

Occurrence and Characterization of Residues and Contaminants in Shrimp and Fish Samples from Aquaculture and Wild Capture in Brazil

ABSTRACT

The consumption of fish is a regular dietary habit among Brazilians, as it represents a significant protein source for the population. However, the emerging presence of residues and contaminants in fish meat has proven to be a substantial public health risk, contributing to cases of human intoxication and resulting in irreversible damage, including gastrointestinal, neurological, and endocrine disorders. This study aimed to conduct a retrospective analysis based on data retrieved from reports published on the official website of the Ministry of Agriculture and Livestock (MAPA) over the past 13 years. The objective was to evaluate the occurrence and characterization of contaminants in shrimp and fish from aquaculture and wild capture in Brazil. The analyzed documents comprised results from the National Residues and Contaminants Plan (PNCRC/Animal) between 2010 and 2023. Residues were monitored according to criteria such as toxicity levels and population exposure potential. Over the 13 years of monitoring under the PNCRC, more than 12,000 samples were analyzed. Non-compliance was identified in 189 samples, distributed among the following classes of substances: inorganic contaminants, antibiotics, dyes, and insecticides. The class of inorganic contaminants accounted for the highest percentage of violations among non-compliant samples, representing approximately 90% of the cases. These findings underscore the importance of the PNCRC/Animal in ensuring the availability of safe food for the Brazilian population while mitigating the harmful impacts on human health.

Keywords: *Aquaculture; contaminants; fish; foods; shrimp;*

1. INTRODUCTION

Fish meat holds significant importance in the dietary habits of the Brazilian population due to its high nutritional value, which justifies the increasing consumption, reflecting a 65% growth since 2004 (Sanchez, 2023). In the global fish production chain, Brazil occupies a prominent position, driven mainly by an increase of almost 50% in aquaculture production since 2016, becoming the fourth largest producer of tilapia (*Oreochromis niloticus*) worldwide according to the Association Brazilian Aquaculture (Sanchez, 2023). In this context, the expansion of fish production raises concerns regarding food safety, particularly the presence of potential chemical residues and contaminants in the production chain and their significant implications for public health.

The impact of residues and contaminants in fish meat on public health is highly relevant. Some substances, when ingested in large quantities, can be lethal, trigger poisoning or cause irreversible damage, and, according to the International Institute of Life Sciences (ILSI, 2022), adverse reactions to these substances include acute effects, such as

gastrointestinal disorders and chronic conditions, including neurological and endocrine disorders, infertility, genetic mutations and carcinogenesis.

In Brazil, the Ministry of Agriculture, Livestock, and Food Supply (MAPA) is the authority responsible for regulating the production of animal-derived products. Through the National Residues and Contaminants Control Plan (PNCRC), MAPA determines the substances to be monitored and establishes maximum allowable limits for these products (Brazil, 1999). The legislation sets acceptable maximum levels for various veterinary inputs, pesticides, inorganic contaminants, organophosphates, dioxins, and others, as outlined in Normative Instructions N^o. 160 and N^o. 161, both dated July 1, 2022 (Brazil, 2022).

In this regard, the present study offers a retrospective analysis aiming to characterize the main types of residues and contaminants identified in shrimp, aquaculture fish, and wild-caught fish samples analyzed by MAPA between 2010 and 2023.

2. MATERIAL AND METHODS

This research consists of a retrospective study based on data gathered from case records and statistical reports. The information was collected from reports published on the official website of the Ministry of Agriculture, Livestock, and Supply (MAPA), accessible via the following link: <https://www.gov.br/agricultura/pt-br/assuntos/inspecao/produtos-animal/plano-de-nacional-de-controle-de-residuos-e-contaminantes>.

These documents present the results of the National Residues and Contaminants Control Plan (PNCRC/Animal) for animal-derived product samples collected between 2010 and 2023. The samples were obtained by the Federal Inspection Service from single-origin animal lots. The respective analyses were conducted in Federal Agricultural Defense Laboratories (LFDAs) and public or private laboratories accredited by MAPA, all of which were certified under the ABNT NBR ISO/IEC 17025:2005 standard.

In accordance with Normative Instruction N^o. 42, issued in December 1999 by MAPA, the substances selected for monitoring were chosen based on criteria such as the actual presence of residues, their toxicity levels concerning consumer health, population exposure potential, misuse of drugs, proper dosage and administration routes, withdrawal periods, and residues that might pose trade barriers for animal-derived products. Accordingly, the PNCRC aims to prevent violations of safety levels and Maximum Residue Limits (MRLs) for authorized compounds, as well as the presence of any level of prohibited substances (Brazil, 1999).

