CLEISEANO EMANUEL DA SILVA PANIAGUA
(ORGANIZADOR)

*Collection:*

# APPLIED ENVIRONMENTAL AND SANITARY ENGINEERING

Atena Editora
Ano 2022

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# PRESENTATION

The e-book:“Collection: Applied environmental and sanitary engineering” consists of nineteen book chapters that address different themes, but which converge to an enormous concern that increasingly threatens the quality and well-being of future generations: use sustainable environment and its different biotic and abiotic factors. In this sense, the organization and presentation of book chapters was carried out in four thematic areas, providing a better organization and sequencing, leading to a better understanding and ease in understanding each chapter in this e-book. Therefore, the e-book was divided into four thematic areas, namely: *i*) evaluation of the quality of water resources intended for human consumption; *ii*) emission of particulate materials from the combustion of fuels by the fleet of motor vehicles and the burning of large green areas in order to meet the interest of the agricultural sector; *iii*) actions to minimize the amount of waste sent to sanitary landfills, controlled or dumps based on the practice of segregating recyclable waste; *iv*) basic sanitation and the increase in the Human Development index, generation of energy and fertilizers from biodigestion processes and the presence of pesticides and pharmaceuticals in foods of animal origin.

The first theme consists of six book chapters dealing with the importance of continuous monitoring of water quality for drinking purposes, with studies being presented that prove the lack of efficiency in removing microorganisms with pathogenic properties. Furthermore, the importance and creation of public policies in order to avoid the eutrophication of aquatic bodies that are increasingly common in urban areas. The second consists of four chapters that evaluated the air quality from the emission of particulate materials from human activities, including the burning of fuels and fires in different biomes and how these have been influencing the increase in the formation of islands of heat in urban centers.

The third theme consists of four book chapters that address the importance of carrying out the construction of residential works (condominiums) in order to encourage residents to develop an environmental awareness in relation to the segregation of waste, especially organic and recyclable ones, and the latter would be intended for people who work and with recycling and who contribute significantly to the reduction in the final disposal of waste. Finally, the fourth theme consists of five chapters that present works that discuss the importance of biodigestion in rural areas, basic sanitation as an important factor in determining the HDI and the importance of monitoring the presence of pesticides and drugs in food of animal origin.

In this perspective, Atena Editora has been working with the aim of stimulating and encouraging researchers from Brazil and other countries to publish their work with a guarantee of quality and excellence in the form of books and book chapters that are available on the Editora’s website and elsewhere. digital platforms with free access.

Cleiseano Emanuel da Silva Paniagua

## SUMÁRIO

# CAPÍTULO 19

# DETECTION AND QUANTIFICATION OF MULTIRESIDE PESTICIDES AND PHARMACEUTICALS IN FOODS OF ANIMAL ORIGIN USING THE QUECHERS METHOD IN PREPARATION OF SAMPLES

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**ABSTRACT:** Contaminants of Emerging Concern (CECs) began to be detected and quantified in the 1990s, after the advent and improvement of analytical techniques capable of quantifying on trace ($\mu$g/L) and ultra-trace (ng/L) in aqueous matrices. The unrestrained growth of the human population and the need to increase the production of food of plant and animal origin, led to the improvement of methods for sample preparation and multi-residue determination such as QuEChERS (Fast, Easy, Cheap, Effective, Robust, Safe). Therefore, the detection and quantification of CECs residues in living organisms used for human consumption raised an alert by developed countries that aroused the interest of the scientific community and enabled both the inclusion of new contaminants, as well as the change of Maximum Allowed Values (MAV) by environmental agencies in the United States (USEPA), European Union (EU). Asian countries that started to require agricultural commodities destined for human consumption to present a quality assurance seal in relation to waste MAV in accordance with legislation. In this sense, this work aims to present and discuss several studies that prove the presence of pesticide and pharmaceuticals residues in foods of animal origin, present in human food.
**KEYWORDS:** Contaminants Emerging Concern, trace and ultra-trace analysis, food, multi-waste and agricultural commodities.

