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Evaluation of the performance of a compartment reactor for the disposal of heavy metals

Avaliação do desempenho de um reator compartimentado para eliminação de metais pesados

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ABSTRACT
The inadequate final disposal of liquid waste, especially effluents from industrial processes and domestic sewage, has caused serious environmental problems, especially in large cities. About 70,000 chemical compounds are available on the market and 500 to 1000 compounds are produced annually, which are somehow disposed of as domestic or industrial effluents, being responsible for the contamination of water bodies. In the face of the environmental catastrophe that is presented to ensure the preservation of the way of life of humanity, sewage treatment systems, especially the so-called alternatives, present themselves as a beneficial and important source for study and development in research centers outside and inside Brazil. Such emphasis on alternative systems is due to the low relative cost of implementation and greater ease of handling and maintenance. Among the alternative treatment systems under constant study are anaerobic reactors. Anaerobic reactors are an important sanitary tool for use because they require low hydraulic detention time and useful area for deployment. They are compartments that promote the treatment from the biochemical reactions present by the bacteria contained in the sewage. The present research included monitoring the removal of existing heavy metals in a pilot system implemented at UNICAMP during the month of January 2023. The system evaluated the input and output concentrations of the system and relied on the synchrotron radiation technique for detection of metals and other chemical elements. For the five-week study period, the initial results showed a percentage efficiency for the compounds of: phosphorus (50.05%); sulfur (already commented); chlorine (51.12%); potassium (51.15%) and zinc (52.63%). Thus, showing the great positive impact of the anaerobic reactor system.

Keyword: Environment, Reuse, Contamination, Sustainability.

1 INTRODUCTION

In recent years there have been witnesses, in the technological advances in sewage treatment, the feasibility of system projects with great efficiency for the treatment of wastewater;

However, these new technologies are mainly aimed at the treatment of large volumes of waste and for large cities.

In large urban centers, particularly in the Southeast and South regions of Brazil, the soil of the river basins presents a high demographic density, due to the presence of industries.

According to Silva and Nour (2005) the water of the rivers of these basins does not indicate characteristics of drinking water, that is, that do not require sophisticated treatment.

For Silva and Nour (2005) the soil is also occupied by agricultural properties that also generate products that pollute and contaminate the surface and underground water bodies (or springs).

In general, little attention is paid to the treatment of liquid effluents generated in rural properties, especially sewage of sanitary origin that, even without presenting a high concentration of polluting compounds, has a considerable amount released without proper treatment in the water bodies closest to this property.

The percentage of sewage that can receive any type of treatment is small, which is why surface water treatment systems for municipal and rural supplies are increasingly sophisticated and costly.

The degree of treatment required depends on the characteristics of the sewage and local possibilities (SILVA and NOUR, 2005).

According to Zanella (1999) few domestic sewage treatment projects propose to develop technologies aimed at the rural population, emphasizing that low-cost anaerobic treatment systems constitute an economically viable alternative, such as septic tanks, stabilization ponds, anaerobic reactors, reuse and disposal in the soil, cultivated beds and others.

One option to solve the problem that zootechnical and agricultural activities cause to water resources is the anaerobic compartmentalized reactor.

According to Zanella (1999) and Silva (2001) the compartmentalized reactor showed promise in the treatment of wastewater, for the efficiency in the removal of organic and solid matter in suspension, for the low cost of implementation and operation and for the simplicity of operation.

Thus, the objective of this research was to evaluate a thematic project of an anaerobic reactor designed and operational in the facilities of the Faculty of Agricultural Engineering (FEAGRI) of the State University of Campinas (UNICAMP).

For this research we focused on the performance of the removal of heavy metals present in the effluent of FEAGRI.

The sewage was collected and later analyzed by the synchrotron radiation technique at the Brazilian Synchrotron Light Laboratory (LNLS) in Campinas.

2 LITERATURE REVIEW

2.1 GENERAL CHARACTERISTICS OF SEWAGE SLUDGE AND ITS RELATIONSHIP WITH THE PRESENCE OF METALS

According to Aisse *et al.* (1999), sludge, a mixture of organic and inorganic matter, is a generic name for the solids that accumulate in the sewage treatment system, which must, periodically or continuously, according to the treatment system, be discarded.