The data retrieved from the reports were organized into Excel spreadsheets and categorized according to animal species/category, substance class, analyzed matrix, number of samples tested, and number of non-compliant samples. The applicable residue and contaminant limits were defined by Normative Instruction N^o. 162 and Normative Instruction N^o. 160, both issued on July 1, 2022. The monitored compounds were classified into distinct groups, as shown in the corresponding table (Table 1).

Table 1. Classes of substances monitored by the PNCRC/Fish and examples

Classes of Substances	Examples of Substances
Antimicrobials	Tetracycline, Chloramphenicol, Ciprofloxacin, Enrofloxacin, Sulfamethazine, Sulfathiazole, Sulfadimethoxine, Nitrofurazone.
Antiparasitics	Flubendazole, Mebendazole, Oxfendazole.
Pesticides/Fungicides/Insecticides	Carbamate, Alachlor, Aldicarb, Bitertanol, Carbofuran, Cyproconazole, Diflubenzuron.
Dyes	Malachite Green, Crystal Violet, Leucomalachite Green.
Inorganic Contaminants	Mercury, Lead, Arsenic, and Cadmium.
Dioxins and Furans	Polychlorinated Dibenzodioxins (PCDDs), Polychlorinated Dibenzofurans (PCDFs), Polychlorinated Biphenyls (PCBs).
Polycyclic Aromatic Hydrocarbons	Benzo(a)pyrene, Benzo(a)anthracene, Chrysene.
Anabolic Substances	Beta-estradiol, Dienestrol, Zeranol, Hexestrol.
Organochlorines	Heptachloro, Endrin, Aldrin, Clordano.

The data underwent descriptive statistical analysis, with results presented as relative and absolute frequencies of non-compliant samples and the identified residue and contaminant categories. Additionally, graphs were generated to enhance visualization and presentation of the results, particularly regarding the temporal distribution of the analyses conducted.

3. RESULTS

The total number of samples analyzed between 2010 and 2023 by animal category is presented in Figure 1. Based on the results, it is evident that the categories of farmed fish and wild-caught fish had the highest number of samples evaluated in 2011, with approximately 796 and 305 samples analyzed, respectively. Conversely, 2020 recorded the lowest number of samples evaluated for both categories, totaling only 229 and 73 samples, respectively. Additionally, the farmed shrimp category reached its peak number of analyses in 2019, with 359 samples, in contrast to 2014, which recorded only 71 samples.

The number of analyzed samples by animal category and substance classes is presented in Table 1. Regarding the substance classes analyzed in farmed fish, the data show that over the 14 years evaluated, the following groups of compounds were tested: antimicrobials, dyes, inorganic contaminants, and anabolic substances. It is noteworthy that the category of Polycyclic Aromatic Hydrocarbons (PAHs) was assessed only in 2010 and 2011. Subsequently, there was a nine-year gap in evaluations until its re-inclusion in 2022. The analysis of organochlorine compounds showed sporadic gaps, with no evaluations in 2013 and 2019. Similarly, anabolic substances were not analyzed in 2016. Furthermore, dioxins and furans, pesticides/fungicides/insecticides, and antiparasitic compounds were included in the analyses only in 2012, 2017, and 2020, respectively.