## 1 | INTRODUCTION

The unrestrained and disorganized

growth of the world population has been contributing to the greater extraction of minerals from resources and foods of plant and animal origin and their derivatives. The production of fish, cattle, pigs and poultry (chickens) in 2020 is illustrated by the graphs in Figure 1.

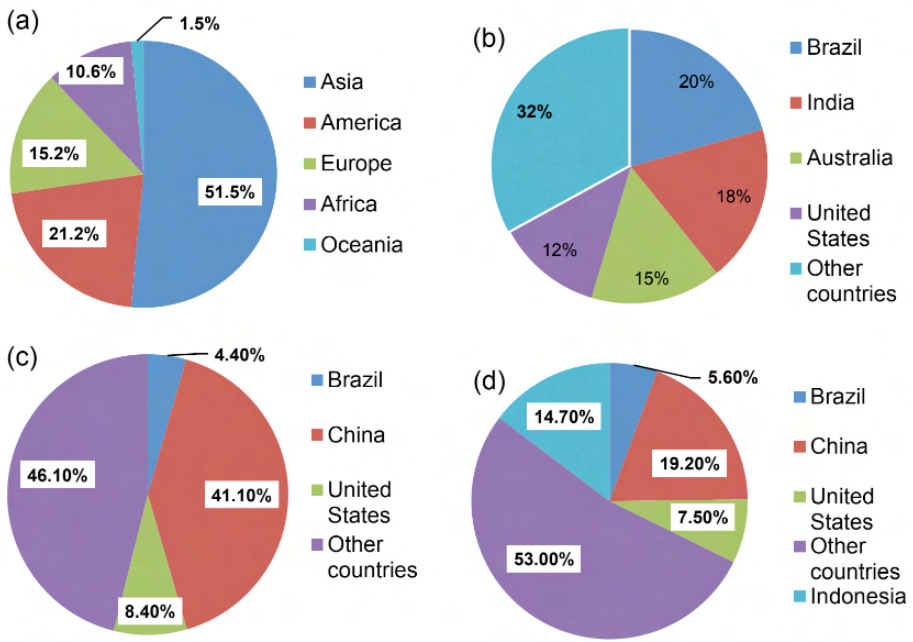


Figure 1: (a) World fish production; (b) beef; (c) swine and (d) birds (chicken) in 2020

Source: EMBRAPA (2020).

These percentages are equivalent to: *i*) 987.5 million (beef); *ii*) 103 million (swine); *iii*) 133.3 million (chickens) and; *iv*) 179 million tons of poultry (chickens). The perspective is that these values will increase annually due to the higher consumption of a population that grows at a fast pace and that until December 30, 2021; reached a population of 7.9 billion people, according to Worldometer.

Therefore, there is an enormous need to produce more and more in a shorter period of time, using substances such as drugs and pesticides that reach the animals through the following ways: *i*) application of medicines and vaccines in different herds in order to ensure the animal's health; *ii*) administration of drugs with different properties in order to ensure the acceleration of the animal's development for slaughter; *iii*) drugs and pesticides present in the composition of administered feeds and supplements; *iv*) use of pesticides to reduce and/or eliminate pests that attack different breeding stocks (ARISEKAR. et al. 2019; BARBIERI et al., 2019; CASTRO; POCURULL; BORRULL, 2020; HAN et al., 2021). In this way, drugs and pesticides are eliminated in the environment through different routes, as shown in the flowchart in Figure 2.

