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Circular Economy in the Biosolids Management by Nexus Approach: A View to Enhancing Safe Nutrient Recycling—Pathogens, Metals, and Emerging Organic Pollutants Concern

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Abstract: Biosolids are a byproduct of sewage treatment that can create synergies and opportunity costs for promoting a circular economy and the nexus approach (water, energy, and food). They enable a cleaner agricultural production, with food safety in local development. The biosolids contain nutrients that can be recycled by agricultural soils. However, they contain heavy metals and few studies report the micropollutants present and the legal requirements of different countries (policies). The present study aimed to contribute to the knowledge of the composition and characteristics of biosolids during four years of monitoring (2016–2019). We investigated the agronomic potential of biosolids in a sequencing batch reactor. The content of biosolids in the crops studied is a potential source of macronutrients, especially N, P, and S. Pathogens fell into class B for Conama 498 (Brazil), Norm 503 (USA), and Directive 86/278 (EU) relative to *Escherichia coli* and enteric viruses. Metals, also compared with the three previous standards, fulfilled threshold concentrations of the respective legislations. Emerging organic pollutants remained below the detection limit, except naphthalene, which a single time was found in the biosolids above the detection limit. Finally, PCA showed that the chemical elements of the biosolids do not vary significantly relative to changes in tropical climatic conditions (resilience to climate change). Our study confirms the safe biosolids' agronomic potential in promoting a circular economy in wastewater treatment plants. In line with a cleaner agricultural production in tropical soils, complying with the legislation on micropollutants and reducing the quantity of biosolids sent to landfill, or inadequately disposed of in the environment.

Keywords: agricultural recycling; sludge; sustainable sanitation

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1. Introduction

The estimate is that the world urban population will reach 9.7 billion inhabitants in 2050 [1], and the high demand for food, which shall double agricultural production and consequently the search for supplies, especially nutrients, is concerning [2]. Yet, there is still a need for basic water, sanitation, and hygiene (WASH) conditions, which are essential to improve the health of the population, especially in developing countries [3]. In this sense, it will result in the construction of more wastewater treatment plants (WWTPs), as established by the Clean Water Act of 1972, in the United States, which proved to be an important step in protecting human health and integrity of the environment [4]. Water and sanitation are under the United Nations Sustainable Development Goal 6. Achieving this goal is recognized as a precursor to achieving several other goals. For example, resource-recovery sanitation systems can help achieve SDG 2 (end hunger) and SDG 12 (sustainable consumption and production). However, it is commonly understood that sewage treatment systems have units that generate waste and the ordinary used method for sludge disposal is landfilling.

The fact is that this waste can be considered a byproduct (biosolids), which may be used in agricultural soils or in restoring degraded lands, to avoid losses of valuable nutrients. Circular economy approaches provide a better way for utilizing these resources in a sustainable manner [5]. Biosolids are organic waste, from the generated and treated sludge, which is common in treating sewage, and have complex composition, with essential nutrients for plants, beneficial and/or potentially harmful/toxic chemical substances, such as the micropollutants that have received attention in recent years, beyond pathogens [6]. To promote the recycling and recovery of biosolids (and other wastes), the European Commission adopted the Circular Economy Package. The goal of a circular economy is closing the loop of product lifecycles, adding value for as long as possible and eliminating waste with benefits for the economy and environment [7].

In this sense, the use of biosolids in agriculture by nexus approach (water–energy–food), especially in developing countries, has been a challenge for managers of the sector. One difficulty is the scarcity of studies and technology adequate to this local reality that can confirm the technical, economic, and environmental viability of biosolids, especially before the law, which defines criteria and procedures for applying biosolids in soils [8]. Furthermore, there is still rejection by society, which is unaware of the biosolids' potential [7], even if the characteristics are beneficial for soil and agriculture, with safety for the health of the population and to the environment. Nonetheless, the criteria and procedures defined in rules and laws make the application unviable in some cases [9]. The fact is that in respecting criteria ensuring the quality of the biosolids before performing applications, the confirmed results are positive and the application in agricultural soil is the best strategy for the sustainable management of agriculture by way of this byproduct of the treatment of sewage.

Therefore, one should study the limitations and different existing conditions to make the application of biosolids in agriculture viable [10,11]. It is known that in the history of humanity, after the invention of agriculture, the exploration of valuable resources embedded in human and animal excrements, such as urine [12] or even agro-industrial waste [13], and their application as fertilizers in the land used for agricultural production [14] were frequent. Recently, there is a concern associated with emerging organic pollutants (EOPs), which extends, too, to the already known inorganic contaminants (metals) and pathogens [6].

There is concern about heavy metals accumulation and their toxic effects at the soil–water–plant interface, with groundwater contamination. However, several studies have documented that the content of metals in biosolids is lower than the concentration of metals in organic waste from animals that are used on a large scale in agriculture as fertilizer [7,10,11,15,16]. Studies present that if the criteria and procedures of the law are followed, the risk of soil contamination or transport of pollutants into deep waters is inhibited [9], for most metals are known to be used by plants as micronutrients [10,17,18].

With regard to emerging organic pollutants (EOPs), their primary escape route within the sewage treatment system is the sorption to the biosolids. Those organic compounds regularly enter in WWTPs and include hormones, pharmaceutical compounds, personal hygiene products, household chemicals, plasticizers, surfactants, flame retardants, and pesticides, among others [19]. According to [20], polycyclic aromatic hydrocarbons (PAHs) and persistent organic pollutants (POPs) groups are the most frequent in biosolids. Although, a study in eight WWTPs, between the years 1993 and 2012, observed concentrations below the detectable limit in biosolids [21]. Therefore, one should monitor and seek knowledge about those micropollutants' behavior, in that, once bioaccumulation occurs in the soil, the risks of chronic or transgenerational effects on the environment and population may be a negative aspect of the application of biosolids.

The fact that the sludge and biosolids management options are burdensome, in that around 50% of operational costs at WWTPs, especially of activated sludge, are related to the management of this byproduct [22], also stands out. In Brazil, for example, almost the whole of biosolids generated in WWTPs have as their final destination the sending to landfills [23], which aggravates the environmental problem even more because it reduces the useful life of the landfills, when they exist. In this context, the agricultural recycling of biosolids may benefit the environment, reducing the use of fossil fuels in the production and transport of fertilizers/agricultural commercial inputs. Moreover, the exploration in natural reserves of nutrients decreases; nutrients such as phosphorus, for which the forecasts point out depletion in the next years [24].

In this context, information concerning the chemical and microbiological characteristics of these byproducts, which may be used in future studies, as in the quantitative assessment of chemical and microbiological risks and agronomic potential, for agricultural purposes and new business models, becomes necessary. Especially because the WWTPs have different processes, which generate byproducts with different characteristics and real-scale cases, such as this, are important for better understanding this type of intelligent and safe strategy for biosolids, and fundamental for food safety.

In light of these observations, the present study aimed to assess in an integrated way the chemical and microbiological characteristics of biosolids, the agronomic potential on the basis of the content of N, P, K, Ca, Mg, and S macronutrients, as well as on the determination of pathogen, heavy metal (micronutrients), and EOP concentrations, including a multivariate principal component analysis (PCA), relating the parameters and the different times of the year and different climatic conditions. The intention is to contribute with information to agricultural recycling of nutrients stemming from biosolids of a WWTP by aerobic process (modified activated sludge), to subsidize new public policies (based on the nexus approach) in similar regions that use the same technology. In addition, to integrate this view into a circular economy model to increase productivity and safety for workers in this trade and end-consumers.

