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Seasonal hot spots of pollution and risks in Western Kenya: A spatial-temporal analysis of almost 800 organic micropollutants

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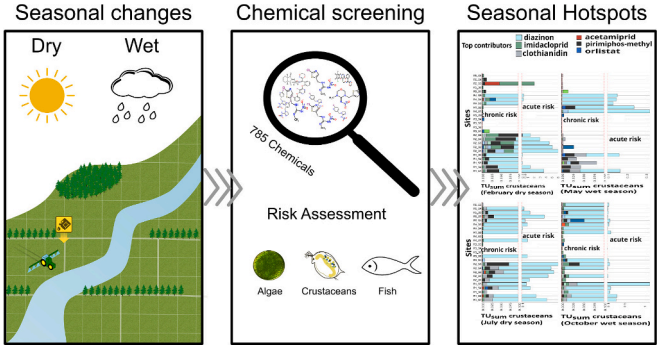
HIGHLIGHTS

- 307 chemicals identified, 18 proposed for regular monitoring and abatement.
- Forty-six chemicals detected in >50 % of the sites.
- Ecotoxicological risk on aquatic organisms driven by pesticides.
- Dry seasons pose high risk for crustaceans and algae.
- Seasonal variation of chemical occurrence and concentrations reported.

GRAPHICAL ABSTRACT



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ABSTRACT

The release of chemicals into the environment presents a significant threat to aquatic ecosystems dependent on the proximity to emission sources and seasonal dynamics of emission and mobilization. While spatial-temporal information on water pollution in Europe is increasing, there are substantial knowledge gaps on seasonal pollution dynamics in tropical countries. Thus, we took Lake Victoria South Basin in western Kenya as a case study to identify spatial and seasonal hot spots of contamination, quantified toxic risks to different groups of organisms, and identified seasonal risk drivers. For this purpose, we analyzed grab water samples from five rivers with agricultural and wastewater treatment plants in their catchment in four different seasons. We used liquid chromatography coupled to high resolution mass spectrometry (LC-HRMS) with a target list of 785 organic micropollutants. A total of 307 compounds were detected with concentrations ranging from 0.3 ng/L to 6.6 µg/L. Using a Toxic Unit (TU) approach based on mixture toxicity to standard test organisms, crustaceans were identified as the most affected group followed by algae and fish. For crustaceans, chronic risk thresholds were exceeded in 96 % of all the samples, while 56 % of all samples are expected to be acutely toxic, with the highest

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risk in February during the dry season. High toxic unit values for algae and fish were recorded in July dry season and May wet season. Diazinon, imidacloprid, clothianidin and pirimiphos-methyl were the major drivers for crustacean toxicity while triclosan and different herbicide mixtures drive risks to algae in dry and wet seasons, respectively. A total of 18 chemicals were found to exceed acute and chronic environmental risk thresholds. With this study, strong spatial-temporal patterns of pollution, risks and risk drivers could be confirmed informing prioritization of monitoring and abatement to enhance water quality and reduce toxic risks.

1. Introduction

Chemicals play an indispensable role in driving advancement and improving the quality of life. However, they also trigger adverse effects when released into the environment (Kosnik et al., 2022; Naidu et al., 2021; Persson et al., 2022). Their toxicity to aquatic organisms (Arslan et al., 2017; Malaj et al., 2014), persistence in the environment and bioaccumulation potential have attracted consequential attention (Zenker et al., 2014). Currently, >350,000 chemicals are registered for use (Muir et al., 2023; Wang et al., 2020), with over 50,000 classified as confidential (Feckler et al., 2023). These chemicals include pesticides, personal care products, biocides, pharmaceuticals, surfactants, food additives, and industrial chemicals (Morin-Crini et al., 2022).

At a continental scale in Europe, mixture toxic pressure by chemical pollution has been shown to be one of the major stressors that explain almost 30 % of the deviance of aquatic ecosystems from a good ecological status (Lemm et al., 2021). In Kenya, we could show that chemical pollution does not only threaten aquatic ecosystems but also impact on human health by paving the way for schistosomiasis transmission (Becker et al., 2020).

The dispersal of chemicals in the environment is aided by various factors including seasonal dynamics (Bloomfield et al., 2006; Fairbairn et al., 2016), agricultural land use (Giri and Qiu, 2016; Liess et al., 2021), improper waste disposals (Ferronato and Torretta, 2019), and discharge from industries and wastewater treatment plants (Fairbairn et al., 2016; Wen et al., 2017). Awareness is increasing that water pollution is not constant throughout the year but undergoes seasonal dynamics related to weather, hydrological and seasonal consumption patterns. Examples are the mobilization of chemicals from road runoff during snow melt (Maurer et al., 2023) and stormwater events (Hilliges et al., 2017), the mobilization of sediment borne pollutants by floods (Quesada et al., 2014), a lack of dilution during dry seasons (Kamjunke et al., 2022) and high pesticide loads in water during application seasons (Vormeier et al., 2023). Also, the fate of micropollutants may undergo seasonal dynamics due to its dependence on temperature and microbial activity (Anh et al., 2023; Barkow et al., 2021; Huang et al., 2020; Noyes et al., 2009). The consumption of some pharmaceuticals has been linked to specific seasons when disease occurrence and transmissions are high among the population (Abong'o et al., 2020; Katz et al., 2012; Ofulla et al., 2013). High concentrations of pharmaceuticals for flu medication during wet or cold seasons have been observed in many parts of the world such as Brazil (Corrêa et al., 2021), the U.S. (Fairbairn et al., 2016), China (Lin et al., 2018), Sweden (Rehrl et al., 2020), Portugal (Barbosa et al., 2018) and Canada (Pulicharla et al., 2022). The increased use and emission of disinfectants during pandemics of infectious diseases such as COVID-19 may be reflected by enhanced water concentrations (Dewey et al., 2021; Zheng et al., 2020). In countries with pronounced seasonal tourism, this may impact on water concentrations of recreational substances such as illicit drugs and caffeine (Maasz et al., 2019; Molnar et al., 2021). While seasonal pollution

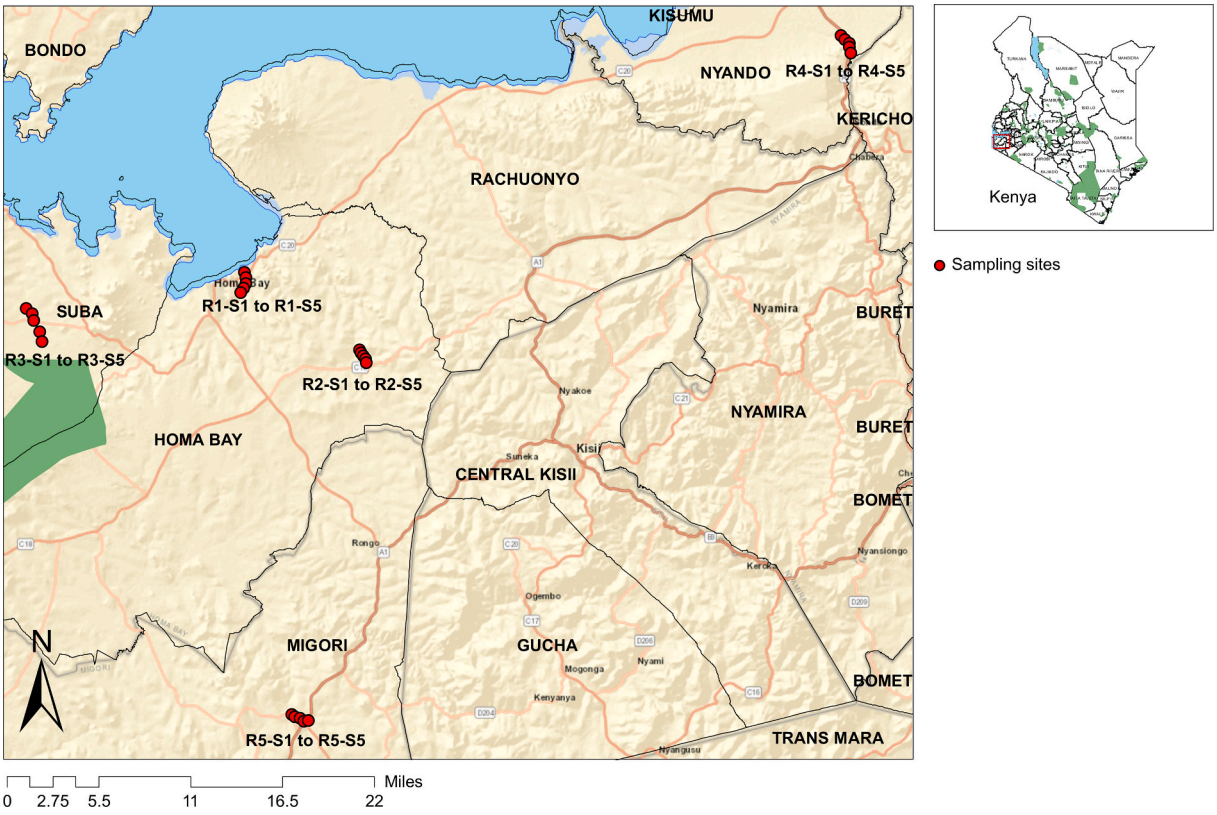

Fig. 1. Location of the study sites in western Kenya.

dynamics have received increasing attention in Europe including the investigation of seasonal variations of complex mixtures of micropollutants from wastewater discharges (Beckers et al., 2018), the knowledge on chemical pollution in general and seasonal dynamics in particular is very limited in many low-income countries with very different climatic and socioeconomic conditions.