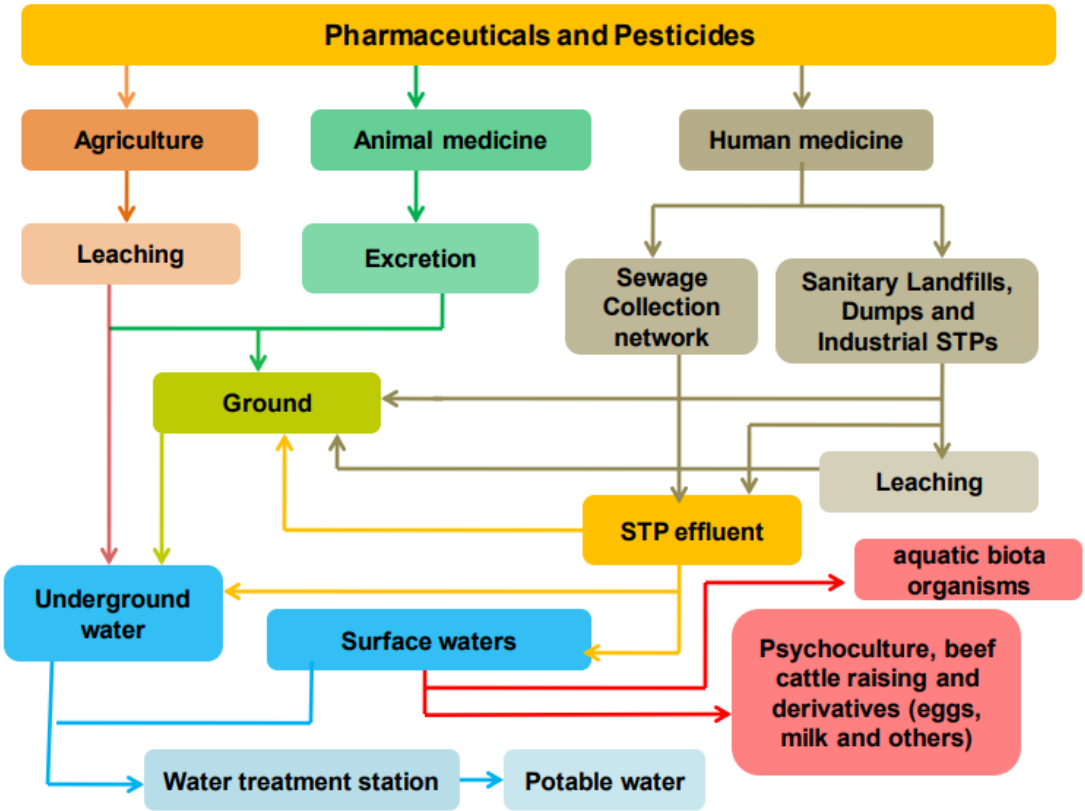


Figure 2: Possible routes of entry and distribution of pharmaceuticals and pesticides in the environment that affect animals destined for slaughter

Source: The authors (2021).

## QuEChERS Method

The growing demand for food of plant and animal origin due to population growth in a disorderly way, requires the production of food on increasingly larger scales in an increasingly shorter time span. Therefore, quality control in relation to the presence of multi-residues from pharmaceuticals and pesticides generated the need for the search for sample preparation methods that were faster, lower costs, reduced solvent use, greater versatility and diversity to analyze a greater number and variety of samples (HAN et al., 2021; LI et al., 2021; MNYANDU; MAHLAMBI, 2021; PANIAGUA, 2021a; PANIAGUA; SANTOS, 2021a).

Over the past 25 years, the scientific community has dedicated itself to the search for a method of sample preparation that contemplates the characteristics described above, as consumers started to demand foods with less diversity and quantity of residues in order to guarantee greater food safety and a greater balance for the environment. Therefore, methods capable of determining residues in low concentrations in samples of high

complexity were emerging, being necessary to promote a sample preparation that aims: the removal of interferents, the promotion of extraction and enrichment of the analytes of interest (PANIAGUA, 2021a; PANIAGUA; SANTOS, 2021a; PRESTES; ADAIME; ZANELLA, 2011).