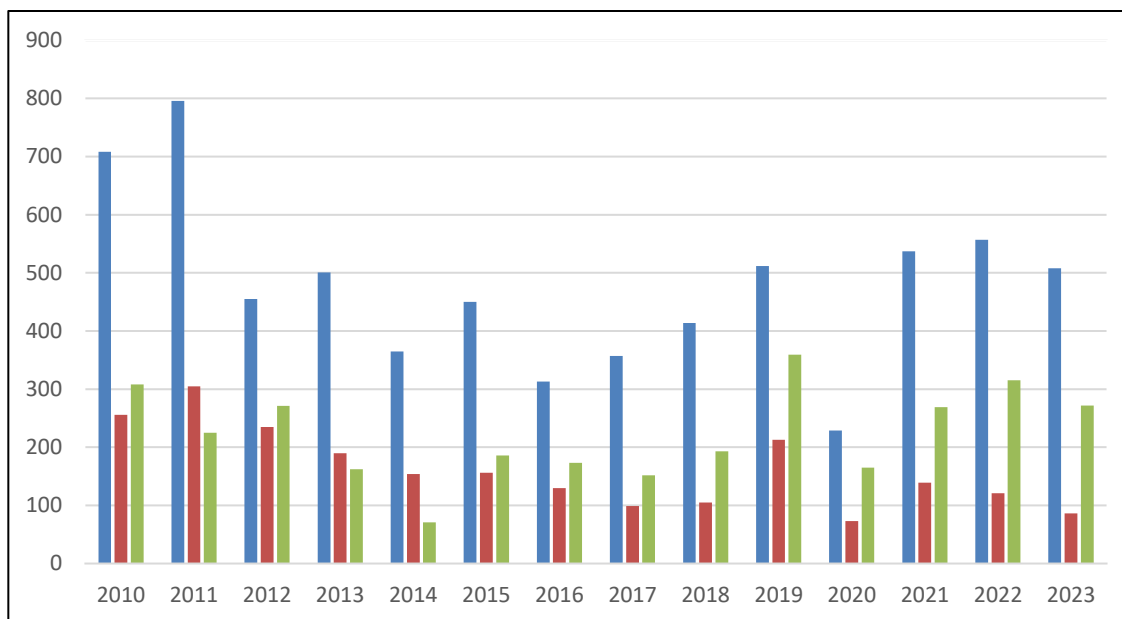


Figure 1. Distribution of Analyzed Samples of Farmed Fish (blue), Wild-Caught Fish (red), and Shrimp (green) from 2010 to 2023, According to the PNCRC.

For wild-caught fish, the total tests revealed that the PAH class was analyzed between 2010 and 2012 but was excluded for six years before being reintroduced in 2019. The dioxins and furans category was absent during the first three years of the study, being incorporated into the PNCRC/Fish program only in 2013. Among the substance classes, inorganic contaminants were the only category consistently analyzed throughout the 13 years of monitoring.

Regarding farmed shrimp samples, substances from the PAHs, antiparasitic, and organochlorine groups are the most recent additions to the PNCRC/Fish program, appearing in results only from 2020 onwards. Other groups, such as antimicrobials, dyes, and inorganic contaminants, were consistently monitored throughout the study period.

Over the period analyzed in this study, a total of 189 non-compliant samples were identified (Table 3) for various residues and contaminants investigated (Table 4). Among these, inorganic contaminants were predominant, with 90.47% (171 samples) testing positive for violations of maximum residue limits. Additionally, other notable substances included antibiotics (7.40%), dyes (1.58%), and insecticides (0.52%). For the farmed fish category, a total of seven non-compliant samples were detected during the analyzed period. Specifically, one sample was found non-compliant for chloramphenicol in 2011; in 2021, violations were identified for diflubenzuron (1 sample) and leucomalachite green (2 samples); one sample for metronidazole was detected in 2022; and finally, in 2023, two samples were non-compliant, one for nitrofurazone and one for leucomalachite green.

Table 2. Total number of samples analyzed, categorized by animal type and substance class.

RESIDUES AND CONTAMINANTS	2010	2011	2012	2013	2014	2015	2016	2017	2018	2019	2020	2021	2022	2023	TOTAL
SHRIMP															
PAHs	-	-	-	-	-	-	-	-	-	-	15	28	12	16	71
Inorganic Contaminants	33	28	57	44	25	59	31	47	41	137	14	46	57	48	667
Antimicrobials	230	127	157	92	39	82	110	54	124	149	82	107	123	103	1,579
Colorants	45	70	57	26	7	45	32	51	28	73	30	56	53	45	618
Antiparasitics	-	-	-	-	-	-	-	-	-	-	8	14	54	44	120
Organochlorines	-	-	-	-	-	-	-	-	-	-	16	18	16	16	66
FARMED FISH															
PAHs	33	61	-	-	-	-	-	-	-	-	-	-	26	22	142
Pesticides/Organophosphates/Fungicides	-	-	-	-	-	-	-	27	40	35	21	33	33	63	252
Antiparasitics	-	-	-	-	-	-	-	-	-	-	16	16	57	104	193
Organochlorines	45	60	10	-	20	24	1	30	12	-	18	18	25	16	279
Dioxins and Furans	-	-	28	62	62	45	56	41	50	7	6	27	15	17	416
Antimicrobials	409	374	263	277	151	188	126	105	121	200	67	180	176	154	2,791
Colorants	59	76	61	60	33	94	69	65	60	84	26	109	121	33	950
Inorganic Contaminants	114	146	62	67	65	74	61	45	76	95	18	109	58	52	1,042
Anabolic Substances	48	79	31	35	34	25	-	44	55	91	57	45	46	47	637
WILD-CAUGHT FISH															
PAHs	30	61	20	-	-	-	-	-	-	62	32	81	40	40	255
Dioxins and Furans	-	-	-	59	38	25	52	52	46	10	4	22	15	20	343
Inorganic Contaminants	226	244	215	131	116	131	78	47	59	141	37	36	66	26	1,533
TOTAL	1,272	1,326	961	853	590	792	616	608	712	1,084	467	945	993	866	12,085

Table 3 - Quantification of non-compliant samples by animal category assessed in the PNCR, including wild-caught and farmed fish, as well as farmed shrimp.