In Africa and Kenya in particular, malaria and influenza infections among the local population peaks after the long rainy season in July to October (Katz et al., 2012). As a control strategy, indoor residual spraying with Actellic 300CS (pirimiphos-methyl) is usually undertaken before the peak malaria transmission to suppress the local mosquito populations (Abong'o et al., 2020). A study by Apopo et al. (2020) noted a peak in the numbers of Newcastle disease positive cases in chicken sampled from eight Kenyan agro-ecological zones during months with higher temperatures (March and August) compared with the cold season (June/July). As for the bacterial zoonotic disease brucellosis, different seasons contribute to different transmission rates. Dry seasons contribute to high transmission rates between domestic and wild animals due to sharing of pastures and water points, whereas wet season increase within-herd transmission due to a high birth rate and abortion storms (Nyerere et al., 2020).

Data on organic micropollutants in Kenyan water resources is slowly increasing though with limited focus on spatial and seasonal variability (Bagnis et al., 2020; Kandie et al., 2020a; K'oreje et al., 2016; Marete et al., 2020; Ndunda and Wandiga, 2020; Ngigi et al., 2020). Earlier investigations focused primarily on short-term studies (one-time sampling) targeting few compounds from specific compound classes (Getenga et al., 2004; Kimosop et al., 2016; Lisouza et al., 2020; Muriuki et al., 2020; Ngumba et al., 2016). Nevertheless, improving the knowledge base on water quality is of great importance due to the direct harmful effects on human health (Skidmore et al., 2023) and the indirect facilitation of zoonotic diseases such as Schistosomiasis (Becker et al., 2020).

The current study builds on this increasing knowledge of compounds, concentrations and sources of water pollution in Kenya and intends to close knowledge gaps on seasonal and spatial variations of chemical pollution in five rivers in Western Kenya through analyzing almost 800 organic micropollutants from different sources. Additionally, the study also assesses mixture composition and risks in dry and wet seasons. Mixture risk assessment is based on a toxic unit (TU) approach considering toxicity to three groups of model organisms including crustaceans, algae, and fish. Major seasonal risk drivers are identified and prioritized for monitoring and abatement. Unraveling spatio-temporal patterns and seasonal and spatial hot spots of risks may help to further prioritize environmental monitoring activities with limited resources.

2. Materials and methods

2.1. Chemicals

LC-MS grade water was obtained from Thermo-Fisher, ammonium formate, formic acid and LC-MS grade methanol was purchased from Honeywell. Ethyl acetate (LC-MS grade) was obtained from Sigma-Aldrich. Analytical standards were bought from various suppliers all with high purity (Above 97 %). More information on internal standards and analyzed compounds is provided in the Supplementary Information B (Table B.1).

2.2. Study site, sampling and sample preparation

The study was conducted in the Lake Victoria South Basin within western Kenya, covering Migori, Kisumu and Homabay counties (Fig. 1).

The region experiences two wet and two dry seasons in a year. Wet seasons typically last from March to May, and from October to December while January and February are the driest months (Evans et al., 2020). Previous studies conducted in the region reported the occurrence of chemicals encompassing a broader spectrum of pesticides, pharmaceuticals and personal care products (Kandie et al., 2020a), endocrine disrupting compounds (Ngeno et al., 2023) and industrial compounds (Kandie et al., 2020b). Five rivers were selected from the region, sampled and analyzed for organic micropollutants in dry and wet seasons. Rivers Rang'wena (R1), Rang'we (R2) and Lambwe (R3) are situated in Homabay County, while rivers Asao (R4) and Sare (R5) are in Kisumu and Migori Counties respectively (Fig. 1). Based on the national census of 2019, the region's population was approximately 3.5 million people (KNBS, 2019) and largely depend on the rivers as a primary source of water. Maize, rice, beans, sugarcane, cotton, sorghum, collard-greens (*Sukuma wiki*), spinach, cabbage, tomatoes, onions, carrots, capsicum, eggplant, peas, and sweet potatoes are major crops grown in the region. Despite the population's reliance on the rivers, the region struggles with sanitation facilities and infrastructure. As a result, water pollutants from rural areas, urban areas and agricultural fields find their way into the rivers through run-offs and improper disposal. Pollution dynamics of mixture composition and chemical concentrations in rivers are expected to undergo seasonal dynamics due to the seasonality of land-use practices, consumption and behavior of the population as well as to hydraulic dynamics.

Sampling was conducted between July 2021 and May 2022 with July 2021 and February 2022 samples representing the dry seasons while October 2021 and May 2022 samples represented wet seasons. Twenty-five sampling sites were identified in the five rivers per season which translated to a total of 100 samples for the entire study period. Prior to sampling, sampling locations were identified by measuring five equidistant points of 500 m apart from each point, along each river. Sites identified were marked as S1, S2, S3, S4 and S5. From each site, 350 mL grab water was sampled for solid phase extraction (SPE) complemented by 1-mL aliquots collected in 2-mL amber autosampler glass vial (Phenomenex, Germany) for direct injection into the LC-HRMS instrument to cover hydrophilic compounds with low SPE recovery. In addition, 125 mL of sample water was collected in Nalgene high density polyethylene (HDPE) bottles for physical-chemical analysis. Samples were stored at −18 °C in a portable freezer and transported to the laboratory for further processing and analysis. More information on sampling is provided in Supplementary information A (Section 1).

Sample aliquots of 350 mL were filtered through Whatman GF/F 50 mm filters using the Duran vacuum filtration system. The sample was solid-phase extracted using HR-X (Macherey Nagel) cartridges containing 200 mg of sorbent (hydrophobic polystyrene divinylbenzene copolymer). Cartridges were eluted into 20 mL amber glass vials using 5 mL of ethyl acetate, 5 mL of methanol, 4 mL of acidified methanol (with 1.0 vol% high purity formic acid) and 4 mL of methanol with 2.0 vol% of 7 N ammonia in methanol (all LC grade, Sigma Aldrich). After elution, each sample was evaporated under a continuous stream of nitrogen (99 % purity) to a volume of 1 mL. This extract was filtered through a 0.2 μm PTFE syringe filter (Whatman) into a 2 mL amber glass, evaporated to dryness using a gentle stream of nitrogen (XcelVap) and reconstituted with 350 μL of LC-MS grade methanol (enrichment factor (EF) of 1000) and vortexed for 2 min. Detailed information on Solid Phase extraction is provided in Supplementary information A (Section 2).

2.3. Instrumental analysis

2.3.1. Solid phase extracted and direct injection samples

From samples prepared by solid phase extraction, samples for LC-HRMS analysis were prepared by taking 50 μL from the enriched sample (EF1000), 15 μL of methanol, 30 μL of water and 5 μL of internal standard (1 μg/mL) containing 48 isotope-labelled compounds (Supplementary Information B, Table B.1). Instrumental blanks were prepared using methanol and water in a ratio of 70 to 30. Matrix-matched calibration standards were prepared from a water sample collected from a pristine stream (Wormsgraben) from the Harz Mountain, Northern

Germany. Eleven aliquots of 1 L were spiked at levels ranging from 0.5 to 1000 ng L^{-1} and subjected to the same solid-phase extraction procedure as the samples.

Aliquots of 5 μL were injected into Thermo Ultimate 3000 LC system coupled to a QExactive Plus high-resolution mass spectrometer (Thermo). Separate runs were performed in negative and positive electrospray ionization modes. For chromatographic separation in both modes a Kinetex Biphenyl LC column (100 × 2.1 mm, 2.6 μm particle size Phenomenex) equipped with inline filter and pre-column of the same type (5 × 2.1 mm) at a temperature of 40 °C was used. For positive ion mode a gradient separation using 0.1 % formic acid/methanol (containing 0.1 % formic acid)/acetonitrile was used, and for negative mode 1 mm ammonium fluoride/methanol (containing 1 mM ammonium fluoride)/acetonitrile. Details of the LC gradient are given in the Supplementary Information A (Table A.1). The HRMS analysis combined a full scan acquisition (*m/z* 80–1200) at a nominal resolving power of 70,000 with six data independent acquisition scans (*m/z* 80–182, 178–282, 278–382, 378–482, 478–682, 682–1200) at a nominal resolving power of 35,000.