2. Materials and Methods

2.1. Study Area

The present study was carried out in the Imbirussu Wastewater Treatment Plant (IBRS WWTP), located in the city of Campo Grande (capital of Mato Grosso do Sul), center-west of Brazil (20°26′37″ S, 54°38′52″ E, 612 m of altitude, 900,000 thousand inhabitants—as informed by the company responsible for the sewage treatment service in the municipality). The IBRS WWTP (Figure 1) receives 10% of the sewage collected in the Mato Grosso do Sul capital and treats effluent through two aerobic, sequencing batch reactors, with the capacity to treat 60 L s^{-1} each (120 L s^{-1} total). The sludge subsided at the bottom of aerobic reactors (SBR—sequencing batch reactors), is pumped into a sludge thickening tank (gravity thickener), and after settling of the water and sedimentation of the sludge, finally, the byproduct is pumped into an aerobic digester tank. After sedimentation, with a solids content between 1% and 2%, it is disposed of in the soil by an irrigation system.

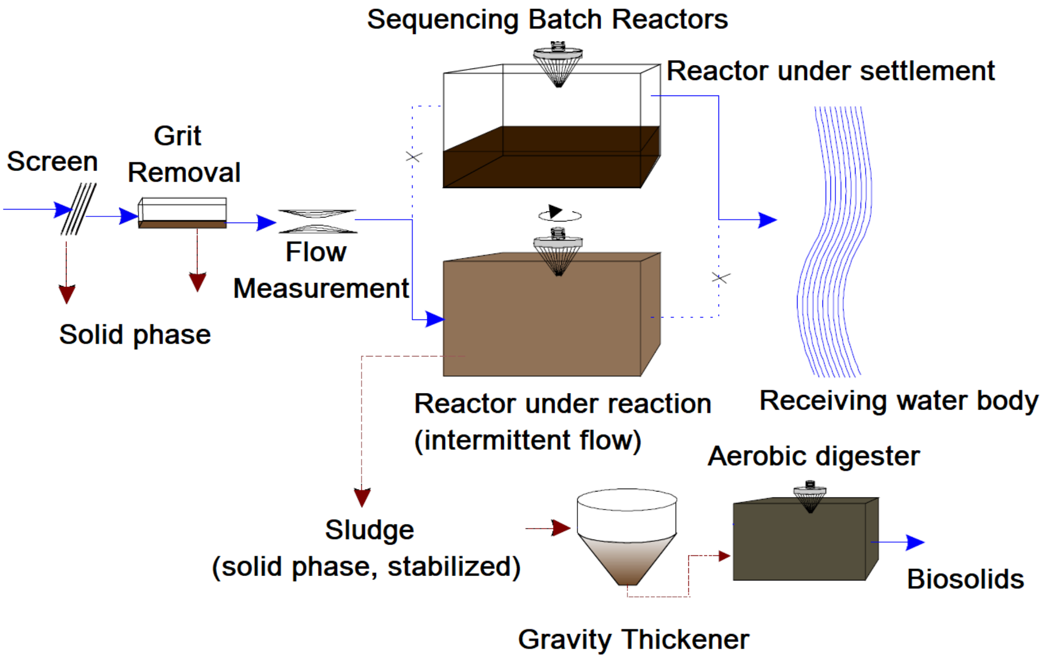

Figure 1. Sewage treatment system of the Imbirussu WWTP (adapted from [25]).

2.2. Analyses and Samples of Biosolids

The data of the present research were obtained through official quarterly reports of self-monitoring of the IBRS WWTP biosolids. Quarterly samples were collected and sent for analysis at a specialized laboratory, four samples were collected (every 3 months) during the years 2016, 2017, 2018, and 2019. The generated biosolids differed in relation to climate, in that the quarterly analyses were carried out with samples that contained summer, spring, winter, and autumn biosolids.

The samples were taken to specialized laboratories using methods prescribed by USEPA (1994), which the CONAMA 375/06 revoked resolution already recommended, and that were kept in the new Resolution 498/20 [26,27]. The assessed chemicals were N Kjeldahl (SMEWW, 22° ed., 2012, 4500-Norg B POP-DAM 106 method), P, K, Ca, and Mg (U.S EPA SW-846), and S (external ref.: CRL 0172: U.S EPA methods 5050 and 300.0). For determining the metals As, Ba, Cd, Cr, Cu, Hg, Mo, Ni, Pb, Se, and Zn the 6010C method was used, as established in U.S EPA SW-846 [27,28]. *E. coli* (POP PA 040), viable helminth eggs (U.S EPA 625/R-92/013), *Salmonella* sp. (IOP—A 5.089 ver. 02), EOPs (ver. 03 and U.S EPA 8270 D ver. 04:2007), and dioxins and furans (U.S EPA 023A/U.S EPA 8290A: 2007) were also assessed.

Quality Assurance and Quality Control (QA & QC)

The company's and contracted (outsourced) laboratories follow international quality protocols (ISO 9001), including the collection procedure. The laboratories are certified by ISO 9001 and also by the local environmental agency that receives this information according to monitoring. Chain of custody (CoC) is included.

2.3. Reference for the Agronomic Potential of Biosolids

In the present research, we seek to know the percentage of N, P, K, Ca, Mg, and S in the IBRS WWTP biosolids for the following cultures: corn, rice, wheat, sugar cane, forage grasses, beans, coffee, and cotton [29], as per Table 1. In parallel, for purposes of comparison of the content of macronutrients present in the IBRS WWTP biosolids, we also show the content of macronutrients from biosolids of other tropical climate WWTPs, in addition to other organic residues, such as pig manure, cattle manure, chicken manure, and sheep manure.

Table 1. Production expected per 1 ha of cultivation and macronutrients demanded. Source: [29].

Culture	Quantity of Production	Macronutrients
Corn	5 tons of grains	106 kgN ha^{-1}; 45 kg P ha^{-1}; 106 kg K ha^{-1}; 19 kgCa ha^{-1}; 26 kgMg ha^{-1}; 13 kgS ha^{-1};
Rice	3 tons of grains	103 kgN ha^{-1}; 39 kgP ha^{-1}; 144 kg K ha^{-1}; 27 kgCa ha^{-1}; 9 kgMg ha^{-1}; 12 kgS ha^{-1};
Wheat	3 tons of grains	56 kgN ha^{-1}; 21 kgP ha^{-1}; 101 kg K ha^{-1}; 7 kgCa ha^{-1}; 9 kgMg ha^{-1}; 8 kgS ha^{-1};
Forage grasses	1 ton dry matter	17 kgN ha^{-1}; 4 kgP ha^{-1}; 27 kg K ha^{-1}; 5 kgCa ha^{-1}; 3 kgMg ha^{-1}; 1 kgS ha^{-1};
Sugar cane	100 tons of stalks	150 kgN ha^{-1}; 46 kgP ha^{-1}; 201 kg K ha^{-1}; 100 kgCa ha^{-1}; 52 kgMg ha^{-1}; 45 kgS ha^{-1};
Beans	1 ton of grains	110 kgN ha^{-1}; 22 kgP ha^{-1}; 119 kg K ha^{-1}; 58 kgCa ha^{-1}; 19 kgMg ha^{-1}; 26 kgS ha^{-1};
Coffee	3 tons of grains	157 kgN ha^{-1}; 23 kgP ha^{-1}; 187 kg K ha^{-1}; 63 kgCa ha^{-1}; 30 kgMg ha^{-1}; 10 kgS ha^{-1};
Cotton	750 kg fiber	90 kgN ha^{-1}; 32 kgP ha^{-1}; 42 kg K ha^{-1}; 15 kgCa ha^{-1}. 12 kgMg ha^{-1}. 10 kgS ha^{-1}.