In the biological processes of treatment, part of the organic matter is adsorbed and converted, being part of the microbial biomass, generically called biological or secondary sludge, composed mainly of biological solids, and therefore also called biosolid. For this term to be adopted, it is still necessary that its chemical and biological characteristics are compatible with productive employment (SPERLING and ANDREOLI, 2001).

Regarding the physical characteristics of the sludge, Ferreira and Andreoli (1999) mentioned that the residue is usually in liquid or semi-solid liquid form, usually containing between 0.25 and 12% of solids.

For Sperling and Gonçalves (2001), moisture influences the mechanical properties of the sludge, which influence the type of handling and final disposal.

A sludge with a dry solids content of 2.0% has a humidity of 98%, which means that out of every 100 kg of sludge, 98 kg is water and 2 kg is solid. The pH of the sludge is close to neutrality (between 6.0 and 7.0); the organic carbon content is approximately 30%; and the nitrogen, phosphorus and sulfur contents are relatively high. The levels of sodium (Na) can be high and the sludge can have high concentrations of micronutrients and heavy metals (MEURER, 2006).

The concentration of heavy metals in sludge is one of the fundamental controls for its safe use (MELO JÚNIOR, 2017).

Some are micronutrients needed by plants, such as copper (Cu) and zinc (Zn). Others, in addition to not being necessary, can accumulate in the soil at levels toxic to plants and man (MEURER, 2006).

The risk associated with heavy metals from sludge is mainly linked to the fact that the soil is able to store these metals. Although heavy metals are cumulative in the soil, several soil factors interfere in the dynamics of their availability, such as pH, cation exchange capacity (CTC), texture and organic matter content. Thus, depending on environmental conditions, metals may be present in the soil in forms not available to plants (MEURER, 2006).

Heavy metals not only exert negative effects on plant growth, but also affect the biochemical processes that occur in the soil (MEURER, 2006).

The decomposition of organic material added to the soil, nitrogen mineralization and nitrification can be inhibited in sites contaminated by heavy metals (TSUTIYA, 2001).

In soil, adsorption, complexation, oxidation/reduction and precipitation reactions control the availability and solubility of metals. Thus, the study of the agricultural use of municipal waste containing high levels of heavy metals is of great importance, as it seeks to reduce polluting waste without, however, polluting the ecosystem (DIONÍSIO *et al.*, 1999).

2.2 NUTRIENTS AND HEAVY METALS IN SOILS AS A FUNCTION OF SEWAGE SLUDGE DOSES

Aiming to evaluate cadmium leaching in a Dystroferric Red Latosol (high concentration of iron oxides) and a Nitosol (high concentration of clay), Prado and Juliatti (2003) installed an experiment in PVC (Polyvinyl Chloride) columns with application of biosolid contaminated with cadmium. The authors concluded that cadmium (Cd) was immobile in the two soils studied.

The movement of iron (Fe), copper (Cu), zinc (Zn) and cadmium (Cd) in a soil fertilized with sewage sludge was studied by Messias *et al*. (2007). These authors assembled columns filled with soil samples mixed with doses corresponding to 0, 25, 50 and 75 t $^{ha-1}$ of sewage sludge and observed an increase in the contents of Cu, Zn, Fe and Cd.

Regarding macronutrients, Trannin *et al.* (2008), in Dystrophic Cambisol (high concentration of aluminum and low fertility), observed higher levels of organic carbon in soils treated with sewage sludge.

2.3 ANAEROBIC TREATMENT SYSTEM BY ANAEROBIC REACTOR

The great success in the development of technologies for anaerobic (or anaerobic) treatment can be attributed to the introduction of high-rate reactors, such as the UASB (*Upflow Anaerobic Sludge Blanket*) and anaerobic filter reactors in the 1970s and 1960s respectively (SILVA and NOUR, 2005).

The various favorable characteristics of anaerobic systems, which can be operated with high solids retention times and very low hydraulic detention times (θ_h), confer great potential for

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their applicability in the treatment of low concentration wastewater according to Chernicharo, (1997) and high concentration, such as pig farming according to Oliveira (2003).

The configuration of the UASB reactor basically consists of the hydraulic regime of upward flow and the incorporation of an internal solid/gas/liquid separation device, dispensing with the use of a support medium for biomass growth. This favors the development and retention of a concentrated and highly active biomass in the digestion zone, in the form of dense flakes or granulated sludge. Consequently, the reactor operates with very high solids retention times (SRT), even when subjected to a very low Hydraulic Detention Time (θ_h) (FORESTI and OLIVEIRA, 1995).