For direct injection samples, samples were prepared for LC-HRMS analysis by adding 25 μL of the internal standard solution (40 ng/mL), 10 μL of 2 M ammonium formate buffer (pH 3.5) and 25 μL of LC-MS grade methanol. Instrumental blanks were prepared in water and methanol at a ratio of 95 to 5. Nineteen sampling blanks and three processing blanks were also prepared the same way as the samples. An aliquot of 100 μL was injected and analyzed on both negative and positive electrospray ionization modes on separate runs as described above in this Section. Thirteen calibration standards ranging from 1 ng/L to 10,000 ng/L were prepared by spiking the respective concentration into 1 mL aliquots of pristine water obtained from the Wormsgraben stream.

2.3.2. Target list of compounds

The target list comprised of 695 compounds for SPE and 90 compounds for direct injection (see Supplementary Information B, Table B.2 and Table B.3 respectively) which were selected according to previous analyses in Kenya (Kandie et al., 2020a, 2020b) and Europe (Finckh et al., 2024). The list comprised a variety of compound classes categorized based on their use and included pharmaceuticals, industrial compounds, biocides and pesticides, insecticides, corrosion inhibitors, flame retardants, food ingredients, per-fluorinated compounds plastic additives, repellents, rubber additives, sweeteners, stimulants, UV filters, cosmetics and surfactants.

2.4. Data processing analysis and visualization

The LC-HRMS raw data was converted to mzML format using ProteoWizard version 3.0.18265 (Kessner et al., 2008) and then processed sequentially in MZmine 2.38 software using peak picking, deconvolution, alignment, gap filling, and peak annotation stages (Finckh et al., 2022; Pluskal et al., 2010). A custom database was used to annotate the target compounds. A CSV file of the annotated compounds was exported from MZmine and the concentration of individual compounds determined using MZquant R package version 0.8.3 (Schulze et al., 2021). Compounds which could not be quantified properly using MZquant package were checked using Tracefinder version 5.1 (Thermo Scientific). For quality assurance, method detection limits (MDLs) were calculated by replicate injection of calibration standards based on (US-EPA, 2016), as applied by Finckh et al. (2022a), Kandie et al. (2020a) and Maurer et al. (2023). MDLs used for the detected compounds in the current study and the SPE method recoveries (percentage) are provided in the Supplementary Information B, (Table B.4). Analysis and visualization of the data was performed using the *ggplot2* package (Wickham, 2016) in R (version 4.2.2) and Microsoft Excel KNBS, 2019. Prior to clustering, data was scaled and log transformed to minimize skewness. Spatial and temporal patterns (clusters) of sampling sites and detected compounds were presented in a heatmap generated using heatmap.2 function in R package 'gplots' (Warnes et al., 2022). Analysis of Variance (ANOVA) was conducted using the 'aov' function from the `stats` package in R (Wickham and Wickham, 2016) to determine the difference in detected compound concentrations across seasons and the study sites.

2.5. Chemical mixture risk assessment

Risk assessment of detected chemicals was carried out using the toxic unit (TU) approach (Finckh et al., 2022; Machate et al., 2023; Maurer et al., 2023) Compound TU values were obtained by dividing the measured environmental concentration of a chemical (MEC) by its effect concentration (LC_{50} or EC_{50}, concentrations that affect 50 % of organisms in an acute toxicity test) for a standard test organism (TU_{algae}, TU_{fish}, $TU_{crustaceans}$), as shown in Eq. 1.

$$TU_{BQE,i} = \frac{MEC_i}{EC_{50,BQE,i}} \quad (1)$$

BQE refers to algae, crustaceans and fish defined as biological quality elements (BQEs) in the European Water Framework Directive. EC_{50} and LC_{50} values were obtained from the ECOTOX database (Kramer et al., 2024; US-EPA, 2023). These values are obtained from experimental data and represent 5th percentile of effect concentration values (EC_{50}) obtained per BQE. In cases where experimental data was lacking, predicted EC_{50}/LC_{50} values from Ecological Structure Activity Relationships model (ECOSAR) were utilized (UFZ Department of Ecological Chemistry, 2021). Mixture risk was assessed using the concentration addition (CA) model adding toxic units for algae, crustaceans and fish, respectively, of all compounds found in a mixture yielding TU_{sum} (Eq. 2).

$$TU_{sum} = \sum_{i=1}^{n} TU_i \quad (2)$$

2.6. Prioritization of detected compounds for monitoring and abatement

Compounds were prioritized based on the exceedance of the acute and chronic risk thresholds as proposed by (Malaj et al., 2014) and applied by (Maurer et al., 2023) and (Kandie et al., 2020a). Chronic risk thresholds for crustacean (daphnia), fish and algae were 0.001 TU, 0.01 TU and 0.02 respectively while acute toxic risk was 0.1 TUs for all organisms. Prioritization relied on the following indicators:

i) Frequency of exceedance of the risk thresholds (Eq. 3) where N is the sum of all the sites sampled and n is the number of sites where TU of a specific compound exceeded the risk threshold.

$$\text{Frequency of exceedance} = \frac{\sum n}{N} \quad (3)$$

ii) The extent of exceedance of the compounds addressing the maximum fold exceedance of the risk thresholds

To obtain the extent of exceedance values (TU_{max}) was normalized to the respective threshold per compound across all the sites (Eq. 4).

$$\text{Extent of exceedance} = \frac{TU_{max}}{\text{Threshold value}} \quad (4)$$

The results were then scaled up from 0 to 1. The results from the two indicators were added together to create a priority score, which was subsequently employed to rank the compounds.

3. Results and discussions

3.1. Detection frequencies

In total, 307 compounds comprising of pharmaceuticals, pesticides,

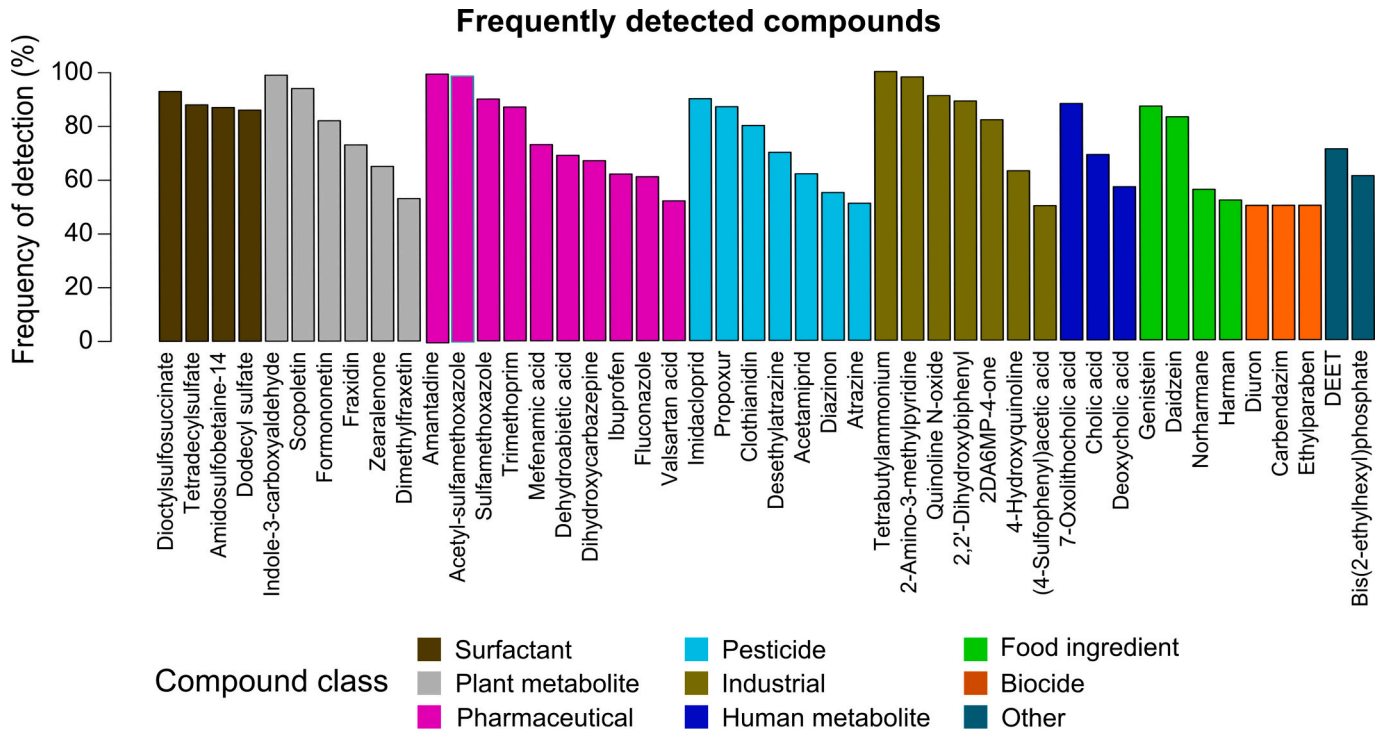

Fig. 2. Compounds detected in >50 % of the sites, detection frequencies arranged according to compound classes.

personal care products, biocides, pharmaceuticals, and industrial chemicals were found with detection frequencies ranging from 1 % to 100 % (Supplementary Information B, Table B.4).