Faced with this challenge, Anastasiades et al. (2003) presented na extraction method, based on adjusting the ionic strengt of the sample solution, inthe middle of a mobilie-phase chromatographic, by additing suitable salts, and thus causing thus causing the salting-out effect capable of extracting, in principle, a variety of pesticide residues. This method was called QuEChERS, na acronym for: Quick, Easy, Cheap, Effective, Rugged and Safe, which can be presented in simplified form in the flowchart of Figure 3.

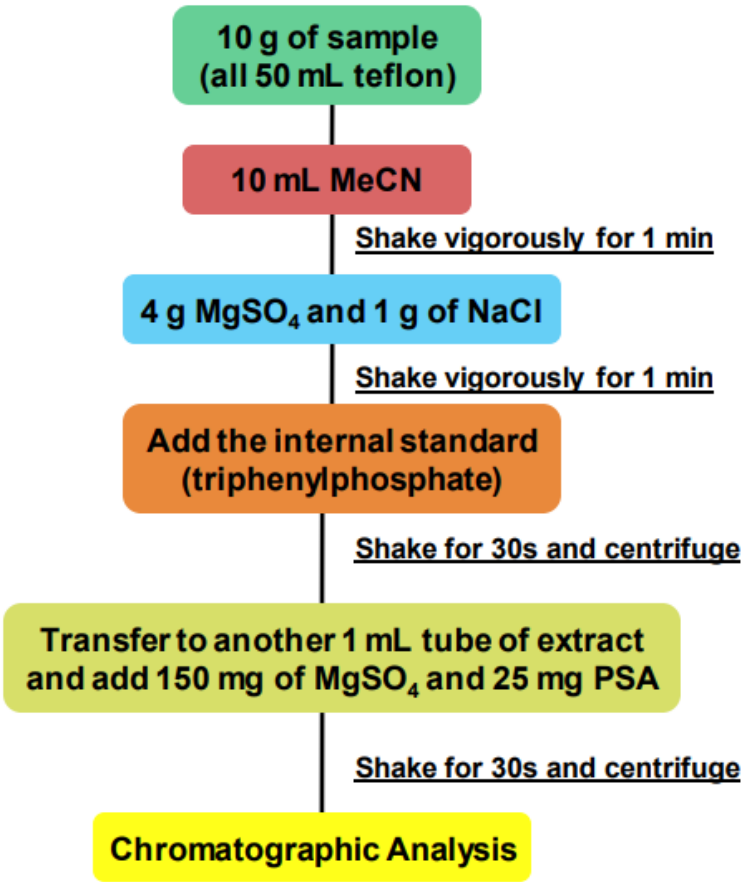


Figure 3: Representative flowchart of the original QuEChERS method

Source: Adapted from Anastassiades et al (2003).

The QuEChERS method has a sample:solvent ratio of 1 g/mL when compared to traditional methods that demand a ratio of 2 to 5 g/mL using nonpolar solvents. Since then,

this method has been increasingly applied to samples of different nature and complexity, such as: pharmaceuticals and pesticides in water, honey and bees, plant foods (PANIAGUA, 2021a; PANIAGUA; SANTOS, 2021a) and those of animal origin that will be presented and discussed in this work.

## 2 | LITERATURE REVIEW

The present work was limited to presenting and discussing some results obtaining using QuEChERS method, regarding the presence of pesticide and pharmaceuticals residues found in samples of food of animal origin (fish, cattle, pigs and chickens) between the years 2017 to 2021. The works carried out to be represented only a small portion in relation to the quantity and diversity of the world. It is not the objetctive of this work to exhaust the presentation and discussion of the topic under discussion. Furthermore, the works seek to present the importance of the QuEChERS method in the determination of multi-residues in the different samples that were the objetc of investigation in this work. Therefore, Tables 1 and 2 present some works and their main results, as presented and highlighted below.