Period	Farmed Fish	Wild-Caught Fish	Farmed Shrimp	Total
2011	01	21	-	22 (0,18%)
2012	-	44	04	48 (0,39%)
2013	-	34	06	40 (0,33%)
2014	-	16	-	16 (0,13%)
2015	-	26	-	26 (0,21%)
2016	-	10	-	10 (0,08%)
2017	-	03	-	03 (0,02%)
2018	-	16	01	17 (0,14%)
2019	-	-	-	-
2020	-	-	-	-
2021	03	-	01	04 (0,06%)
2022	01	-	-	01 (0,008%)
2023	02	-	-	02 (0,016%)
Total	07/6.702	170/2.262	12/3.121	189/12.085

Table 4 - Quantification of non-compliant samples by category of residues/contaminants assessed in the PNCR, regardless of the animal category.

Categories	2011	2012	2013	2014	2015	2016	2017	2018	2021	2023	Total
Inorganic Contaminants	21	44	34	16	26	10	03	17	-	-	171
Antibiotics	01	04	06	-	-	-	-	-	02	01	14
Colorants	-	-	-	-	-	-	-	-	02	01	03
Insecticides	-	-	-	-	-	-	-	-	01	-	01
Total	22	48	40	16	26	10	03	17	05	02	189

In the wild-caught fish category, a total of 170 non-compliant samples were identified throughout the study period. In 2011, 21 non-compliant samples were found within the group of inorganic contaminants, including arsenic (18), cadmium (2), and mercury (1). In the following year, 44 samples showed violations, comprising arsenic (40), lead (2), and mercury (2). In 2013, 34 non-compliant samples were detected, with arsenic (33) and mercury (1).

In 2014, the number of non-compliant samples decreased to 16, all testing positive for arsenic. In 2015, 26 samples were positive for arsenic violations, and two of these also exhibited mercury and cadmium simultaneously. In 2016, arsenic (9) and mercury (1) were identified in non-compliant samples. During the subsequent two years, 19 muscle samples showed high concentrations of arsenic.

For the farmed shrimp category, 12 non-compliant samples were identified during the study period. In 2012 and 2013, four and six samples, respectively, tested positive for nitrofurazone and semicarbazide. Additionally, in 2018, one sample was found to violate arsenic limits, and in 2021, a sample showed excessive levels of oxytetracycline.

4. DISCUSSION

Based on the results described above, the number of samples collected and analyzed over the 13-year period under the PNCRC/Fish program remains minimal compared to the current production levels in Brazil. According to data from the Brazilian Aquaculture Association (Sanchez, 2023), Brazil's farmed fish production in 2023 totaled approximately 860,000 tons, alongside shrimp production, which reached 113,300 tons in 2022 (Sanchez, 2023). Furthermore, the Fishing Audit in Brazil (Oceana, 2020) reported that marine wild-capture fisheries production in 2020 was estimated at 500,000 tons. These figures highlight a significant disparity between the number of samples evaluated under the PNCRC program and the actual scale of production, potentially undermining the effectiveness of residue and contaminant control measures in this food matrix.

Another critical aspect is the scope of the program, which is designed to monitor the chemical safety of food from establishments under Federal Inspection Service (SIF) control (Brazil, 1999). This excludes establishments regulated by state and municipal inspection services. The reduced sampling size relative to the actual production scenario contradicts the PNCRC/Animal's goal of achieving a representative analysis of animal-derived food produced in Brazil (Brazil, 1999).

Inorganic contaminants were the most frequently identified compounds throughout the study period. Fish represent a significant source of contamination for these substances due to industrial discharges and untreated urban effluents being directed into coastal areas, exposing marine life to these pollutants (Mantovani, 2005). The primary inorganic compounds detected were arsenic (As), cadmium (Cd), mercury (Hg), and lead (Pb). The presence of these heavy metals in fish tissues can be attributed to both the natural occurrence of these elements in soil and marine organisms and the impact of industrial contamination (Mapa, 1999). Wild-caught fish are particularly susceptible to higher concentrations of these substances, as they inhabit uncontrolled environments, unlike farmed fish (Embrapa, 2023).

Arsenic, the most prevalent compound found during the 13 years studied, is naturally present in the environment due to mining activities, petroleum refineries, pesticide use, and other sources (Furlan and Vaz, 2004). As a widely distributed element in the Earth's crust (Cetesb, 2022), arsenic was found at concentrations exceeding permissible limits in 85.18% of the samples analyzed by MAPA. Chronic ingestion of elevated levels of this compound poses significant health risks, including carcinogenic potential, as well as circulatory and neurological dysfunctions (Lima et al., 2005).

Regarding antibiotics, the main compounds identified were nitrofurazone, semicarbazide, chloramphenicol, metronidazole, and oxytetracycline. Similarly to polychlorinated biphenyls and organochlorine pesticides, antibiotics were also identified by Justino et al. (2016) as one of the main contaminants in samples originating from aquaculture. These substances exhibit a broad spectrum of toxicity in humans and can contribute to bacterial resistance when used indiscriminately (Brazil, 1999). According to the 2022 Good Aquaculture Practices Manual, the only antimicrobials registered and approved for use in Brazilian aquaculture are florfenicol and oxytetracycline (Embrapa, 2023). Other drugs, such as amoxicillin, enrofloxacin, and norfloxacin, are currently used off-label, though unapproved, due to reduced efficacy of registered molecules against resistant pathogens (Mapa, 2022).