Forty-six compounds were detected in >50 % of the sites and included particularly pesticides (diazinon, imidacloprid, propoxur, clothianidin, desethylatrazine, diuron, carbendazim, ethylparaben acetamiprid, atrazine) and pharmaceuticals (sulfamethoxazole, trimethoprim, mefenamic acid ibuprofen and fluconazole). Amantadine and tetrabutylammonium were detected at all the sites and all the seasons (100 % detection) (Fig. 2).

Amantadine is an antiviral pharmaceutical used for the treatment of influenza viruses and movement disorders (Peng et al., 2018; Zhao et al., 2023). In Kenya, influenza is prevalent throughout the year with spiking cases of the infection observed during the wet seasons (Matheka et al., 2013). Tetrabutylammonium is a quaternary ammonium compound (QACs) used as an ingredient in the manufacture of disinfectants and biocides, it is also used in paints and coating additives (Vereshchagin et al., 2021). The demand for these compounds escalated in response to the COVID-19 pandemic (Arnold et al., 2023). We also detected anticancer (etoposide up to 26 ng/L), anti-HIV drugs (efavirenz up to 16 ng/L) and chemicals which have been banned from use in the EU (bentazone, up to 4.6 ng/L, imidacloprid up to 3.7 μg/L, atrazine up to 72 ng/L, and metolachlor up to 26 ng/L).

3.2. Compound concentrations

A list of the individual compound concentrations is included in Supplementary Information B, (Table B.4). A total of 80 pesticides and biocides were detected with concentrations ranging from 0.4 ng/L to 3.7 μg/L. The highest measured concentration was obtained for the pesticide imidacloprid (3.7 μg/L), which is a synthetic neonicotinoid frequently used in Kenya for controlling aphids, thrips, leafhoppers and whiteflies in horticultural crops (PCPB, 2018). Similar water concentrations of the imidacloprid were reported in South Africa (Curchod et al., 2020).

Sixty-seven pharmaceuticals were detected with concentrations ranging from 0.35 ng/L to 2 μg/L. The highest concentration was detected for oxypurinol (detected up to 2 μg/L), a pharmacologically active metabolite of allopurinol drug. In Kenya, allopurinol is used for the treatment of gout and hyperuricemia (Kirigi et al., 2010). Acetaminophen and ibuprofen were detected at concentrations of up to 0.52 μg/L and 71 ng/L, respectively. Both compounds are used as a primary drug for managing chronic and acute pain and are sold over the counter (Ohashi and Kohno, 2020). Similar concentrations of ibuprofen and acetaminophen were found in Nairobi river (Waleng and Nomngongo, 2022).

A total of 21 plastic and rubber additives were detected with concentrations ranging from 0.6 ng/L to 530 ng/L. Phenyl-phosphate, which is a transformation product of triphenylphosphate used in the manufacture of flame retardants such as triaryl phosphates (Stapleton et al., 2009), was detected in concentrations up to 530 ng/L. Phenyl-phosphate is also used in fertilizers, pesticides, and many household products. Additionally, bis(2-ethylhexyl), a transformation product of tris(2-ethylhexy) phosphate was detected with a maximum concentration of 10 ng/L. Four rubber additives were detected for the first time in Kenya, hydroxybenzothiazole (14 ng/L), dicyclohexylurea (9 ng/L), 6PPD-quinone (1 ng/L), and 1-cyclohexyl-3-phenylurea (1 ng/L). 6PPD-quinone is a transformation product of N-(1,3-dimethylbutyl)-*N*′-phenyl-*p*-phenylendiamine (6PPD), an antioxidant used in tires. These compounds reach the environment via road runoffs during rainfall events. 6PPD-quinone has been identified as highly toxic to specific fish species (Brinkmann et al., 2022) and the driver of coho salmon kills in the U.S. (Tian et al., 2021). Due to the worldwide use of 6PPD in tires, 6PPD-quinone is expected to be a global scale road runoff contaminant. Recently, its occurrence has been confirmed in urban snow melt and road runoff in Germany (Maurer et al., 2023; Seiwert et al., 2022).

Industrial compounds were detected in most of the sites with concentrations ranging from 0.4 ng/L (*N*-methyl-dicyclohexylamine) to 6.7 μg/L for the surfactant dodecyl sulfate used in large volumes in laundry detergents, soaps and shampoos and enter the river via washing of clothes and other utensils and bathing in the river. Six human metabolites and thirteen plant metabolites were detected with concentrations ranging from 1 ng/L (gramine) to 1700 ng/L (cholic acid). Cholic acid is a bile acid secreted in human faeces making it a suitable biomarker for

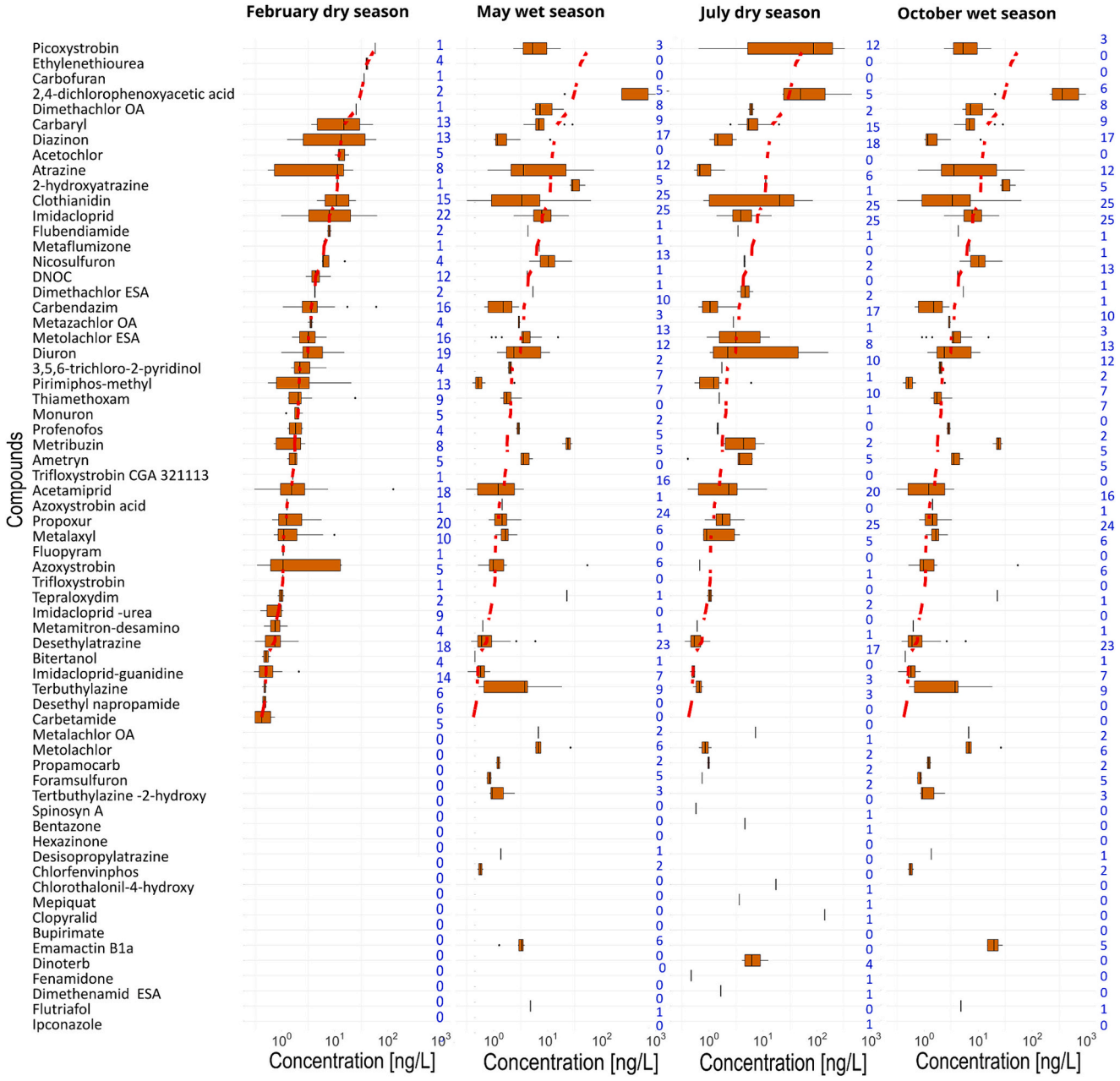

Fig. 3. Box plot summary showing concentration of detected pesticides across four seasons. The centre dark line in the box-plot represents the median concentration of the compound and the hinges represent the 25th and 75th percentiles. The upper and the lower whiskers represent the interquartile range. The values at each boxplot represent the number of compounds generating the boxplots. The dotted red line cutting across the boxplots from the compound with the highest median concentration to the compound with the lowest median concentration in February dry season indicates whether the median concentrations of individual compounds in the subsequent seasons (May, October, July) increased or reduced. DNOC: dinitro-ortho-cresol.

wastewater contamination (Bull et al., 2002). The detection of this compound in surface water systems raises particular concern on the hygienic quality of river water in western Kenya often used as drinking and bathing water. Zearalenone, a mycotoxin produced by the *Fusarium* fungi and which has adverse effects on animal and human health (Ropejko and Twarużek, 2021) was also reported with concentrations up to 91 ng/L. Runoffs from maize fields along the rivers may contribute to the detected levels of zearalenone in the rivers.