Treating sewage using anaerobic reactors is a positive option, especially for regions with a hot climate as in the case of the Brazilian Northeast (VANHAANDEL and LETTINGA, 1994).

According to Chernicharo (2007) some of the advantages of anaerobic treatment are: the low production of total suspended solids (STS); low energy consumption, usually associated with an arrival lift.

Foresti and Oliveira (1995) comment that the anaerobic reactor system has low operating costs; low area demand; low implementation costs; in addition to the production of methane (CH_4), a fuel gas of high calorific content; possibility of preserving the biomass, without feeding the reactor, for several months; tolerance to high organic loads; applicability on a small and large scale and low nutrient consumption.

2.4 SYNCHROTRON RADIATION

The synchrotron radiation technology consists of the use of excitation radiation beams for multielemental analysis of different areas of scientific knowledge (MELO JÚNIOR, 2007).

The Synchrotron Radiation is the electromagnetic radiation emitted by a charge moving at a speed of 90% of the speed of Light, 270,000 $km.s^{-1}$, along a curved trajectory. This relativistic condition applies, in particular, to particles circulating in Accelerators from Electrons or Positrons, whose corresponding radius of curvature has in the initial order of several meters to tens of meters. The name of this radiation derives from a specific type of accelerator, the synchrotron of electrons (LNLS, 2023).

Synchrotron radiation can also have a natural origin, being produced by astronomical objects, such as remnants of Supernovae (Pulsars), Quasars and nuclei of active galaxies (MELO JUNIOR, 2007).

Radiation is produced when relativistic electrons spiral along the magnetic fields produced by these objects (ZEILIK, 1987).

Synchrotron radiation is the source of use for techniques to study behaviors from diffraction, electron microscopy, to total reflection, known as the technique of X-ray fluorescence by total reflection. Where the beam is calibrated for different energy levels (LNLS, 2023).

2.5 TOTAL REFLECTION OR X-RAY FLUORESCENCE BY TOTAL REFLECTION

The technique of X-ray fluorescence by total reflection due to the small thickness of the samples and the high energy of the X-rays normally used in excitation, there is no occurrence of the absorption and reinforcement effect and, consequently, the correction for the matrix effect is not necessary (ZEILIK, 1987).

In this case, equation 1 represents the relationship between the fluorescent intensity of the characteristic line and the concentration of the element of interest.

$$I_i = S_i . C_i \qquad \textbf{Equation 1}$$

Where:

Ii – represents the net intensity of the X-rays (cps = counts per second) of the characteristic line K or L of the element i of interest;

Ci – its concentration (ppm or $\mu^{g.mL^{-1}}$) in the pipetto solution in the holder, and

Si – the relative sensitivity of the system (µcps/g or cps/ppm), for the element of interest "i".

According to Melo Júnior (2007) the elements to be used as internal standards should not be present in the samples, and so the elements Ge (Germanium) and Ga (Gallium) have been the most used for water samples.

3 MATERIALS AND METHODS

The research aimed to study a compartmentalized anaerobic reactor system (RAC) in which its construction took place between the months of August to December 2022. Being implanted in the dependencies of the experimental field of the Faculty of Agricultural Engineering (FEAGRI) of the State University of Campinas (UNICAMP).

The system was implemented to study several sanitary parameters, and the focus of this research was the monitoring and behavior of the removal of heavy metals present in the sewage.

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And its subsequent analysis by the synchrotron radiation technique developed at the Brazilian Synchrotron Light Laboratory (LNLS).

3.1 COMPARTMENTALIZED ANAEROBIC REACTOR (RAC)

The compartmentalized anaerobic reactor (RAC) was designed during the months of August to December 2022 with the indications of researchers Barros and Campos (1992), Povinelli (1994) and Nour (1996) for a flow of 4.6 m 3.d-1, with a hydraulic detention time (ϴ h) of 12 h.

The total volume of the reactors was 2.3 m3, the first with 1.4 m3 and the second with 0.9 m3.