Normative Instruction N^o. 9, issued on June 27, 2003, prohibited the use of chloramphenicol and nitrofurazone-based compounds in veterinary medicine and animal feed due to severe

adverse effects on human health (Guidi *et al.*, 2015). Nitrofurans, including nitrofurazone and its metabolite semicarbazide, pose risks to humans because of their carcinogenic, mutagenic, and teratogenic potential (Wang *et al.*, 2020). Additionally, these antibiotics are critical in human medicine, and their use in food-producing animals is discouraged to prevent antimicrobial resistance (Guidi *et al.*, 2015).

Chloramphenicol, nitrofurazone, and semicarbazide are active pharmaceutical ingredients (APIs) with Maximum Residue Limits (MRLs) not recommended by the National Health Surveillance Agency (ANVISA), as specified in Normative Instruction N^o. 162 of July 6, 2022. Positive results for these substances constitute a violation of ANVISA Resolution RDC N^o. 730 of 2022 and IN N^o. 162/2022, representing a sanitary infraction under Law N^o. 6,437 of 1977 (Brazil, 2022; Brazil 1977). These findings likely result from the clandestine use of these veterinary drugs in treating diseases, reflecting the reality of prohibited drug use in various animal production systems in Brazil (Moreira, 2012).

Oxytetracycline, a tetracycline-group antimicrobial, is among the most commonly used in Brazilian aquaculture (Takeshita, 2019). It has a bacteriostatic effect and is widely used to treat furunculosis and erythrodermatitis in carp and as prophylaxis for hepatopancreatic necrosis and psychrophilic bacteria in shrimp (Brazil, 1999). The presence of non-compliant samples in tests may reflect the indiscriminate use of this substance, overdosing, feed contamination, and disregard for the established withdrawal period of 21 days (MAPA, 2022).

Metronidazole is another substance listed in Normative Instruction 162 as having a non-recommended Maximum Residue Limit (MRL) according to ANVISA. In aquaculture, this drug is still widely used in combination with sulfadimethoxine to control bacterial infections due to its broad-spectrum action (Ikefuti, 2016). However, its use is still questioned, as the withdrawal period for these drugs in fish muscles is not widely known (Ikefuti, 2016). Due to the lack of more medicinal options for aquaculture, many unregulated or unauthorized drugs have been improperly used (Assis *et al.*, 2018), thus leading to numerous positive results during monitoring.

Another class of compounds found in the tests conducted were dyes, specifically malachite green and its metabolite leucomalachite green. This compound is one of the APIs listed in ANVISA's IN No. 162 with a non-recommended MRL for aquaculture use. This decision is based on the evidence of its carcinogenic, mutagenic and teratogenic properties in animals, with an impact on aquaculture products (Silveira, 2019). Malachite green is a dye in the triphenylmethane class, introduced in aquaculture as an ectoparasiticide, fungicide, and antiseptic; however, its use is authorized only for ornamental fish (Silveira, 2019).

Nevertheless, due to its effectiveness in treating numerous infections, easy access, and low cost, it is acknowledged that producers continue to administer malachite green improperly (Silveira, 2019), a fact that explains its detection in tests conducted during the PNCRC/Fish program. Another factor worth noting is that this dye is also used in the textile industry, where 10 to 15% of dye residues are discarded in effluents during the production process, polluting aquatic environments (Majeed *et al.*, 2014). Therefore, in addition to improper use, the abundant discharge of residues into the water may be one of the causes of the compound's presence in samples and raises concerns about contamination of aquatic life and subsequent transfer to humans through food (Vyahare *et al.*, 2018).

The diflubenzuron was one of the substances found in tests conducted in 2021. It is an insecticide whose use is regulated only for agricultural activities to combat pests affecting

maize, cotton, and wheat crops (Dantzger, 2013). However, since the 1990s, it has been widely used in aquaculture to control parasitic infestations due to its low toxicity in fish (Fujimoto *et al.*, 1999). It is effective against parasites of the *Ergasilus*, *Lernaea*, and *Argulus* genera (Benze, 2014), although it has been proven that its long-term use can cause harm to fish health, affect water quality in water bodies, and damage other sensitive aquatic species such as algae and crustaceans (Dantzger, 2013).