3.3. Seasonal variation of individual compounds

Statistical results revealed a significant difference between the concentrations of detected compounds across seasons and between the studied rivers. Overall, the obtained p-values ($F(3, 6083) = 6.800$, $p < 0.001$), at level the 0.05 indicate that mean concentrations of detected compounds are affected by seasonal changes. In particular, significant differences were observed between the wet season of May and, the dry season of February, the dry season of July and the wet season of October with within group p-values of $p < 0.006$, $p < 0.01$, and $p < 0.0001$ respectively. No statistically significant difference was found between the dry season of July and the dry season of February, the wet season of October and the dry season of February or the wet season of October and the dry season of July, as the obtained p-values were all >0.05 threshold. Results obtained from statistical analysis are provided in the supplementary Information A (Table A.2). Furthermore, the number of compounds detected in each season is provided in Supplementary

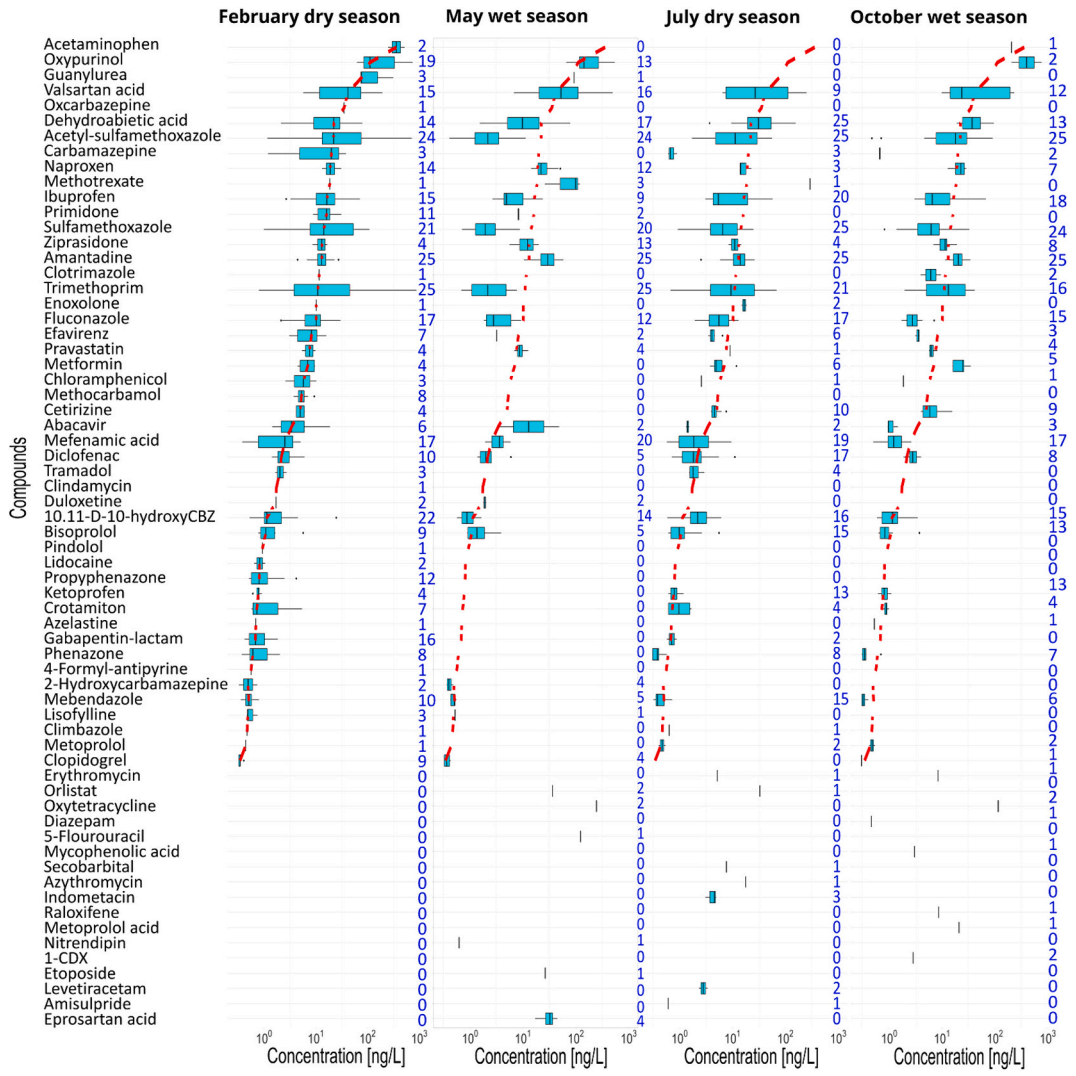

Fig. 4. Box plot summary showing median concentration of detected pharmaceuticals across four seasons. The center dark line in the plot represents the median concentration of the compound and the hinges represent the 25th and 75th percentiles. The upper and the lower whiskers represent the interquartile range. The values at the end of each plot represents the number of compounds generating the boxplots. The dotted red line cutting across the boxplots from the compound with the highest median concentration to the compound with the lowest median concentration in February dry season, indicates whether the median concentrations of individual compounds subsequent seasons (May, October, July) increased or reduced. 10,11-D-10-Hydroxycbz: 10,11-dihydro-10,11-dihydroxycarbamazepine. 1-CDX: 1-(3-carboxypropyl)-3,7-dimethylxanthine.

Information A, (Fig. A.1a).

In this study, 22 pesticides were detected across the four seasons while others were detectable only in specific seasons (Fig. 3).

For visualization of changes, median concentrations of detected pesticides during the February dry season (dotted-red line) were taken as a reference for the other seasons since the highest number of pesticides was detected in February. Several herbicides including 2,4 dichlorophenoxy acetic acid, metribuzin, 2-hydroxyatrazine, nicosulfuron, terbuthylazine-2-hydroxy, and tepraloxydim exhibit increased concentrations during the wet seasons (May and October, Fig. 3). These compounds are used in western Kenya for the control of weeds in agricultural crops and predominantly sprayed at the start of the rainy season. Teprolaxydim and nicosulfuron are post emergent herbicides used for controlling broadleaved weeds from rice and soybeans (PCPB, 2018). Nicosulfuron exhibits high selectivity on maize and is active on grass weeds interfering with maize crops (Mekki and Leroux, 1994; O'Sullivan et al., 2000; PCPB, 2018). Metribuzin and 2,4 dichlorophenoxy acetic acid are pre- and post-emergent herbicides for the control of weeds in potatoes, carrots, tomatoes, maize sugarcane and tomatoes (PCPB, 2018). Weed control in maize farms is done during the wet season of May while weed control in the vast sugarcane plantations in Migori county is mostly done during the wet season of October. Terbuthylazine-2-hydroxy and emamectin B1a pesticides were detected only during the wet seasons. Terbuthylazine-2-hydroxy is a metabolite of the herbicide terbuthylazine while emamectin B1a is an insecticide used for the control of leaf miners, thrips and other lepidoptera pests in vegetables.

In agreement with expectations, most pharmaceuticals concentrations were higher during the dry seasons, with limited dilution potential at low water levels (Fig. 4).