2.4. Legal Requirements of the Biosolids Viability—Policies

For analyzing the legal requirements, we analyzed the Brazilian, USA, and European Union policies. The presence of pathogenic microorganisms was relative to the limits established by Conama Resolution 498/20 [27], Norm 503 [28], and Directive 86/278 EEC [30]. The same reference base was adopted for evaluating inorganic contaminants (metals). For EOPs, we used only the Conama Resolution n° 375/06 [26] (repealed) and EU Directive 2007—EUR 22,805 EN [31] since the other legislation did not include them and the values present in the resolutions are related to EOP concentrations in the soil. The Conama Resolution n° 375/06 [26] was repealed and currently the Conama Resolution n° 498/20 [27] is in vigor. The new resolution broadens the opportunities to use sewage sludge in soils, which after treatment becomes the named biosolids [15]. Yet, for comparison with regard to EOPs, we considered the previous resolution since the new one does not deal with those substances.

2.5. Principal Component Analysis (PCA)

Multivariate analysis, as a tool that allows verification of the interrelationships with a large number of variables, is known for its varied applications, including studies related to biosolids [32–34]. According to [35,36], this technique allows for analyzing sets of complex data without information loss. The interpretation of the analysis occurs via the observation of the angular measure formed between the data, where the weight of the variable lies further from the largest center. The larger an angle a lower correlation and the lower angle formed indicative of a high correlation, as opposite variables, too, are inversely correlated [37]. From linear combinations between variables, it seeks to reduce, as well as to eliminate data for better representativeness, which provides a smaller set of variables that is called components. Quantification of the significance of variables allows for an explanation of the groupings, in which new variables, called orthogonal, are explained by a set of data not correlated with each other and reduced, which are the principal components (PCs). Thus, our focus was to analyze whether there was a significant difference or not, between chemical elements and pH within the categories of seasons of the year in the tropical climate.

Different from other types of climates, the seasons of the year boil down to two in tropical climates: a rainier and warmer one (summer and spring) and another drier and colder (winter and autumn). Although one usually uses the name “summer” and “winter”, these seasons are broadened and encompass months from “spring” and “autumn” [38]. Yet, from the practical point of view, there exist only two seasons per year. A rainy season and a dry season, in countries of tropical climates. We include the four seasons matter as categorical columns in PCA because the biosolids generation occurs in different seasons. In addition, we tested the hypothesis that the chemical elements may differ in sensitivity to the seasons of the year and these environmental conditions may take up the role of variable for changes in the concentrations of the chemical elements.

3. Results and Discussion

The results are discussed according to (i) agronomic potential, (ii) pathogenic microorganisms, (iii) inorganic contaminants, and (iv) EOPs in a (v) comparison between the policies of Brazil, the USA, and the European Union. At the end, the principal component analysis (PCA) presents chemical characteristics of the biosolids of greater relevance relative to climatic variations (seasons of the year), with data from four years of monitoring in the tropical climate.

3.1. Agronomic Potential

3.1.1. Macronutrients of the Biosolids and Animal Waste

The average concentration of macronutrients (kg ton^{-1}) in the biosolids (Table 2) of the present research, throughout four years of monitoring (2016 until 2019), by and large, is higher than the values for WWTPs in South Africa (also of tropical climate) and of organic residues from animals such as manure of pig, cattle, chicken, and sheep. However, it was lower than in other WWTPs in Brazil. This variability is common and is reported in the literature [8–10,14,15]. This reveals the nutritional and consequently agronomic potential of Brazilian biosolids but also attention that must be given to agronomic projects, taking into consideration and giving due attention to each type of macronutrient of interest. Therefore, studies that provide information on the quality of biosolids from different effluents (domestic, agro-industrial) and processes (anaerobic, aerobic) are necessary.

Thus, some details are important. For example, for nitrogen and phosphorus, the present research obtained values similar to those in other WWTP in Brazil and South Africa, considering standard deviation, but the concentrations are significantly higher from WWTP than the organic waste of animals. However, with regard to phosphorus, we highlight that when comparing values of the present research (WWTP—aerobic system with biological removal of phosphorus) with a WWTP in the region that is not by an anaerobic system, the difference is significant considering the standard deviation. On the other hand, the average potassium values are higher for animal waste, especially for residues from chickens and sheep.

Refs. [39,40] also observed that for the N and P macronutrients, WWTP values were higher than for animal waste, which in turn, had a higher quantity of K. Furthermore, the authors also found that aerobic systems with (or without) biological removal of phosphorus showed a higher quantity of P in sludge and/or biosolids.

Table 2. Macronutrient content (kg ton^{-1}—nutrient mass per ton of biosolids) in wastewater treatment plants (WWTP) biosolids in Brazil and South Africa and other organic residues (pig slurry and manure of cattle, chicken, and sheep).

Macronutrients	**Present Work Sequencing Batch Reactors (SBR) ***	**WWTP UASB in the Same Region of the Study (1)**	**19 WWTPs, Brazil (2)**	**7 WWTPs South Africa (3)**	**Pig Slurry (4)**	**Cattle Manure (4)**	**Chickens (5)**	**Sheep (5)**
N	33.64 ± 6.08	31.6 ± 6.72	42 ± 14	29.95 ± 7.54	3.92 ± 0.54	3.45 ± 1	15	7.5
P	20.55 ± 3	14.5 ± 1.31	13 ± 3.6	18.33 ± 5.89	1.15 ± 0.21	1.18 ± 0.2	15	3.8
K	1.16 ± 1.11	1.85 ± 0.95	1.5 ± 1.2	0.15 ± 0.02	3.25 ± 1.19	3.25 ± 2.5	8	11.9
Ca	3.33 ± 2.59	13 ± 6.32	31 ± 43	11.46 ± 1.22	0.68 ± 0.14	0.57 ± 0.2	24	5.9
Mg	0.64 ± 0.37	6 ± 2.66	2.6 ± 0.8	2.61 ± 1.44	1.93 ± 0.05	1.7 ± 0.6	NF	NF
S	11.13 ± 1.76	11.6 ± 2.66	17 ± 8.8	NF	0.2 ± 0	0.42 ± 0.15	NF	NF

Source: (1) [15], (2) [34], (3) [41], (4) [39], (5) [40]. * Four samples were collected (every 3 months) during 2016, 2017, and 2018 and two samples in 2019. NF: not found.

Biosolids in the WWTP of the present research obtained average N (33.64 ± 6.08 kgN ton^{-1}) and P (20.55 ± 3 kgP ton^{-1}) concentrations similar to the biosolids of tropical climate WWTPs from Brazil and Africa, considering the standard deviation. However, average values for nitrogen and phosphorus were higher when compared with anaerobic reactors in the same region of the study [15]. [34] found average N and P results in 19 WWTP in Brazil of 42 ± 14 kgN ton^{-1} and 13 ± 3.6 kgP ton^{-1}. [41] in 7 WWTP in Swaziland (South Africa) found average concentrations of 29.95 ± 7.54 kgN ton^{-1} and 18.33 ± 5.89 kgP ton^{-1}.