Among other classes of compounds that were not found in non-compliant samples but deserve mention are organochlorines, anabolic substances, PAHs, and dioxins and furans. Most of the organochlorine compounds included in the analyses have parasitocidal effects and are banned for use in agriculture and livestock (Brazil, 1999). However, these substances are highly persistent in the environment and may leave residues in animal-derived products (Brazil, 1999). They are also known as the most persistent pesticides ever manufactured (Larini, 1999). There are few authorized compounds in this class, and their use is limited and specific due to their high bioaccumulation potential in the food chain (Brazil, 1999) and lack of selectivity for target species (Silva, 2009).

The class of anabolic substances has diethylstilbestrol as its main representative since it was the only compound analyzed in this category until 2018, with others added the following year. It is a nonsteroidal estrogen whose function is to induce the natural maturation of oocytes in fish (Tokumoto *et al.*, 2004) and is currently used in aquaculture for Nile tilapia feminization during the initial stage of creating monosex male populations (Marin-Ramirez *et al.*, 2015). Another method for reversing the sex of Nile tilapia to cultivate males is the use of methyltestosterone in feed, with a potential production of 98% males (Zanardi *et al.*, 2000). In Brazil, tilapia production is one of the main pillars of Brazilian aquaculture (Embrapa, 2023), making the monitoring of these hormones' residues in fish products highly significant.

Polycyclic Aromatic Hydrocarbons (PAHs) are substances widely distributed in the environment due to anthropogenic sources such as oil exploration, vehicle fuel combustion, urban stormwater runoff, and natural sources, including biosynthesis by marine or terrestrial organisms, natural oil seepages in oceans, atmospheric exchanges, and incomplete forest combustion (Araujo, 2010). These compounds can settle in marine sediments and bioaccumulate in the tissues of exposed animals (Martins, 2019).

However, even unprocessed fish from highly contaminated areas often exhibit low PAH concentrations due to their metabolic system's significant ability to biotransform these elements, which may explain their absence in the analyses (Azeredo *et al.*, 2006). In contrast, shrimp can metabolize and excrete PAHs; however, this process requires a high energy expenditure, potentially reducing the growth rate of juvenile shrimp (Roth and Baltz, 2009). Another noteworthy observation by Roth and Baltz (2009) is that, unlike fish, shrimp lack the ability to avoid habitats contaminated by oil, increasing their exposure to these pollutants. Therefore, shrimp have proven to be an effective tool for health risk assessments (Guo *et al.*, 2007).

According to the European Food Safety Authority (EFSA), dioxins and furans are a class of organic pollutants resulting from thermal and combustion processes. These substances are widely distributed in the environment and pose risks to human health, potentially causing damage to the nervous, endocrine, reproductive, and immune systems (Efsa, 2012; Cetesb, 2022). Marine sediment contamination allows species to accumulate dioxins and furans, as these substances disperse extensively through air, soil, and water (Montagner, 2017).

Despite their widespread environmental distribution, no non-compliant samples for this category were detected. This may be attributed to the effectiveness of the actions and strategies outlined in the National Implementation Plan of the Stockholm Convention, of which Brazil has been a signatory since 2001. This plan aims to limit the production, use, and safe disposal of Persistent Organic Pollutants (POPs), including dioxins and furans (Brazil, 2017).

Despite these findings, Mota et al. (2024) recently reaffirm that further advancements are still necessary in the investigation of emerging contaminants in Brazilian aquaculture, as the number of existing studies remains lower compared to global figures. Moreover, the authors emphasize that most research in this field is overly concentrated in the southern and southeastern regions, with few studies focusing on the analysis of cultivation sites to identify the contaminants present and their sources, particularly in the farming of native species.

In light of the findings, the PNCRC/Animal program is a critical tool for monitoring residues and contaminants in aquaculture products in Brazil. Its relevance extends beyond food safety, significantly impacting the Brazilian economy, as the country currently engages in exports where certain compounds monitored under the PNCRC could act as trade barriers. Continuous control and monitoring are, therefore, essential to ensure ongoing economic growth and the food safety of animal-derived products. The program plays a pivotal role in safeguarding public health and maintaining the quality standards required for international trade in aquaculture products.

4. CONCLUSION

From the data obtained during this retrospective study, it can be concluded that over the 13 years of PNCRC monitoring, more than 12,000 samples were analyzed. However, the total number of samples analyzed during the study period appears low compared to the current reality of aquaculture production in Brazil. Non-compliances were identified in 189 samples, distributed across the following substance classes: inorganic contaminants, antibiotics, dyes, and insecticides. Among these, the inorganic contaminant class showed the highest violation rate, accounting for approximately 90% (171 samples) of the non-compliant results. This indicates that fish can serve as a contamination source for these elements due to their natural occurrence in the environment and industrial and anthropogenic pollution. Some substances detected in the tests are prohibited for use in animal production, such as chloramphenicol, while others, like malachite green and diflubenzuron, are authorized for other purposes but are improperly used in aquaculture. This demonstrates the risks to the food safety of animal-derived products when such substances are involved in their production systems. The tests conducted allow the detection of MRL violations for authorized substances and the presence of any level of prohibited compounds. It is crucial to highlight those certain substances, when present in concentrations exceeding permissible limits and ingested in high amounts by humans, can lead to varying degrees of acute or chronic illnesses. Thus, the importance of the PNCRC/Animal program is reinforced as a tool for monitoring and ensuring the safety of food offered to the Brazilian population. Through surveillance and verification, it helps to ensure the correct and safe use of medications, evaluate the technologies employed, and control the environment in which animals are produced. This is vital for safeguarding public health and maintaining the integrity of the food supply chain.