Similar findings have been reported in other countries such as Nigeria and China (Ogunbanwo et al., 2022; Pan et al., 2020). The median concentration of acetaminophen (analgesic), sulfamethoxazole (antibiotic), trimethoprim (antibiotic), carbamazepine (anti-convulsant), ibuprofen (NSAID), and fluconazole (antifungal) were high in February dry season. The occurrence of trimethoprim and sulfamethoxazole can be associated to the treatment of bacterial infection. A study by Shah et al. (2016) noted that bacterial infections in Kenya were particularly prevalent in dry seasons. During the wet seasons, nine pharmaceuticals including methotrexate, amantadine, and abacavir

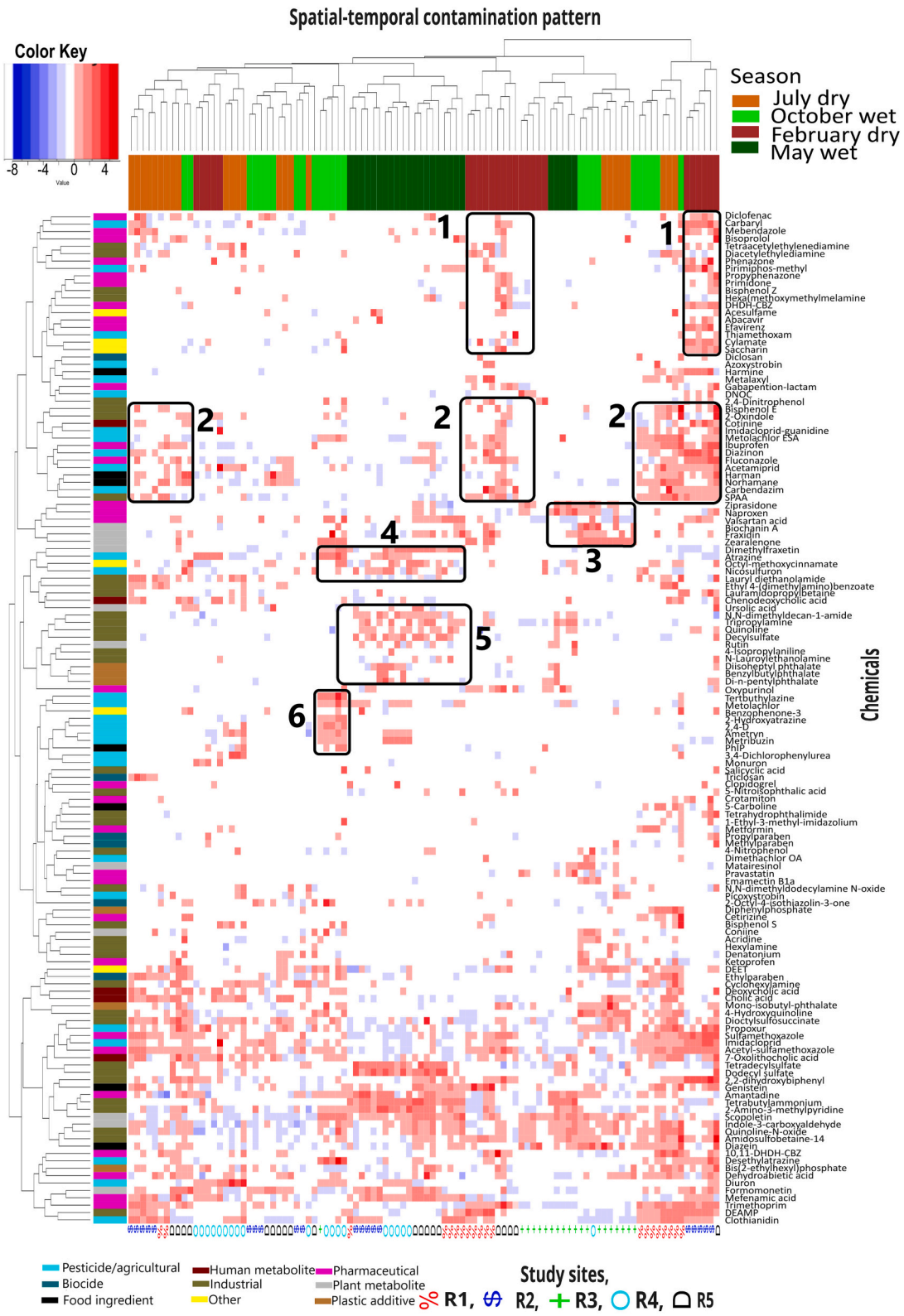

Fig. 5. Spatial-temporal contamination pattern in 25 sites of 5 Kenyan rivers for 4 seasons. Pattern generated from log transformed and centered data.

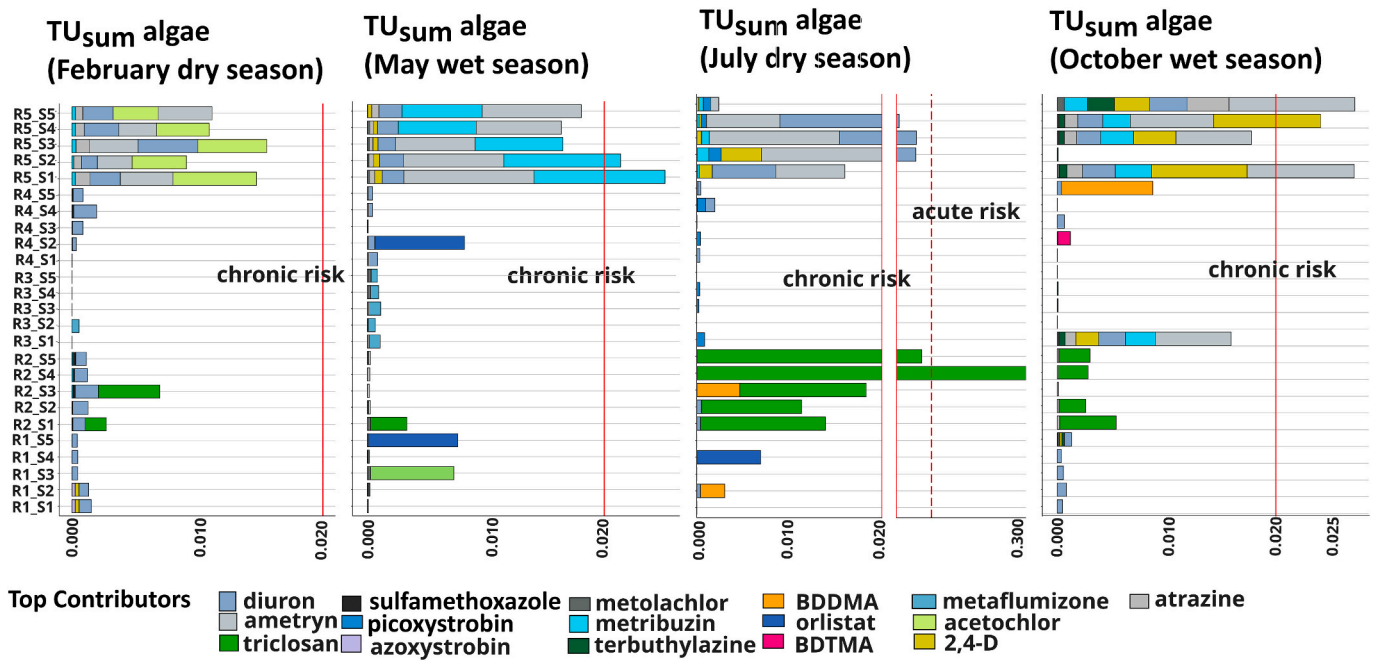

Fig. 6. TU_{sum} for algae in each sampling site throughout the dry and wet seasons. The red solid line indicates sites which exceeded chronic threshold and the red dashed line indicate sites which exceeded acute threshold. BDTMA: benzyldimethyltetradecylammonium. 2,4-D: 2,4-dichlorophenoxyacetic acid, BDDA: benzyldimethyldodecylammonium.

(May wet season) and oxypurinol, dehydroabetic acid, metformin, cetirizine, diclofenac and naproxen (October wet season) recorded increased median concentrations (Fig. 4). Methotrexate is used in Kenya for managing rheumatoid arthritis (Owino et al., 2009), which exacerbates during cold seasons. The high concentrations of cetirizine and amantadine in wet seasons could be linked to the treatment of cold-related illness and prevention of influenza respectively (Katz et al., 2012; Matheka et al., 2013). It is worth noting that oxytetracycline, an antibiotic used for the treatment of bacterial infections in animals and humans (Lianou and Fthenakis, 2022), was detected only in the wet and not in the dry season. Additionally, climbazole, enoxolone, tramadol and gabapentin-lactam were detected only during the dry seasons but fell below detection limits with high dilution in wet seasons.

Twenty-one industrial chemicals including surfactants and plastic additives were detected across the four seasons while the rest were season specific. The concentrations of individual compounds in this category are provided in the Supplementary Information A (Fig. A.2). Surfactants and phthalates including dodecylsulfate, tetradecylsulfate, decyl sulfate, n*N*,*N*-dimethyldecane-1-amide, dicylohexyl phthalate, diisoheptyl phthalate exhibited high concentration during the wet seasons (May). Similar findings have been reported in Malaysia and Spain (Alsalahi et al., 2014; Lara-Martín et al., 2010). During wet seasons, the majority of the people in western Kenya bath and clean their clothes, dishes and vehicles in the rivers. Runoffs from settlements may also be contributing to increasing concentration in the rivers. Some compounds were detected specifically in the dry seasons or wet seasons. As an example, tetraglyme (a glycol ether), a compound used in ink and toners was detected in both May and October wet seasons, suggesting a possible entry into rivers from rain runoffs.

Spatio-temporal contamination patterns were analyzed using hierarchical clustering of all samples from the different seasons and sites in order to identify groups of chemicals with similar occurrence patterns (Fig. 5).

In the following paragraphs, we will discuss six groups of compounds as examples.

