central Java, Indonesia	(Hidayati *et al.*, 2021)
10	Spring	21 contaminants including tramadol, DEET and 1H-benzotriazole	0.3-50ng/L	Dinaric karst, Croatia	(Selak *et al.*, 2022)
11	Sewage sludge from Waste water treatment plants	250 emerging contaminants most of which are PECs	24.4-72.4 mg/kg	Lagos, Nigeria	(Nikolopoulou *et al.*, 2023)
12	Sea water	Trimethoprim, sulfadiazine, trimethoprim, sulfadiazine	0.02ng/L-0.11mg/L	Ionian sea, Greece	(Matinaiou *et al.*, 2022)

Key: Conc. range- Concentration range of the contaminants

Conclusion

Pharmaceutical chemicals are very broad which include solvents, water, reactants and others. These contaminants were found in both water and fish samples at relatively higher concentration; which could be the highest concentration found in both fish and water sample in Nigeria. There is no doubt that the methods employed in this study detected pharmaceutical contaminants in water and fish samples. There is need for incorporation of other detection methods such as LC-MS since GC-MS is more suitable for detection of volatile substances.

Declarations

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Conflicts of interest: Hafsat Tukur Rumah, Rilwan H.B., Hauwa'u Yakubu Bako and Abdullahi Maikudi Nuhu declare that they have no conflicts of interest.

Financial Interest: The authors declare that they have no financial interest.

Author's contribution statements: Methodology, investigation and Analysis, Hafsat Tukur Rumah; Investigation, editing and review, Rilwan H.B.; editing and review, Hauwa'u Yakubu Bako; Supervision, Abdullahi Maikudi Nuhu

Ethics approval: The questionnaire and methodology for this study was approved by Institutional based research and development committee (IBR) Kaduna State University Kaduna Nigeria. All participants provided their verbal informed consent.

Data availability statement: All the data used in this research are available in the supplementary information files. For any reasonable request, the corresponding author should be contacted.

Consent for publication: Not applicable

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