| Result | County | Reference |
|---|---|---|
| From a QuEChERS method developed to determine 340 types of pesticides in samples from fish muscle and liver with a detection limit (from 0.05 to 1.2 $\mu$g/kg). In 54 fish samples, ten types of pesticides were detected at concentrations ranging from 5 to 47 $\mu$g/kg, being superior to the proposed method. | Poland | [KAZYNSKI et al., 2017] |
| A QuEChERS method was applied to determine 13 organochlorine pesticides in fish muscle samples. Being quantified a concentration in a range of 1 to 100 $\mu$g/kg, being below the limit of quantification (LOQ) of the proposed method (1.94 to 4.93 $\mu$g/kg). | China | [WANG et al., 2017] |
| From the development and validation of a QuEChERS method to quantify 80 types of pharmaceuticals for veterinary use in psyculture for the species *Oplegnathus punctatus*. The results showed a concentration below the LOQ (0.25-25 $\mu$g/kg) of the validated method. | China | [ZHÃO et al., 2017] |
| The pharmaceutical sulfamethazine (SMZ) was administered to tilapia (Oreochromis niloticus) for 11 days at a concentration of 422 mg/kg using a QuEChERS method. After 10 days, the Maximum Allowed Value - MAV - (100 mg/kg) was reached in accordance with European Union legislation. In 10 days there was an accumulation of SMZ in the Tilapia muscle in the order of 1.6 mg/kg, which allowed us to infer that SMZ is rapidly excreted by the fish's body. | Brazil | [NUNES et al., 2018] |
| A QuEChERS method was applied in the determination of four tetracyclines in fish samples (salmon and panga) that presented concentrations below the LOQ (4.4g/kg) of the proposed method. | Spain | [GRANDE-MARTINEZ et al., 2018] |

| | | |
|---|---|---|
| A QuEChERS method was applied to determine pesticides in fish samples. The results showed a concentration of 0.01 to 26.05 $\mu$g/kg (fish muscle); 0.01 to 40.56 $\mu$g/kg (in the gills) and 0.01 to 65.14 $\mu$g/kg (liver), which are above the established by the World Health Organization (WHO). | Índia | [ARISEKAR et al., 2019] |
| A QuEhERS method was developed to determine five types of ivermectin and a milbemycin with a quantification limit, on average, from 0.07 to 1.3 $\mu$g/kg, according to European legislation. The methodology was applied to fish samples, with values below the method limit being quantified. | Greece | [MOSCHOU;DASENAKI; THOMAIDIS, 2019] |
| A QuEChERS method was developed for 52 types of pesticides in fish samples with a limit of quantification of 10 ng/g. When applying the method, eight pesticides whose concentration was below the limit of the proposed method were quantified. | Spain | [BARBIERI et al., 2019] |
| From a proposed QuEChERS method to determine organophosphate esters (OPEs) in fish samples. The method has a limit of quantification from 0.05 to 2.0 ng/g which after being applied to fish samples obtained 5.31 ng/g, above that established by the method. | Spain | [CASTRO; POCURULL; BORRULL, 2020] |
| A QuEChERS method validated for 219 target compounds, detecting six pesticides and one PAH in 22 fish samples that presented values below those established by USEPA. | United States | [HANA; SAPOZHNIKOVA, 2020] |
| A QuEChERS method was developed for detection and quantification of 66 pesticides. When applied to 78 fish samples, it was tested positive for 10 samples, whose concentrations were below the Quantification Limit of the method (10 µg/kg). | Republic of Korea | [SHIN; KIM; KANG, 2021] |

Table 1: Some works involving the QuEChERS method to monitoring residues of pesticides and pharmaceuticals present in fish samples in the period between 2017 and 2021

Source: The authors (2021).