Disclaimer (Artificial intelligence)

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

UNDER PEER REVIEW

REFERENCES

Araujo, L. P. (2010). Assessment of fish exposure to polycyclic aromatic hydrocarbons in Guanabara Bay, Rio de Janeiro - Brazil (Master's thesis). Universidade Federal da Paraíba, João Pessoa.

Assis, D. C. S., *et al.* (2018). Antimicrobial residues in fish. Technical Notebooks of Veterinary and Animal Science.

Azeredo, A., *et al.* (2006). Determination of benzo(a)pyrene in fish. Food Science and Technology, 26(1), 89–93.

Benze, T. P., *et al.* (2014). Subchronic exposure to diflubenzuron causes health disorders in Neotropical freshwater fish, *Prochilodus lineatus*. Environmental Toxicology, 10.

Brazil. (1977). Law No. 6,437 of August 20, 1977. Defines violations of federal health legislation, establishes the respective sanctions and contains other provisions. Official Gazette of the Union.

Brazil. (1999). Normative Instruction SDA/MAA No. 42. Amends the National Plan for the Control of Residues in Products of Animal Origin and the Residue Control Programs for Meat - PCRC, Honey - PCRM, Milk - PCRL and Fish - PCRFP. Official Gazette of the Union.

Brazil. (2022). Resolution of the Collegiate Board - RDC No. 730. Provides for the assessment of the risk to human health of veterinary drugs. Official Gazette of the Union.

Brazil. (2022). Normative Instruction No. 162. Establishes the maximum residue limits (MRLs) for active pharmaceutical ingredients (APIs). Official Gazette of the Union.

Brazil. (2017). Decree No. 9,013. Regulation of industrial and sanitary inspection of products of animal origin. Official Gazette of the Union.

Cetesb. (2022). Environmental Company of the State of São Paulo. Division of Human Toxicology and Arsenic Health. São Paulo.

Dantzger, D. D.; *et al.* (2013). Evaluation of Toxicity Diflubenzuron and p-chloroaniline in Biochemical Indicators of Fishes *Oreochromis niloticus*. In: XLII annual meeting of the Brazilian society of molecular biochemistry. Jornal da Unicamp. Foz do Iguaçu.

Efsa. (2012). European Food Safety Authority. Endocrine Active Substances.

Dantzger, D. D., Andrade, C. A., Anjos, E. F., Jonsson, C. M., & Aoyama, H. (2013). Evaluation of toxicity diflubenzuron and p-chloroaniline in biochemical indicators of fishes *Oreochromis niloticus*. Jornal da Unicamp.

Embrapa. (2023). Fisheries and Aquaculture. Available at: <https://www.embrapa.br/tema-pesca-e-aquicultura/nota-tecnica>.

Fujimoto, R. Y., Martins, M. L., Moraes, F. R., & Onaka, E. M. (1999). Use of diflubenzuron in the control of parasitic crustaceans of piauçu, *Leporinus macrocephalus*. Annals of the XI Brazilian Seminar on Veterinary Parasitology, 133.

Furlan, E. F., Vaz, F. S. (2005). Inorganic contaminants in fish. Food Technology; Physical, Chemical and Biological Topics (2st ed.).

Guidi, L. R., Silva, L. H. M., Fernandes, C., Engeseth, N. J., Gloria, M. B. A. (2015). LCMS/MS determination of chloramphenicol in food of animal origin in Brazil. Scientia Chromatographica, 7(4), 1-9.

Guo, W., He, M., Yang, Z., Lin, C., Quan, X., & Wang, H. (2007). Distribution of polycyclic aromatic hydrocarbons in water, suspended particulate matter, and sediment from Daliao River watershed, China. Chemosphere, 68(1), 93–104.

ILSI (2022). Chemical Contaminants in Food. International Life Sciences Institute.

Ikefuti, C. V. (2016). Ecotoxicological, biochemical, histo-hematological evaluation and efficacy of therapeutics in controlling diseases in pacu (*Piractus mesopotamicus*). Course completion thesis. São Paulo State University Júlio de Mesquita Filho. Jaboticabal.