Regarding animal waste, the average concentration of N in the WWTP of the present research (IBRS WWTP) was 975% higher than the average concentration in cattle manure (lowest concentration of N between animal residues), with a mean concentration of 3.45 ± 1 kgN ton^{-1} and 224% higher than the average concentration of N in chicken manure, 15 kgN ton^{-1}, which contains the highest concentration of N between organic residues from animals.

When comparing the average concentration of P in IBRS WWTP between animal residues, we observed that it was higher than in the manure of chickens, with the highest concentration of P (15 kg P ton^{-1}) between animal residues. Yet it was 1787% higher than the quantity of P (1.15 ± 0.21 kg P ton^{-1}) in pig manure, which contains the lowest quantity between organic residues from animals.

Regarding potassium, the average concentration of K in the WWTP of the present research was similar to the concentration of other tropical climate WWTPs. A highlight was for chicken manure with a concentration of 8 kgK ton^{-1} and sheep manure with 11.9 kgK ton^{-1}. IBRS WWTP's Ca and Mg had low quantities, differing from the other WWTPs presented in the present study, which can be explained by the fact that the WWTP of the present study receives industrial and treated effluent, which reduces the concentration of those nutrients. Another possible explanation is that the sewage treatment systems differ in their techniques and procedures before they generate biosolids [41]. It is known that the biosolids composition varies significantly between the systems, as well as over time [42]. Among organic residues from animals, the quantity of Ca for manure of chickens, 24 kgCa ton^{-1}, significantly stands out. At last, another nutrient with a high concentration in the biosolids was S in the WWTP of the present research (11.13 ± 1.76 kgS ton^{-1}).

Despite the clear potential of this byproduct (biosolids), given the values here presented and compared with different types of effluent and processes generating biosolids, the population still gives a certain attention to organic waste [43]. Especially in regulatory and supervisory bodies, when the byproduct is of human origin, there is a rejection, particularly toward biosolids [7]. Ref. [44] observed that efforts can be made in three aspects to encourage the adoption of food fertilized with biosolids, namely, provision of nutritional information and microbiological quality of the product to resolve the population's doubts; effective price system that can make composting products more competitive; and more flexible policies to facilitate the applications.

3.1.2. Macronutrient in Biosolids and the Amount Required by Different Agricultural Crops

One can observe in Figure 2A, that the N content of IBRS WWTP (of the present research) (33.64 ± 6.08 kgN ton^{-1}) met 32% for corn demand, 33% for rice, 60% for wheat, forage grasses was >100%, for sugar cane 22%, for beans 31%, 21% for coffee, and 37% for cotton. Ref. [45] explained that generally most of N is found in its organic form in biosolids and, therefore, is readily available for plants after the degradation of organic matter.

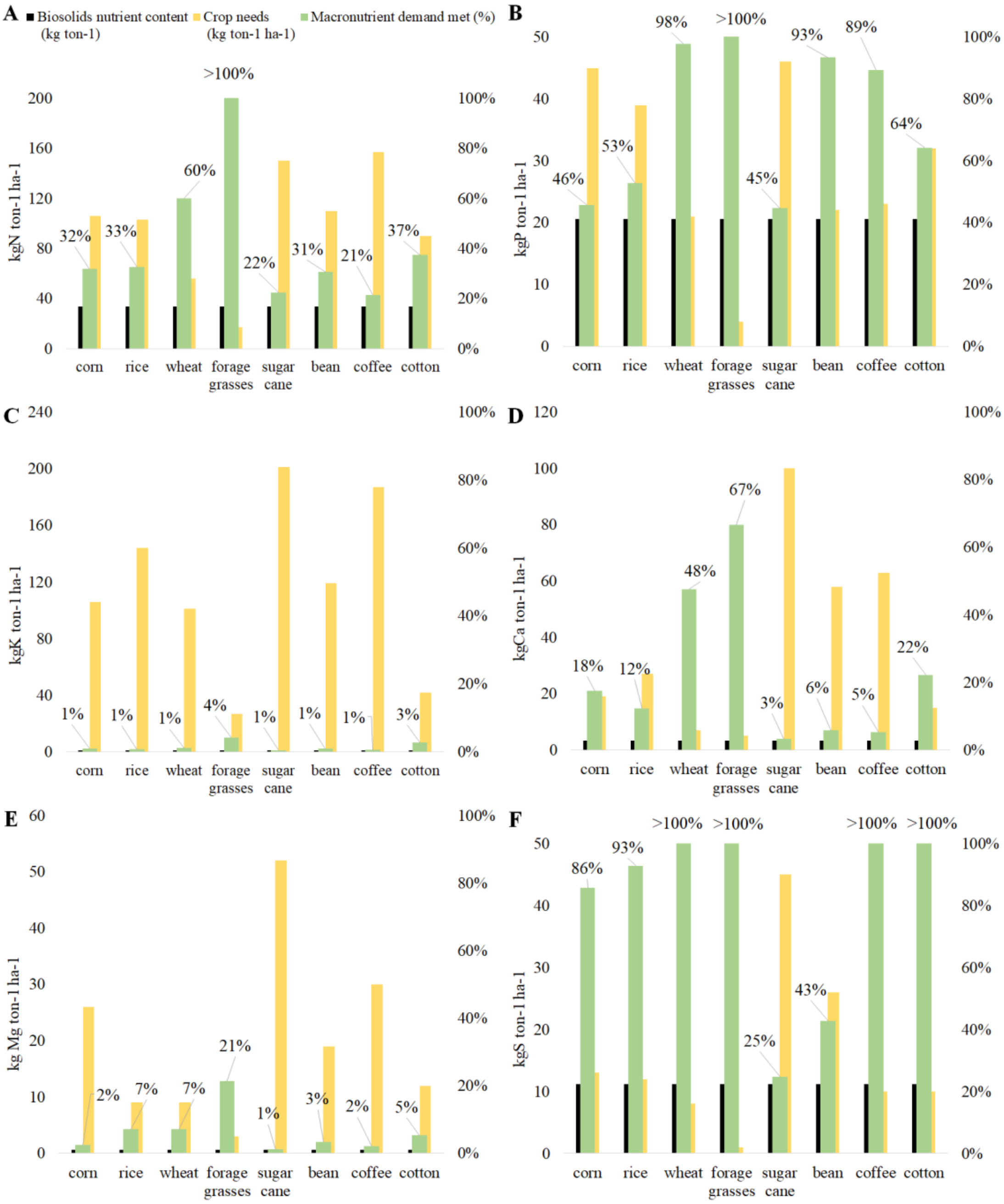

Figure 2. Percentage meeting macronutrients in corn, rice, wheat, forage grasses, sugar cane, beans, coffee, and cotton crops per 1 ha and 1 ton ha^{-1} application rate. N: nitrogen (**A**); P: phosphorus (**B**); K: Potassium (**C**); Ca: calcium (**D**); Mg: magnesium (**E**); and S: sulfur (**F**).