Tabele 1 was limited to presenting works carried out with fish samples due to the high amount and diversity of pesticidde and pharmaceuticals residues present in these samples, obtained from the commerce of different cities in numerous countries. The results reveal the contamination of different aquatic biota, since fish is one of the countless living organisms that inhabit these ecosystems and that may be an indication that multi-residues have already contaminated countless fauna and flora organisms from different organisms. aquatic compartments. Therefore, water resources are the main entry point for multi-waste that contaminate the environment and that, due to different trophic levels, can accumulate in increasingly higher concentrations along the food chain and in increasingly complex organisms as shown in Table 2.

| Result | Country | Reference |
|---|---|---|
| A QuEChERS method was applied for the determination of multiresidues of β-antagonist substances in muscles and viscera of animals, with a limit of quantification (LOQ) of 1.0 $\mu$g/kg. The analyzed samples presented values that were below the LOQ of theproposed method | Taiwan | [LIN et al., 2017] |
| Using a QuEChERS method, the presence of drugs (avermectins, benzimidazoles and nitroimidazoles) was evaluated in tissue samples from bovine muscle, with a limit of quantification from 0.011 to 113.674 $\mu$g/kg. In all samples evaluated, the concentration of pharmaceuticals was below the LOQ of the proposed method. | Brazil | [SILVA et al., 2017] |
| A QuEChERS method was applied in the determination of fenobucarb residues in food samples of animal origin (swine muscle, fish, eggs and milk) with a limit of quantification of 2.0 $\mu$g/kg. In samples acquired in different places in the country, the target compound of interest was not detected. | Republic of Korea | [ZHENG et al., 2017] |
| From the application of a QuEChERS method, ractopamine was determined in samples from different parts of the pig (muscle, kidney, liver and fat) with quantification limits from 3.65 to 4.86 µg/kg (kidney samples) and from 6.27 to 7.21 µg/kg (liver samples), which are values far belowthe established by Codex Alimentarius, which is 90 (kidney) and 40 µg/kg (liver). Numerous samples were collected from different places with a concentration of 22.5 (liver) detected; 92 (kidney) and 1003 µg/kg (lung) of the pig. The results indicate that for kidney and lung samples from pigs, they are above both the proposed method and the one established by Codex. | Brazil | [FEDDERN et al., 2018] |
| A QuEChERS method was developed for the determination of 200 pesticides and 65 environmental contaminants in bovine, porcine and poultry muscle samples with a limit of quantification of 5.0 ng/g for 219 target compounds. When applying to samples, none were found with compounds within the quantification limit of the method. | United States | [SAPOZHNIKOVA, 2018] |
| A QuEChERS method was developed and applied for the determination of the pesticide chlordecone and its metabolite chlordecol in animal liver samples. In twelve samples of bovine and porcine liver, concentrations above the detection limit for chlordecone (1.36 $\mu$g/kg) and below for the metabolite chlordecol (2.50 $\mu$g/kg) were found. | France | [SAINT-HILAIRE et al., 2018] |
| A QuEChERS method, with a detection limit of 10 $\mu$g/kg, was developed for the determination of endrin and its metabolite (δ-keto endrin) in five types of animal samples (chicken, swine, bovine, egg and milk), being that the maximum value allowed by CODEX Alimentarius is 10 times the method limit. In 20 samples collected in different places in the country, the target compounds were not detected. | Republic of Korea | [RAHMAN et al., 2018] |
| A QuEChERS method was developed to determine 126 pesticides in chicken muscle, with a limit of quantification of 10 $\mu$g/kg. The evaluated samples showed concentrations below the method limit, with the exception of the pesticides oxadiazone, clomazone and lactofen. | China | [WENG et al., 2020] |

Table 2: Some works involving the QuEChERS method to monitoring residues of pesticides and drugs present in samples of pork, beef and poultry (chicken) from 2017 to 2020.

Source: The authors (2021).

The results presented in Table 2 demonstrate that the residues of drugs and pesticides are present in the extensive activities of raising and slaughtering poultry, cattle and swine for commercial purposes, the multi-residues that were detected are mostly from the following sources: *i*) pharmaceuticals used for the best development of the animal for slaughter; *ii*) pesticides used both to control pests that infect the herd and can cause the animal to become ill, and from the ingestion of animal feed and/or foods of plant origin, such as in cattle breeding.