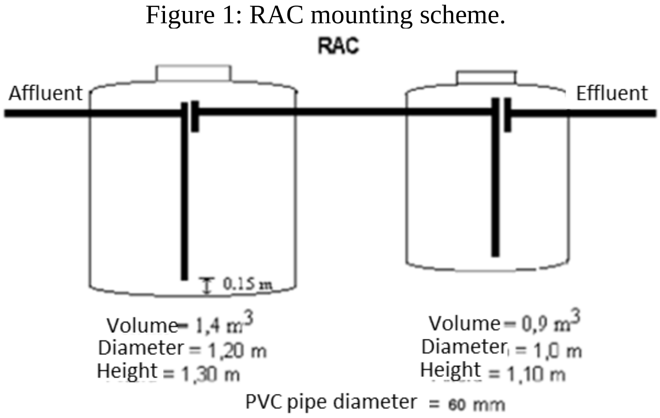
The tributary of each reactor was introduced by a 60 mm pipe extended to the center of the compartments and redirected by a "tee" pipe to the bottom of the reactor by a second pipe up to 15 cm from the bottom.

The introduction of the tributary near the bottom of the compartments allowed an increase in the contact between the substrate and the blanket of sludge formed in the lower layer.

In the execution project was adopted for construction, the ferrocement technique. The ferrocement technique is a construction technique in which a layer of cement is applied on an iron structure, this one made of rebar surrounded by a metal screen. It's not as tough as reinforced concrete, but it's a low-cost alternative in applications that don't require a lot of strength.

Figure 1 shows a schematic of the RAC (Compartmentalized Anaerobic Reactor) system installed and its dimensions.

Figure 1: RAC mounting scheme.



The circular shape was used for the facilities it offers in the construction of ferrocement reinforcements and for the better distribution of external tensions, which are concentrated in the straight corners, in the case of cubic structures.

Figure 2 shows the two tanks during the application of the mortar of the external finish. It is possible to observe the interior bottom of the first tank only with the external coating, highlighting the perfect visualization of the elements of the structure - the iron bars, the wire mesh and the plastic mesh - still exposed (Figure 3).

Figure 2: View of RAC under construction Figure 3: Internal view of RAC

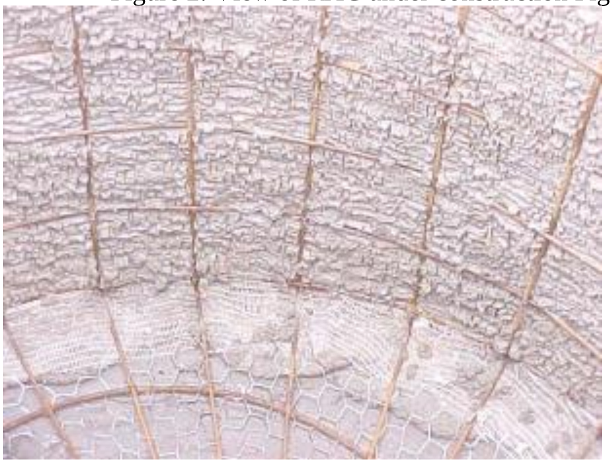


Figure 4 shows in detail the effluent inlet device, with the 50 mm PVC pipe directing the effluent to the center and bottom of the reactor.

While Figure 5 presents an overview of the RAC after its completion, with the hydraulic connections of inlet and outlet of the effluent and the pipe (white PVC of 100 mm) of inlet for a sludge suction hose, when necessary for cleaning and disposal.

Figure 4: System Detail on Internal RAC Input Figure 5: Overview of the Finished RAC

3.2 SAMPLE COLLECTION

The research to monitor the concentrations of heavy metals and other chemical elements was implemented during the month of January in the period from January 3 to 31, 2023 (five weeks of study) with weekly samples of entry and exit of the RAC system. Thus, totaling 10 samples, five of which are inlet (affluent) and five outflow (effluent), are preserved in PET bottles at a temperature of -5oC.

The volume removed was 500 ml per bottle for analysis by the synchrotron radiation technique (Figure 6).

Figure 6: PET bottle used for collections.

3.3 METHODOLOGY FOR USING THE SYNCHROTRON RADIATION TECHNIQUE WITH TOTAL REFLECTION

In the analysis by total reflection, an aliquot of 1 ml of wastewater should be removed from the study sites and added with the use of an automatic pipette a Gallium standard of 100 μl (102.5 mg. L-1) used as the internal standard, resulting in a concentration of 9.32 mg. $^{L-1}$ of the standard in each sample.

The internal standard was used to eliminate the non-uniformity of the aliquot in the support, because the thin film formed on the substrate does not have regular geometry. Thus, the intensity of the X-rays obtained in the irradiation of the sample depends on the position in which it was placed on the support. With the addition of the internal standard, the result obtained will always be in relation to this standard, not mattering, in this way, the position of the sample (MELO JÚNIOR, 2007).