Concerning the P content of IBRS WWTP (20.55 ± 3 kgP ton^{-1}) (Figure 2B), the forage grasses P demand was met at >100%, corn 40%, rice 53%, wheat 98%, sugar cane 45%, beans 93%, coffee 89%, and cotton 64%. The relevance of the P found in the biosolids is in its bioavailability, which provides its use by the plants, and in its nutritional function [46], especially in tropical soils suffering from very low levels of P [47]. Instead of what occurs naturally in the soil, where P has low bioavailability and is strongly absorbed in the clay minerals in tropical soil, most of this element in the biosolids is readily available for plants in the first months of the production cycle [10]. Ref. [48] documented that the use of biosolids during ten years in tropical soils, provides adequate amounts of N and P for the corn crop (*Zea mays* L.), sunflower (*Helianthus annuus* L.), and crotalaria (*Crotalaria juncea* L.).

The concentrations of N and P were a positive result, meeting a relevant percentage of the needs of crops, especially for the forage grasses culture (>100%). This result corroborates the previous study with another WWTP from the same municipality (Campo Grande, Brazil), where the agronomic potential of biosolids coming from anaerobic digestion has been found for the soy, orange, eucalyptus, and tomato crops [15]. Ref. [15] showed in their study that biosolids were as efficient as conventional fertilizers in increasing the production of wood in the eucalyptus plantation. Ref. [49] documented that after 10 and 17 years of the first and only application of biosolids, organic matter content in the soils, total N and P, and cation exchange capacity were generally more elevated in soils treated with biosolids. That being so, after a single application of biosolids, it is possible to detect a beneficial residual effect on the soil, even after 17 years.

The recycling of P through biosolids has enormous potential and avoids this byproduct from being sent to landfills, which does not add value to the production of food, especially due to the imminent scarcity of this element. Moreover, this process can be optimized by way of the biosolids pyrolysis (biochar), which provides it with a considerable amount of P in its soluble form, which in turn increases the bioavailability for the plants' consumption [50]. Ref. [24] used acids for the precipitation of P, such as struvite, hydroxyapatites, and other calcium phosphates; however, the conjoint precipitation of metals was a negative aspect of this method and subsequent procedures are needed for its extraction, which has a high cost. Ref. [51], focusing on solving this negative effect, obtained positive results with the alkaline leaching of biosolids. Ref. [52] sought to extract P from incinerated biosolid and documented that they obtained a high-purity P extract, by the method developed by the researchers (two-step extraction), in which they used ethylenediaminetetraacetate, followed by sulfuric acid.

The K concentration in the WWTP of the present research (1.16 ± 1.11 kg K ton^{-1}) was low in the reference crops (assessed in this study), with the highest percentage at 4%, for forage grasses. Ref. [53], in a field investigation with long-term application of biosolids, documented that the K deficiency in the soil was evident during 17 years of application of biosolids. Moreover, the barley and corn crops obtained reduced growth, with symptoms of nutritional deficiency of K, such as white necrotic lesions and brown necrotic spots, similar to the K deficiency in all the cycles grown with biosolids. However, when there was the addition of inorganic K in the biosolids, there was growth improvement in both crops, high development, dry matter, and an increase in the K content in the soil. Therefore, researchers suggest that it is necessary to complement the K contents with mineral fertilizers, in that the biosolids require additional K fertilization already in the second or third growing season for maintaining an acceptable K concentration in the plants [54].

As regards Ca, the corn crop demand was met at 18%, rice 12%, wheat 48%, forage grasses 67%, sugar cane 3%, beans 6%, coffee 5%, and cotton 22%. As to Mg, the percentage meeting crop demand was 2% for corn, 7% rice, 7% wheat, 21% forage grasses, 1% sugar cane, 3% beans, 2% coffee, and 5% cotton. At last, S with an average concentration of 11.13 ± 1.76 kg S ton^{-1} obtained the best result among the macronutrients since this nutrient is available in large quantities in the biosolids and the needs of the crops are lower for S, relative to the other macronutrients. The nutrient met demands of forage grass, coffee, and cotton at >100%, and 86% in corn, 93% rice, 25% sugar cane, and 43% in beans. Fertilizing tropical soils with S is of extreme importance because there exist high growth demands in areas naturally deficient in this macronutrient that experience an increase in the demand for greater productivity [55].

Results in the present study corroborate others that show evidence that biosolids can replace or complement mineral fertilizers for N and P [15,47–49,56], as well as Ca, Mg, and S, with mineral K complementation [17,53]. Furthermore, the biosolids of IBRS WWTP in the present research and of other tropical climate WWTPs obtain a content of nutrients higher than the organic waste of animals found in the literature. Therefore, the doses to be applied to the soil must take into consideration the availability of nutrients and the characteristics of the soil intrinsic to each culture [27].

3.2. Pathogens

Table 3 presents the average pathogenic microorganism concentration found in biosolids, disposed of in the soil, throughout the three years of the IBRS WWTP monitoring. In comparison with the legislation of Brazil, the United States, and the European Union, the three norms establish two classes of quality, class A and class B. Biosolids in the WWTP of the present research were classified in three norms as class B. The biosolids had a concentration of *Escherichia coli* higher than the threshold concentration allowed for class A (<10^3 MPN g^{-1} TS), as well as enteric viruses above the class A limit (<0.25 PFU g^{-1} of TS). The average result of 381.5 ± 23,955 (MPN g^{-1} of TS) for *Escherichia Coli* and enteric viruses <1 (PFU g^{-1} of TS), according to [56], may be caused by the fact that the samples had been collected with biosolid freshly disposed of in the soil. However, several processes help the biosolids achieve adequate microbiological conditions, such as liming, thermal drying, or composting maintaining a thermal condition above 60 °C for at least three days [27].

Thus, there are no restrictions to the class B biosolids of the WWTP of the present study to being recycled in planted forests and recovery of soils and degraded areas, only to growing food products consumed raw, according to Conama Resolution 498/20 [27]. As regards the crops used as references to evaluate the agronomic potential of biosolids in this study, forage grasses cannot receive applications of biosolids two months before the grazing of animals. Furthermore, corn, rice, wheat, coffee, sugar cane, beans, and cotton can be fertilized with biosolids, as long as the interval between application and harvest is of 24 months [27]. In the old resolution, the 375/06 [26], class B biosolids only could be applied in coffee plantations, fiber, and oilseed crops, or in forestry, as long as they were incorporated into the soil mechanically and restrictions of harvest and public access were obeyed. According to EU legislation, the interval between application and harvest must be from 12 to 30 months for all edible crops [30]. In the USA, in turn, the interval between application and harvest must be of 14 months for food crops grown close to the ground, including vegetables, and 20–38 months for edible roots [28].

Class B biosolids of tropical climate WWTPs containing 30 helminth eggs (g TS^{-1}), 10^6 *Escherichia coli* (g TS^{-1}), and 10^5 *Salmonella* (g TS^{-1}) were tested in the cultivation of carrot and spinach. At the time of harvest of the spinach and carrots, respectively, seven and twelve weeks after planting, no *Salmonella* was detected in the soil, but about 10^3–10^5 *Escherichia coli* g TS^{-1} were found and around 1–6 helminth eggs (g TS^{-1}), according to the study of [57], in South Africa. Ref. [58] applied class B biosolids to grow lettuce and carrot, with up to 2 × 10^6 of *Escherichia coli* (g TS^{-1}), which resulted in lettuce and carrot contamination levels below the limit of Brazilian regulation about the sanitary quality of consumed raw vegetables [59]. Thus, it is evident that in Brazil, the Conama 498/20 legislation is aligned with international law, toward promoting a circular economy and the nexus concept (water, energy, and food), except with respect to crops that are consumed raw, which cannot receive class B biosolids.