Justino, C.I., Duarte, K.R., Freitas, A.C., Panteleitchouk, T.S., Duarte, A.C., Rocha-Santos, T.A. (2016). Contaminants in aquaculture: Overview of analytical techniques for their determination. TrAC Trends in Analytical Chemistry, 1(80):293-310.

Larini, L. (1999). Toxicology of Pesticides (1st ed.). São Paulo: Editora Manole.

Lima, D. P., *et al.* (2005). Heavy metal contamination in fish and water from the Cassiporé River basin, State of Amapá, Brazil. Amapá: Acta Amazônica. 45(4), 405-414.

Mapa. (2022). Manual of Good Practices in Fish Transport. Ministry of Agriculture, Livestock and Supply. (1st ed.).

Mapa. (1999). Ministry of Agriculture, Livestock and Supply: National Contaminant Control Plan PNCRC / Animal.

Majeed, S. A., *et al.* (2014). Cytotoxicity, genotoxicity, and oxidative stress of malachite green on the kidney and gill cell lines of freshwater air-breathing fish *Channa striata*. Environmental Science and Pollution Research, 21, 13539–13550.

Mantovani, D. M. B. (2005) Inorganic contaminants in the fish production chain. In: Fish Control Symposium: Quality and Sustainability. Fisheries Institute. São Paulo, 13-23.

Marin-Ramirez, J. A. *et al.* (2016). Feminization of Nile tilapia *Oreochromis niloticus* (L.) by diethylstilbestrol growth and gonadosomatic index. Ecosistemas y recur. agropecuarios, Villahermosa, 3(7), 51-61.

Martins, A. B. O. (2019). Bioaccumulation of polycyclic aromatic hydrocarbons (PAHs) in the bivalve *Crassostrea rhizophorae*: Laboratory tests and environmental application (Master's thesis). Universidade Federal da Bahia.

Montagner, C. C., Vidal, C., & Acajaba, R. D. (2017). Emerging contaminants in aquatic matrices in Brazil. Química Nova, 40(9), 1094–1110.

Moreira, R. P. L. (2012). Development and validation of multi-residue methods for determining veterinary medicines in food and feed using CL EM/EM. (Doctoral Thesis in Science – Chemistry). Belo Horizonte: Department of Chemistry, UFMG. Minas Gerais.

Mota, J.B., Neves, T.D., Costa, D.P., Starling, M.C., Amorim, C.C., Miranda-Filho, K.C. (2024) Emerging Contaminants Related to Brazilian Aquaculture. *Reviews in Aquaculture*, 17:e12967. <https://doi.org/10.1111/raq.12967>

Oceana Brazil. Independent Auditor's Report on the Financial Statements for the Year Ended on December 31st, 2020.

Roth, A. M., Baltz, D. M. (2009). Short-term effects of an oil spill on Marsh-edge fishes and decapod crustaceans. *Estuaries and Coasts*, 32, 565-572.

Sanchez, N. M. (2023). Brazilian Production of Farmed Fish Grows 2.3% in 2022, Exceeding 860 Thousand Tons. *Aquaculture Brazil*.

Silva, G. R. (2009). Levels of organochlorine pesticides and food profile in Cidade dos Meninos, Duque de Caxias-RJ, Brazil, between 2003 and 2004. Dissertation (Master's in Public Health and Environment). Sergio Arouca National School of Public Health, Oswaldo Cruz Foundation. Rio de Janeiro.

Silveira, J. G. F. (2019). Evaluation of the elimination of malachite green residues in fish using LC-MS/MS. University of São Paulo. Ribeirão Preto.

Takeshita, N., Chiste, B., Jonsson, C., Mattioli, C., & Hisano, H. (2019). Evaluation of acute toxicity of oxytetracycline in Nile tilapia postlarvae. In *Interinstitutional Congress of Scientific Initiation*, 13.

Tokumoto, T., *et al.* (2004). Diethylstilbestrol induces fish oocyte maturation. *Proceedings of the National Academy of Sciences of the United States of America*, 101, 3686–3690. <https://doi.org/10.1073/pnas.0308392100>

Vyahare, G. D., *et al.* (2018). Response surface methodology optimization for sorption of malachite green dye on sugarcane bagasse biochar and evaluating the residual dye for phyto and cytogenotoxicity. *Chemosphere*, 194, 306–315.

Wang, K., *et al.* (2020). Determination of nitrofurantoin metabolites in fish by ultraperformance liquid chromatography-photodiode array detection with thermostatic ultrasound-assisted derivatization. *ACS Omega*, 5, 3125–3133. <https://doi.org/10.1021/acsomega.9b03660>

Zanardi, M. F., *et al.* (2011). Hormone concentrations in the carcass of Nile tilapia and early maturation after sex reversal. *Brazilian Journal of Animal Science*, 40(1), 7–11. <https://doi.org/10.1590/S1516-35982011000100002>