3.3.1. *Chemical group 1*

Chemicals in group 1 exhibit high concentrations in February dry season in rivers 1, 2 and 4. Notably, pharmaceuticals are dominant in this group and constitute >50 % of the compounds. This group includes artificial sweeteners, diacetylethylenediamine and tetraacethylediamine (EDTA) from detergents (Deluchat et al., 2002), bisphenol Z and hexa(methoxymethyl)melamine from plastics and cars (both mainly in river 4). Two insecticides, namely carbaryl which is used against insects in households and pirimiphos-methyl used for controlling malaria vectors (Abong'o et al., 2020; Kitungulu et al., 2022; Mukabane et al., 2022) exhibit similar trends. This group is mainly composed of chemicals closely related to urban use and emission in densely inhabited areas (Rangwe (R2), Asao (R4) and Homabay (R1)) exhibiting peak concentrations during February as the season with particularly low dilution potential.

3.3.2. *Chemical group 2*

Similar to group1, group 2 exhibits very similar peak contamination in February and rivers 1, 2 and 4 and is characterized by chemicals from urban use and emission. The group includes human metabolites (e.g. cotinine) representing population density (Zhao et al., 2021), industrial compounds (2-oxindole, 4-(sulfophenyl) acetic acid), food ingredients (harman, norharmane), but also a larger proportion of pesticides (insecticide diazinon, acetamipirid, fungicide carbendazim) used for controlling biting insects in vegetables, flowers and fruit trees (Route to Food Initiative., 2019; PCPB, 2018). In contrast to group 1, group 2 exhibits high concentrations in river 1 also in July (dry) and October (wet). The detection of pesticides during wet season can be associated with intense agricultural practices accompanying October wet season in the region.

3.3.3. *Chemical group 3*

Group 3 shows occurrence patterns that are contradictory to the groups 1 and 2. They are typical contaminants of river 3 occurring there at high concentrations in the wet seasons (October and May) as well as in dry July. River 3 is located on the slopes of Gwasi hills, where most of the population lives in small, settlements. It is prone to flooding as the

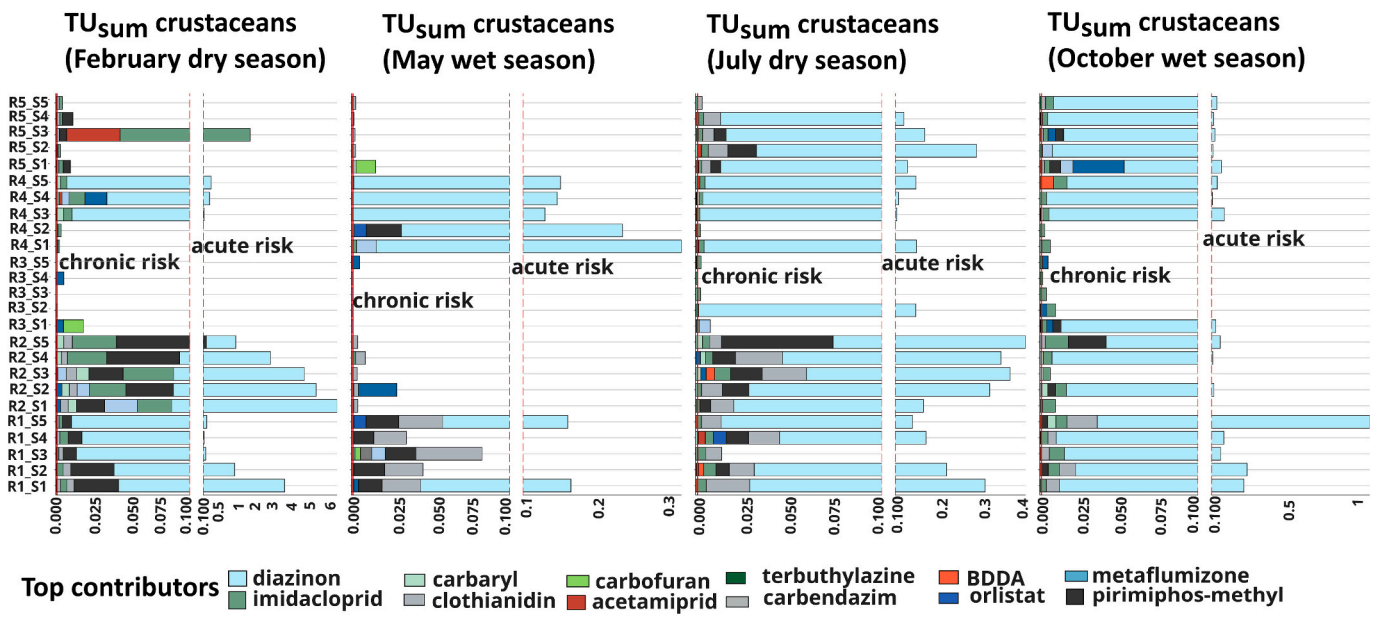

Fig. 7. TU_{sum} for crustaceans in each sampling site throughout the dry and wet seasons. The red solid line indicates sites which exceeded chronic threshold and the red dashed line indicate sites which exceeded acute. BDDA: benzyldimethyldodecylammonium.

region receives high rainfall. The land is covered by semi-natural vegetation which is mainly used as grazing fields. Prominent group members are the pharmaceuticals ziprasidone which is an antipsychotic drug (Yuan et al., 2013) and naproxen used as fever and pain killer. In addition, the plant metabolite zearalenone (Ropejko and Twarużek, 2021) and fraxidin portray similar patterns in these rivers which could be attributed to leaching of natural compounds from vegetation into rivers during rain events.

3.3.4. Chemical group 4

Group 4 is again complementary to the groups discussed so far with peak contamination in the wet season of May and partly of October in all the rivers representing chemicals leaching from agricultural fields and vegetation by rain runoff. This group comprises the herbicides atrazine and nicosulfuron which are commonly used for controlling weeds in maize plantations in the wet season (PCPB, 2018) and the plant metabolite dimethylfraxetin. Interestingly, in this group also the sunscreen ingredient octyl-methoxycinnamate can be found.

3.3.5. Chemical group 5

Group 5 exhibits occurrence patterns similar to group 4 with peak concentrations in May wet season in all rivers but without the high concentrations in October. They are mainly plasticizers and industrial chemicals (diisoheptylphthalate, benzylbutylphthalate, triprolamine, decylsulfate) which leach from infrastructure and plastic materials to the rivers during heavy rains in May.

3.3.6. Chemical group 6

Group 6 consists of co-occurring herbicides which were specifically detected in river 5 during the wet season of October and partly in wet season of May including metolachlor, terbuthylazine, 2,4 dichlorophenoxy acetic acid, metribuzin, ametryn and the atrazine transformation product 2-hydroxyatrazine. The occurrence of these herbicides is linked to the intensive application on sugarcane farms mainly in October and leaching to the river.

3.4. Risk assessment

Based on the toxic unit calculations, crustaceans were the organisms potentially at greatest risk compared to fish and algae. TU values across the seasons ranged from 0.02 to 0.32 for algae, 0.324 to 6.193 for crustaceans and 0.006 to 0.011 for fish. Supplementary information B, (Tables B.6, B.7, B.8, B.9).

3.4.1. Algae

Enhanced risks to algae were related mainly to the rivers 2 and 5 with very different patterns. In river 2 the major exceedance of chronic and even acute risk thresholds (maximum 0.32 TU) occurs in July dry season and is mainly driven by triclosan and benyzyldimethyldodecylammonium, both used as antimicrobial and disinfection products in health care. The frequent use of this river as a bathing place, the presence of hospitals such as Rangwe sub-county hospital and Rangwe dispensary and the low dilution capacity in July are likely to be the reasons for this deterioration of water quality (Fig. 6).

Triclosan inhibits growth and photosynthesis in algae (Machado and Soares, 2021) while benyzyldimethyldodecylammonium disrupts the cell membranes in microbes (Hsu et al., 2019). Mixture risks to algae in river 5 exceeds chronic risk thresholds for algae mainly in the wet seasons October and May, and is driven almost exclusively by agricultural herbicides used in the sugar cane plantations including diuron, ametryn, 2,4-dichlorophenoacetic acid, metribuzin.

3.4.2. Crustaceans

For crustaceans, chronic risk thresholds were exceeded in 96 % of all samples all over the year, while even acute toxicity (TU > 0.1) can be expected at 56 % in all samples from all seasons. Highest maximum TU values (6.1 TU) were recorded in February dry season and lowest (0.3 TU) in May seasons. A fraction of 14 out of 25 sites exceeded acute risk threshold (0.1 TU) of crustaceans in February dry season, 18 in July dry season, 17 sites in October wet season and only 7 sites in the wet season of May (Fig. 7).

The risk was mainly driven by diazinon, imidacloprid, pirimiphos-methyl and 3,5,6-pyridinol.

For the neonicotinoid imidacloprid, TU values of up to 2.2 TU were obtained in R5-S3 in February (Fig. 7). Imidacloprid binds to the nicotinic acetylcholine receptors in insects thereby interrupting the transmission of impulses. In addition, this compound has been associated with the decline of plant pollinators. Despite imidacloprid and diazinon are banned in Europe, these insecticides are still used in Kenya for controlling agricultural pests.