Table 3. Concentration of pathogenic microorganisms in biosolids in the wastewater treatment plant of the present work in comparison with the legislation of Brazil, USA, and European Union.

Parameter	**Units**	**Results**	**Conama 498 (Brasil, 2020) (1)**			**Norm 503 (U.S EPA, 1994) (2)**			**Directive 86/278 EEC (European Union, 1986) (3)**		
			Class A	**Class B**	**Present Work**	**Class A**	**Class B**	**Present work**	**Class A**	**Class B**	**Present Work**
Viable helminth eggs	egg g of TS^{-1}	<0.08 ± 0.6 g^{-1} of TS	<0.25 g^{-1} of TS	<10	A	<1 in 4 g^{-1} TS	NR	A	NS	NS	B
Salmonella	g of TS^{-1}	ND	ND	NS	A	<3 MP	NR	A	ND	NS	A
E. Coli **(MPN g^{-1} TS%) or fecal coliform (USA)**	MPN	381.5 ± 23955 MPN g^{-1} of TS	<10^3	<10^6	B	<10^3	<2 × 10^6	B	<10^3	<10^5	B
Enteric viruses	PFU	<1 PFU g^{-1} of TS	<0.25 PFU g^{-1} of TS	ND	B	<0.25	NR	B	NS	NS	B

MPN: most probable number; PFU: plaque-forming unit; TS: total solids; ND: not detectable; NS: not specified; NR: not required. (1) [27], (2) [28], (3) [30].

3.3. Inorganic Substances (Metals)

The IBRS WWTP biosolids content showed potential for meeting the need for macronutrients of crops used as a reference for agronomic potential in the present research. This was a positive aspect in the matter of agricultural recycling of biosolids in tropical soils. Yet, bearing in mind the application of biosolids in the soil, it is important to know what are the potentially toxic substances that can hit the soil, the food, and groundwaters, in view of the health and safety of the population. Therefore, identifying and quantifying these substances are necessary because they are considered heavy metals and are, too, micronutrients essential to the plants, in adequate amounts, beyond being scarce in tropical soils.

Thus, as far as the use of biosolids in Brazilian territory is concerned, it is necessary to meet the requirements proposed by the Conama Resolution 498/20 [27], including the concentration of inorganic contaminants (metals). On a global scale, it is important to verify compliance with foreign legislation. In this case, Norm 503 [28] and the 86/278 European Community's Directive [30] were chosen. It is possible to observe in Table 4, a comparison of the concentration of samples of biosolids relative to the limits established by the Brazilian (class 1) [27], American [28], and European [30] legislation.

Table 4. Concentration of metals (mg kg^{-1}) in the IBRS WWTP biosolids in comparison with legislation from Brazil, USA, and European Union.

Parameter	Results	(1) Conama Resolution 498 (Brazil) Class 1	(2) Norm 503 (USA)	(3) Directive 86/278 EEC (European Union)	Fulfillment
As	0.49 ± 1.77	41	75	NS	Fulfills
Ba	102.35 ± 101.46	1300	NS	NS	Fulfills
Cd	0.04 ± 0.14	39	85	40	Fulfills
Pb	9.14 ± 12.30	300	840	NS	Fulfills
Cu	92.45 ± 84.49	1500	4300	1750	Fulfills
Cr	33.44 ± 43.53	1000	NS	NS	Fulfills
Hg	0.46 ± 1.29	17	57	25	Fulfills
Mo	4.83 ± 9.86	50	75	NS	Fulfills
Ni	17.11 ± 21.28	420	420	400	Fulfills
Se	<1 ± 0	36	100	NS	Fulfills
Zn	349.23 ± 319.92	2800	7500	4000	Fulfills

Fulfills: meets all requirements of the analyzed legislation; BR-US-EU: (1) [27], (2) [28], (3) [30]; NS: The maximum limit is not specified by legislation.

The average concentration of metals in the WWTP of the present research is within limits established by Brazilian, American, and European norms. Ref. [9] reviewed cases of application of biosolids in agricultural soils that followed the criteria and procedures of local legislation and identified that when the concentrations followed the quality standards of the legislation, they would not cause negative effects on the population and the environment.

In Brazil, Ref. [34] documented that only three from 19 batches of biosolids generated in 19 WWTPs were considered inadequate for direct application in agricultural soils due to the high Zn concentration, of >2.800 (mg Zn kg^{-1}), and one batch for excess Ni (>420 mg Ni kg^{-1}), as per legislation [27]. However, it is known that tropical soils are deficient in these elements which are essential micronutrients for diverse agricultural crops [49]. Ref. [34] reported that biosolids that were considered inadequate could be recurrent in sewage networks with clandestine discharge from industries, which the authors report being a common practice in the state of São Paulo (Brazil), where they did their investigation, and different from the Brazilian reality in general.

Zn is an element common in several industrial products and processes and in the human diet, therefore, among the metals, it is one of the most abundant elements in sewage [60]. Some studies reported that the application of biosolids with high Zn concentrations may occur, as long as with a low application frequency, in that the soils obtained the same benefits without a strong significant change in the quantity of Zn [61]. Therefore, one should respect the legislation's threshold concentrations, as Zn, along with other metals, may be toxic to the soil–plant system and in inadequate concentrations, inhibits the development of seeds, fruits, and roots [61].

Various studies consider the bioaccumulation by metals insignificant when biosolids are applied in agricultural soil again and again [62,63]. Ref. [63] documented that 16 years in a row of applications of biosolids in agricultural soils, in Spain, did not cause toxic effects on the soil–plant system. Ref. [62] documented that three years of application of biosolids did not significantly alter the concentrations of metals in the soil. Conversely, Ref. [64], in a study where biosolids were applied in 31 areas for more than 100 years without audit or control of the application rates, documented that the total concentrations of metals in the soil were a common source of contamination in all the areas. The authors reported that Zn was the most abundant element (122–2050 mg kg^{-1}), Cu varied from 25.3 to 766 mg kg^{-1}, Cd from 0.43 to 48.6 mg kg^{-1}, Cr from 43.2 to 1670 mg kg^{-1}, and Pb from 68.6 to 688 mg kg^{-1}. However, the results of the previously cited study may and should be contested since no control of the application rate is conducted over time.

Ref. [65] conducted an application of biosolids for ten years in Chinese soils and reported that the biosolids in their study could be applied safely to the soil at an application rate of 7.5 (tons ha^{-1}) for 18 years without exceeding the limits for Hg. As for Zn, the biosolids could be applied for 51 years, and more than 51 years for other heavy metals. In addition, the content of heavy metals in wheat and corn grains met the Chinese legislation's hygienic standard, and the Zn content in wheat straws and corn linearly increased with the increase in the biosolids application rates. Ref. [66] aimed to study three years of application of biosolid treated with and without lime for forage fertilization aimed at animal consumption and concluded that in relation to the Cu and Zn metals the quality of forage plants was adequate for consumption.

In a similar context, it is known that biosolids have a concentration of metals lower than that in diverse organic residues, for example, manure of cattle, chickens, sheep, and pigs [15]. Ref. [67] investigated the concentration of metals in bovine manure, broiler chickens, laying hens, sheep, and swine for slaughter, and found out that the As concentration in those fertilizers was higher than the average concentration found in our study of 0.49 ± 1.77 (mg As kg^{-1}), as per Table 2. The 0.04 ± 0.14 (mg Cd kg^{-1}) Cd concentration met the limits of the three norms, as well as was lower than that found in swine manure and other organic manures, such as manure of beef and dairy cattle and laying hens, of the study of [67].