At the end of the preparation, 5.0 μL of the resulting solution was then pipetted under the lucite plate and the sample was dried with the aid of an infrared lamp, as shown in Figure 7.

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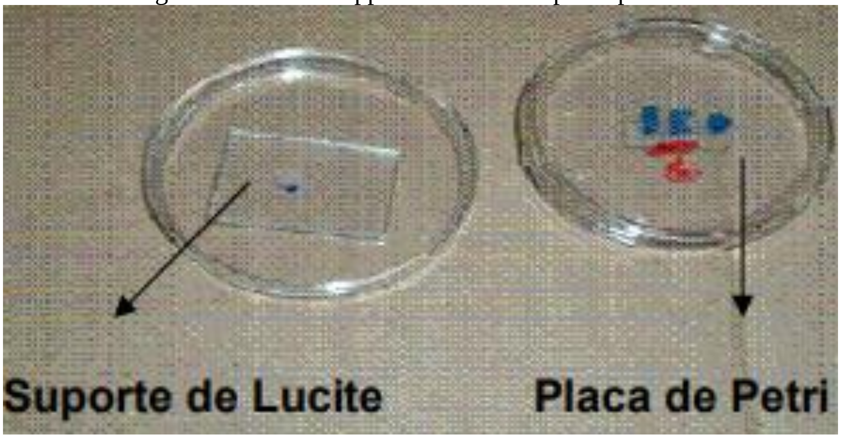
Figure 7: Lucicite support used for sample deposition.



3.4 INSTALLATIONS OF THE SYNCHROTRON RADIATION LINE – TOTAL REFLECTION TECHNIQUE

The synchrotron radiation line for total reflection is intended for the analysis of multielemental chemical composition ($Z \geq 13$) in scientific applications of trace element determination in environmental, biological and materials sciences, chemical depth profile of thin films and chemical mapping (LNLS, 2019).

The line of the Brazilian Synchrotron Light Laboratory (LNLS) operates with a beam of 4 to 23 KeV, where Table 1 shows other characteristics of the line (MELO JÚNIOR, 2007).

Table 1 - General Characteristics (Source: Brazilian Synchrotron Light Laboratory)

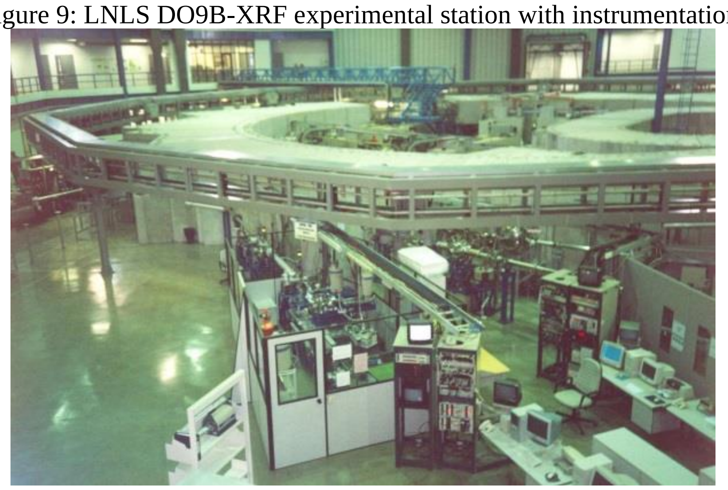
Source	Deflector magnet D09B(15º), σy = 0.222 mm, sample flow: 4 x 109 photons/s at 8 keV
Monochromator	Monochromator *channel-cut.*
Crystals	Si (111) (2d=6,217 Aº): 4-14 keV (E/ΔE=2800); Si (220) (2d=3.84 Aº): 5-23 keV (E/ΔE=15000).
Detectors	Hyperpure Ge (150 eV resolution) and Si(Li) (165 eV resolution) solid-state detectors; photodiodes and ionization cameras.
Optics	Capillary optics with 20 μm spatial resolution.
Sample handling	Vacuum chamber (2-10 mbar) with conventional excitation geometry (45º - 45º). Stations for experiments the grazing incidence and 2D mapping, both with total control of sample positioning.

Figure 8 shows the pipe of the DO9B – XRF line, used in the experiment, through which the synchrotron light beam passes from the ring to the fluorescence experimental station.