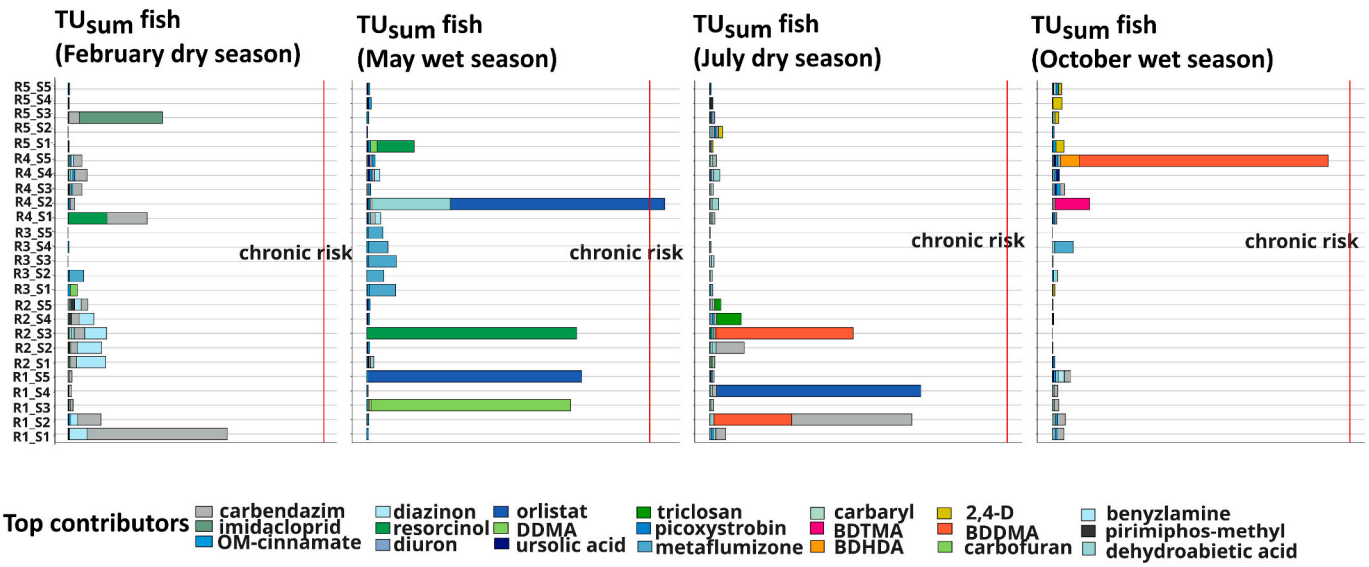

Fig. 8. TU_{sum} for Fish in each sampling site throughout the dry and wet seasons. The red solid line indicates sites which exceeded chronic threshold and the red dashed line indicate sites which exceeded acute. BDDA: benzyldimethyldodecylammonium. OM-Cinnamate: octyl-methoxycinnamate. BDTMA: benzyldimethyltetradecylammonium, BDHDA: benzyldimethylhexadecylammoniu, 2,4-D: 2,4-dichlorophenoxyacetic acid.

Table 1
List of compounds prioritized for regulation and abatement based on the extent (scaled) and frequency of exceedance of the risk thresholds. The threshold values are according to Malaj et al. (2014).

Compound	Class	Organism	Sites exceeding threshold (n)	Total Sites (N)	Frequency of Exceedance (n/N)	Threshold Value	Extent of exceedance	Priority Ranking
Diazinon	Pesticide	Crustacean	55	100	0.55	0.001	1.00000	1.55
Imidacloprid	Pesticide	Crustacean	83	100	0.83	0.001	0.35874	1.19
Triclosan	Biocide	Algae	2	100	0.02	0.02	1.00000	1.02
Clothianidin	Pesticide	Crustacean	58	100	0.58	0.001	0.00666	0.59
Pirimiphos-methyl	Pesticide	Crustacean	36	100	0.36	0.001	0.03325	0.39
Carbaryl	Pesticide	Crustacean	23	100	0.23	0.001	0.00137	0.23
Acetamiprid	Pesticide	Crustacean	11	100	0.11	0.001	0.00638	0.12
3,5,6-Trichloro-2-pyridinol	Pesticide	Crustacean	9	100	0.09	0.001	0.00382	0.09
Carbofuran	Pesticide	Crustacean	3	100	0.03	0.001	0.00224	0.03
Benzyldimethyldodecylammonium	Biocide	Crustacean	3	100	0.03	0.001	0.00120	0.03
Orlistat	Pharmaceutical	Crustacean	3	100	0.03	0.001	0.00107	0.03
Carbendazim	Biocide	Crustacean	2	100	0.02	0.001	0.00024	0.02
Diuron	Biocide	Algae	2	100	0.02	0.02	0.00000	0.02
Didecyldimethylammonium	Biocide	Crustacean	1	100	0.01	0.001	0.00098	0.01
Triclosan	Biocide	Crustacean	1	100	0.01	0.001	0.00029	0.01
Dehydroabietic acid	Pharmaceutical	Crustacean	1	100	0.01	0.001	0.00022	0.01
Benzyldimethyltetradecylammonium	Biocide	Crustacean	1	100	0.01	0.001	0.00002	0.01
Terbuthylazine	Pesticide	Crustacean	1	100	0.01	0.001	0.00000	0.01

3.4.3. *Fish*

Based on the calculated TU values, potential toxicity to fish was generally lower (TU up to 0.008) across the seasons compared to algae and crustaceans. The highest TU_{sum} recorded in the four seasons ranged from 0.0063 TUs to 0.0106 TUs. All obtained values were below acute threshold (0.1) and only one site exceeded chronic threshold (0.01) (Fig. 8).

The major compounds driving fish toxicity were orlistat, dehydroabietic acid, triclosan, didecyldimethylammonium, benzyldimethyldodecylammonium, resorcinol and carbendazim. Dehydroabietic acid induces changes in metabolic enzymes of rainbow trout (Pandelides et al., 2014). Carbendazim, induces the display of abnormal behavior in zebrafish embryos, such as hyperactivity, spiral swimming and equilibrium loss (Andrade et al., 2016).

3.5. *Prioritization of compounds for monitoring and abatement*

The prioritization of chemicals for monitoring and abatement based on the current set of samples from Western Kenya according to the exceedance of both acute and chronic risk thresholds resulted in a list of 18 compounds led by the insecticides diazinon and imidacloprid that have been identified already as major risk drivers in Western Kenya earlier (Kandie et al., 2020b) but which are also risk drivers in water bodies of other parts of the world including Europe (Finckh et al., 2022, 2024). The most prominent risk driver for algae is triclosan, a compound of high concern also in Europe (Carsten Von Der Ohe et al., 2012). Based on the TU values, majority of compounds that were proposed for regulation and abatement were pesticides and biocides (Table 1).

4. Conclusions

This comprehensive study reported 307 compounds in western Kenyan rivers and thus the highest number ever reported in Kenya with individual compound concentrations ranging from 0.3 ng/L to 6.64 μg/L. For the first time, seasonal dynamics and spatio-temporal patterns were explored and could be linked to the existing land use practices, disease prevalence and seasonal variations in the studied location. Water bodies with urban areas in the catchment and those impacted by

agricultural activities both exhibited severe contamination, however with different seasonal patterns. They could be interpreted as a result of seasonal application of pesticides and mobilization as well as bathing and washing activities in the rivers during wet seasons on the one hand, and a lack of dilution of permanently emitted household and industrial chemicals during dry seasons on the other hand. High toxic risks were observed particularly for crustaceans as model organisms also for other invertebrates including insects with the insecticides diazinon and imidacloprid as major risk drivers. Algal communities are at risk of triclosan and several agricultural herbicides. In total, we identified 18 candidate priority compounds for monitoring and abatement to protect the environment and humans from chemical contamination and to avoid impacts on health, economic stability and ecosystem integrity. Spatio-temporal patterns identify high-risk compound-water body-season combinations for prioritization of monitoring activities.

CRediT authorship contribution statement

Isaac Cheruiyot Tanui: Writing – review & editing, Writing – original draft, Visualization, Investigation, Formal analysis, Conceptualization. **Faith Kandie:** Writing – review & editing, Supervision, Investigation, Conceptualization. **Martin Krauss:** Writing – review & editing, Validation, Supervision, Resources, Methodology, Investigation, Data curation, Conceptualization. **Aleksandra Piotrowska:** Formal analysis. **Ambrose Kiprop:** Writing – review & editing, Supervision, Project administration. **Naeem Shahid:** Writing – review & editing, Visualization, Funding acquisition, Conceptualization. **Matthias Liess:** Writing – review & editing, Funding acquisition, Conceptualization. **Werner Brack:** Writing – review & editing, Validation, Supervision, Resources, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scitotenv.2024.175036.

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