Regarding Cu, with an average concentration of 92.45 ± 84.49 (mg Cu kg $^{-1}$), in addition to meeting the legislation limits, it was lower than the one found by [67,68] in pig manure. Ref. [69] documented that applications of chicken and swine manure over three years significantly reduced the accumulation of Pb and Cd in rice grains, and decreased the Cd and Pb available in the soil. The values found in the authors' organic residues were 6.90 (mg Pb kg^{-1}) and 0.41 (mg Cd kg^{-1}) for chicken manure, and 9.20 (mg Pb kg^{-1}) and 0.54 (mg Cd kg^{-1}) for pig manure, which are higher than the average concentrations found in the biosolids of the present study (Table 2).

3.4. Emerging Organic Pollutants (EOPs)

Concerning emerging micropollutants, the Conama Resolution 375 [26] presented reference values (limits) but was revoked on 19 August 2020. The resolution determined that 34 organic substances must be monitored in biosolids, and did not establish maximum concentration limits in waste, but in the soil. The new resolution, Conama 498 [27], which is in vigor, does not determine threshold concentrations nor provide for EOPs. Therefore, the comparison used in the present study was with the previous resolution.

However, the environmental agency may request the characterization or monitoring of potentially toxic organic chemical substances and establish the frequency of monitoring. The American legislation (Norm 503), reference for Brazilian legislation, does not establish limits for micropollutants, which may have influenced the new Brazilian legislation not to determine limits for these compounds. The European norm of 2007 establishes them [31] for EOPs concentration in the soil, the same way as the Conama 375 of 2006, old Brazilian legislation that included such limit as a precaution but made the use of biosolids not viable.

Table 5 shows that there was no detection above the limit for EOPs in the IBRS WWTP biosolids, except naphthalene. Naphthalene, of the group of aromatic polycyclic hydrocarbons, was the only one that exceeded the analyses detection limit (of 2.5 ± 2.25 μg kg^{-1}), a single time, showing the value of 5.1 (μg kg^{-1}); however, this value was low when compared to the maximum allowed concentration in agricultural soils (0.12 mg kg^{-1}) [26,31].

Table 5. Concentration of EOPs in biosolids in comparison with Conama Resolution 375 and EU Directive 2007.

EOPs	**Result (μg kg^{-1})**	**MQL**	**Resolution 375 (mg kg^{-1})**	**EU Directive EUR 22,805 EN 2007 (mg kg^{-1})**	**Fulfillment**
1.2-Dichlorobenzene	NF	10 ± 0	0.73	0.035	Fulfills
1.3-Dichlorobenzene	NF	10 ± 0	0.39	0.04	Fulfills
1.4-Dichlorobenzene	NF	10 ± 0	0.39	0.004	Fulfills
1.2.3-Trichlorobenzene	NF	10 ± 0	0.10	0.5	Fulfills
1.2.4-Trichlorobenzene	NF	10 ± 0	0.011	0.5	Fulfills
1.3.5-Trichlorobenzene	NF	10 ± 0	0.5	0.5	Fulfills
1.2.3.4-Tetrachlorobenzene	NF	5.5 ± 4.5	0.16	0.1	Fulfills
1.2.4.5-Tetrachlorobenzene	NF	5.5 ± 4.5	0.01	0.1	Fulfills
1.2.3.5-Tetrachlorobenzene	NF	5.5 ± 4.5	0.0065	0.1	Fulfills
Di-n-butyl Phthalate	NF	5.5 ± 4.5	0.7	0.1	Fulfills
Di(2-Ethylhexil)Phthalate	NF	5.5 ± 4.5	1.0	0.5	Fulfills
Dimethyl Phthalate	NF	5.5 ± 4.5	0.25	0.5	Fulfills
Cresols	NF	11 ± 9	0.16	0.5	Fulfills
2.4-Dichlorophenol	NF	5.5 ± 4.5	0.031	NS	Fulfills
2.4.6-Trichlorophenol	NF	5.5 ± 4.5	2.4	0.5	Fulfills
Pentachlorophenol	NF	27.5 ± 22.5	0.16	0.05	Fulfills
Benzo (a) anthracene	NF	2.75 ± 2.25	0.25	0.05	Fulfills
Benzo (a) pyrene	NF	2.75 ± 2.25	0.052	0.005	Fulfills
Benzo (k) fluoranthene	NF	2.75 ± 2.25	0.38	0.50	Fulfills
Indene [1.2.3-cd] pyrene	NF	2.75 ± 2.25	0.031	0.50	Fulfills
Naphthalene	5.1	2.75 ± 2.25	0.12	1.0	Fulfills
Phenanthrene	NF	2.75 ± 2.25	3.3	0.1	Fulfills
Lindane	NF	1.65 ± 1.35	0.001	0.06	Fulfills
Aldrin	NF	1.65 ± 1.35	NS	0.01	Fulfills
Dieldrin	NF	1.65 ± 1.35	NS	0.06	Fulfills
Endrin	NF	1.65 ± 1.35	NS	0.5	Fulfills
Chlordane	NF	2.75 ± 2.25	NS	0.04	Fulfills
Heptachlor	NF	0.55 ± 0.45	NS	0.03	Fulfills
DDT	NF	0.55 ± 0.45	NS	10	Fulfills
Toxaphene	NF	5.5 ± 4.5	NS	0.01	Fulfills
Mirex	NF	0.55 ± 0.45	NS	NS	Fulfills
Hexachlorobenzene	NF	2.75 ± 2.25	NS	NS	Fulfills
PCBs	NF	0.55 ± 0.45	NS	0.1	Fulfills
Dioxins and furans	NF	$1 \times 10^{-6} \pm 0$	NS	0.5	Fulfills

NF: not found; NS: the maximum limit is not specified by legislation. MQL: minimum quantifiable limits.

It is known that naphthalene is one of the most abundant APHs in polluted environments [70], which generally derive from anthropogenic (fires, industrial activities, and in residences) and natural (volcanoes and biosynthesis by algae) processes. It is known that pure sugar and sugar products are important sources of APHs in the population's diet; Ref. [71] warned that the concentration of APHs in beverages (spirit) is no news because sugar cane burning generates high contamination of these pollutants. The authors found a high presence of naphthalene in beverages derived from burnt sugar cane.

Furthermore, other important sources of APHs in the population's diet are related to olive oil and smoked meat, as well as food processing, in that the APHs concentration in edible oils and smoked products is an emerging concern [72]. To contribute to the application of biosolids in agriculture, Ref. [73] studied a laccase-enzyme-based product for the improved removal of three APHs (naphthalene, phenanthrene, and pyrene) from biosolids with high concentrations of APHs. Results in the authors' study proved to be effective as an environmentally adequate treatment for removing APHs associated with biosolids with potential for application on a large agricultural scale.

Ref. [74] investigated the concentration of APHs for the application of three doses of anaerobic treatment biosolids in urban soils. The authors discovered that applications did not significantly influence the total concentration in 16 APHs. Among the APHs cited in that study, only seven were indicated by Conama Resolution 375/06 as far as the concentration in the soil is concerned [26]. Another group is the organochlorine insecticides (OCs), which are considered one of the most effective for agricultural benefit with immediate results in the control of weeds and other pests. Moreover, these insecticides are EOPs of the groups of POPs and APHs. Ref. [75] analyzed 16 OCs in biosolids from 19 WWTPs in Spain and reported that aldrin with a concentration of 21–142 (ng g^{-1}) and DDT 33–100 (ng g^{-1}) were the pollutants detected with the greatest frequency. However, these concentrations are low when compared with the concentrations allowed in soils by legislation [26,31].