Given these results, it is necessary to adopt practices and establish international legislation in order to reduce the indiscriminate use of pharmaceuticals and pesticides in the production of food of animal origin. In addition, the excretions of these animals when in contact with the soil and through leaching are capable of reaching different aquatic compartments [ground and surface water (stream, river, pond, dam, headwaters, among others)] that will reach the water and/or sewage treatment plants and will not be removed by conventional treatment processes, as they were not designed for a specific purpose (PANIAGUA, 2021a,b; PANIAGUA; SANTOS, 2021b). The treated water will be destined for public supply, in which man will be contaminated by multi-waste from another source.

## 3 I CONCLUSION

The presence of drug and pesticide residues in samples of animal origin needs to be urgently addressed by international organizations, in order to curb the indiscriminate use and management in all countries and with the possibility of suffering sanctions in relation to consumption and export of these foods.In addition, the health of the environment and greater food safety need to be addressed in the form of international agreements as a matter of urgency, since such indiscriminate and even illegal use of drugs and pesticides are directly impacting the health of people who they live in countries where the resource destined for this area is little or scarce, such as Brazil.

The QuEchERS method has been helping and providing an even greater advance in the preparation of increasingly complex samples. This method has contributed to the reduction of time, energy consumption, reagents and waste generation. The versatility of this method has enabled the development of specific QuEchERS for a certain class of residues and samples, enabling an expansion in the detection and quantification of residues, contributing to the solidification of the twelve principles of Green Chemistry.

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## SOBRE O ORGANIZADOR

**CLEISEANO EMANUEL DA SILVA PANIAGUA** - Technician in Chemistry from the Professional College of Uberlândia (2008), Bachelor of Chemistry from the Federal University of Uberlândia (2010), Degree in Chemistry from the University of Uberaba (2011) and in Biological Sciences from the Faculdade Única (2021). Specialist in Teaching Methodology of Chemistry and Teaching in Higher Education at Faculdade JK Serrana in Brasília (2012), specialist in Teaching Science and Mathematics at Instituto Federal do Triângulo Mineiro (2021). Master in Chemistry from the Federal University of Uberlândia (2015), with emphasis on development of bioadsorbent to remove As (V), Sb (III) and Se (IV) ions in different aquatic matrices. PhD in Chemistry from the Federal University of Uberlândia (2018), with emphasis on Advanced Oxidative Processes [heterogeneous photocatalysis ($TiO_2$/UV-A and $TiO_2$/Solar) and $H_2O_2$/UV-C for removal of contaminants of emerging cocern (CEC) in different aquatic matrices. She carried out her first Post-Doctoral internship (from May 2019 to June 2021) at the Federal University of Uberlândia with an emphasis on the application of new oxidizing agents using solar radiation to remove CEC in effluents from a sewage treatment plant. He is currently carrying out his second Postdoctoral Internship (July 2021 - current) at UFU in the same line of research. Degree in Physics at Faculdade Única (September 2021 to November 2022), studying *lato sensu* postgraduate studies in Advanced Instrumental Analysis at Unileyva College. He has 11 years of experience as a chemistry technician at the Federal Institute of Goiás, having acted as responsible for the analysis of physical-chemical and biological parameters of water and effluents from a sewage treatment plant. It operates in the following lines of research: (*i*) Development of new methodologies for the treatment and recovery of chemical waste generated in the laboratories of teaching and research institutions; (*ii*) CEC monitoring studies; (*iii*) Development of new advanced technologies for CEC removal in different aquatic matrices; (*iv*) Application of advanced oxidative processes ($H_2O_2$/UV-C, $TiO_2$/UV-A and photo-Fenton and others) to remove CEC in effluents from a sewage treatment plant for reuse purposes; (*v*) Study and development of new bioadsorbents for environmental remediation of CEC in different aquatic matrices; (*vi*) Environmental Education and; (*vii*) literacy and scientific processes in the area of Science of Nature, especially biology and chemistry.

## ÍNDICE REMISSIVO

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