In the study of [76], by detecting EOPs in biosolids in a WWTP in Rio de Janeiro (Brazil), the authors found close results. They detected only the presence of di(2-ethylhexyl) phthalate (DEHP) at the concentration of 23.833 (mg kg^{-1}). Although the results of the present research are encouraging, there is a concern due to the risks to human health and the environment, especially because of the lack of knowledge about the behavior of EOPs, which shows the need for additional research [77,78].

Nonetheless, Ref. [72], by analyzing 127 papers that dealt with EOPs between 1997 and 2017, concluded that the methods for detecting EOPs, which include GC-MS as the most frequent, constitute a critical point and also a barrier for determining with safety of the concentrations. Information is corroborated in the study of [79], who documented the detection of these pollutants in diverse environmental matrices, and the greatest challenge for more accurate and complex studies is the methods of detection, even with the significant improvements in equipment, methods, reagents, etc.

3.5. Principal Component Analysis (PCA)

In Figure 3, one can observe the variables of greatest influence and relationships between them by way of the graph of interaction between the 17 chemical elements, organic matter (total organic carbon—TOC), and pH, categorized as per the different seasons of the year—summer (gray), spring (yellow), autumn (blue), and winter (red).

To verify if there was a significant difference in the chemical elements' concentration between different tropical climatic conditions, we submitted the data with categories of summer and spring (hot and rainy) and winter and autumn (cold and dry), similar to the study of [80]. Therefore, we observed that chemical elements, as well as organic matter content and pH, were not sensitive to the tropical climate conditions and did not significantly vary in their concentration. Fourteen chemical elements and pH are observed to cluster; the exception is K, P, and S (Figure 3).

Furthermore, we observed (Figure 3) that the contribution of variables of weight higher than 7.5%, that is, with the greatest statistical contribution, were the Cu and Ba metals, as well as the Ca macronutrient. Ref. [34], by analyzing diverse samples of biosolids from WWTPs in the state of São Paulo (Brazil), also found the Cu, Ba, and Ca elements as high-contribution variables through the PCA method. With a contribution from 3% to 4%, we have the As metal and the N macronutrient, which met with a low percentage of the need for the related crops. The K, P, and S parameters also had a low contribution, lower than 2.5%. With median contribution values between 5% and 7.5% the Se, Mg Zn, Ni, Cr, Pb, Hg, Cd, and Mo elements, pH, and total organic carbon (organic matter); furthermore, elements, TOC, and pH clustered. The same substances were significant in the study of [81], along with Cd, Zn, Ca, Mg, Ni, and TOC, and substances and TOC also clustered with pH. According to [82], pH is a way of controlling the concentrations of metals in biosolids.

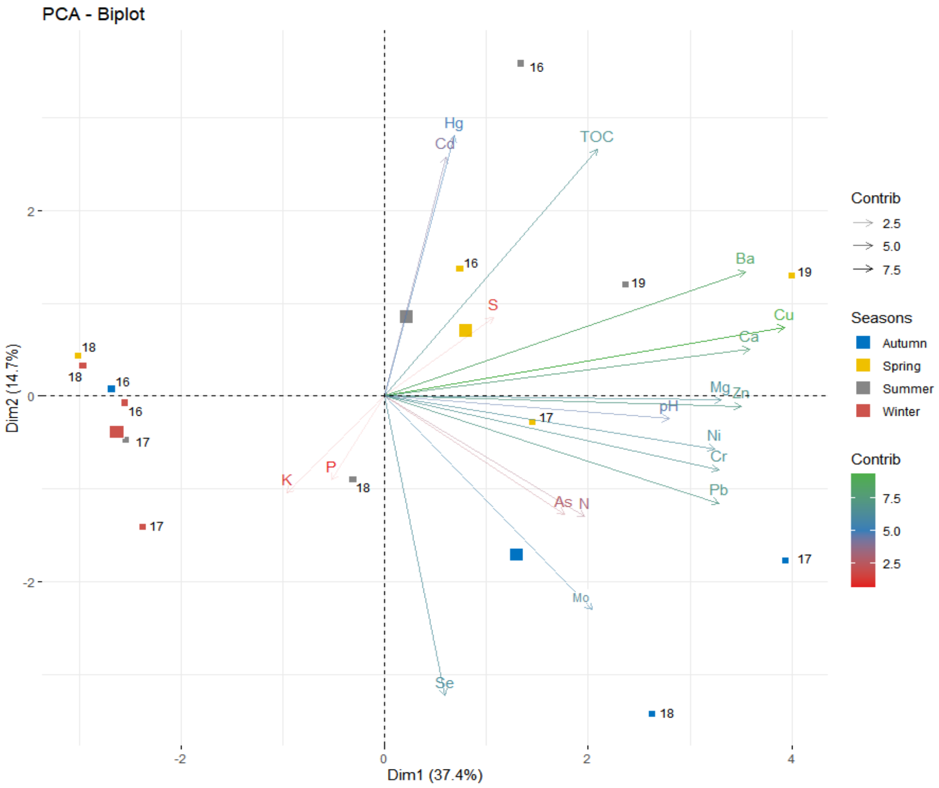

Figure 3. Results of the principal component analysis.

At last, we can document with this PCA analysis that the concentrations of chemical elements in biosolids are not sensitive to tropical climatic variations, as well as intense weathering. Yet the pH is the key element and should be used as a basic parameter for environmental control.

In summary, besides the sludge potential for energy and renewable-gas production [83] and other processes such as: incineration, gasification, anaerobic digestion, and nutrient recovery by using the struvite production pathway [84], our results show the possibility of using biosolids in a safe and simplified way. In-depth research on water–energy–food, focusing on biosolids, by business models, will improve rural-urban water and sanitation planning and decision making with a focus on the circular economy and support the interest in wastewater management and pave the way for future feasibility studies [84,85].

4. Conclusions

Results in the present study show that the contents of biosolids are potential sources of macronutrients for corn, rice, wheat, forage grasses, sugar cane, beans, coffee, and cotton crops, particularly N, P, and S.

Pathogens fell into class B for the Conama 498 resolution (Brazil), Norm 503 (USA), and Directive 86/278 (EU), relative to the concentrations of *Escherichia coli* and enteric viruses. This class lacks studies for more simplified and sustainable business models, to promote a circular economy in developing countries, which demand low-investment solutions but with safety for the local population.

Metals, also compared with the three previous norms, fulfilled the threshold concentrations of the respective norms.

Emerging organic pollutants remained below the analytical detection limit, except naphthalene, which a single time was found in the biosolids above the detection limit.

The PCA showed that the chemical elements did not have significant sensitivity throughout the climatic variation of the tropical climate but had the pH parameter as a basis for environmental control. This result is significant, as it demonstrates its resilience to climate change.

Our results confirm the safety agronomic potential of biosolids toward promoting the nexus concept (water, energy, and food) and circular economy in WWTPs. This economy, in turn, aligned with a cleaner agricultural production in tropical soils, reducing the quantity of biosolids sent to landfill or without adequate disposal.

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