Figure 8: Photo of the ring pipe for the DO9B – XRF line of LNLS.

In the experimental station of X-ray fluorescence, for the detection of X-rays, a hyperpure semiconductor Ge detector was used, with a beryllium window of 8 μm thick, an active area of 30 mm2, coupled to an amplifier module and with a multichannel analyzer plate, inserted in a microcomputer (Figure 9).

Figure 9: LNLS DO9B-XRF experimental station with instrumentation.



The experimental arrangement *(setup*) allows the rotation and translation of the sample in order to obtain the condition for the total reflection of the incident beam on the sample that is allocated in the rectangular plate of lucite (Perspex) fixed in the sample holder, allowing the measurement of the chemical elements contained in the sample.

Figure 10 shows in detail one of the RAC study wastewater samples.

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Figure 10: Experimental arrangement of SR-TXRF.

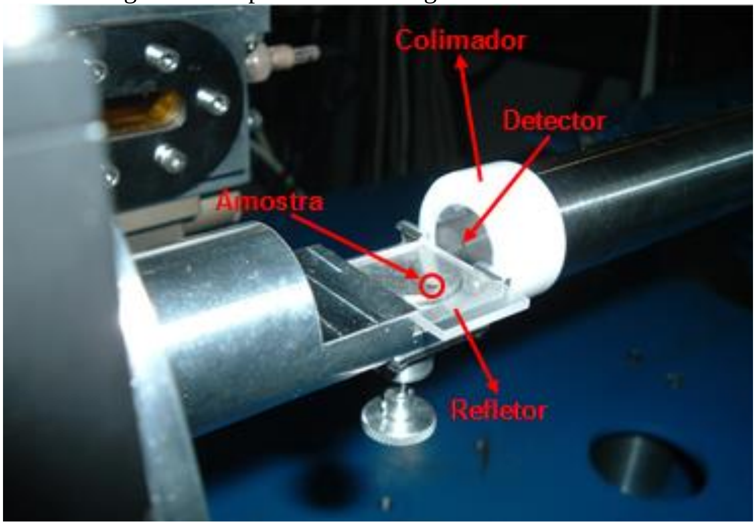

Figure 10 highlights the location of the sample after drying with UV lamp deposited on the reflector and the detector with the collimator of the beam for synchrotron radiation.

4 RESULTS AND DISCUSSIONS

4.1 VALIDATION OF ANALYSES

To ensure that the detected values are reliable, a characteristic curve with defined patterns was constructed. Thus, a degree of reliability was obtained to later evaluate the samples collected from the RAC.

Elemental sensitivity was calculated using five standard solutions with known elements and at different concentrations, plus the element gallium (Ga) used as the internal standard. For this, the range for this pattern curve is called the K series.

The experimental sensitivity for the elements contained in the solutions was determined and the results obtained are presented in Table 2.

Table 2 - Experimental sensitivity for the K series

Atomic number	Chemical element	Sensitivity
19	K	0,083270
20	Ca	0,086387
22	Ti	0,199891
24	Cr	0,393820
26	Fe	0,593576
28	Ni	0,796228
30	Zn	0,897568
31	Ga	0,975819
34	Se	0,809614
38	Sr	0,306673

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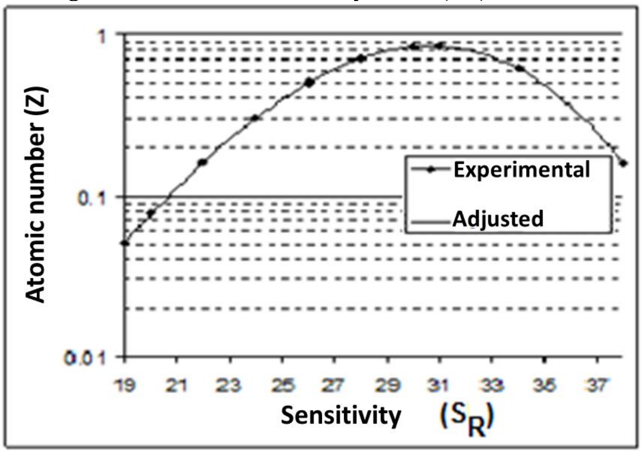
Using the data obtained from Table 2, the reliability degree curve was raised from a characteristic graphic curve, called the relative sensitivity curve for K series.

Figure 11 shows the characteristic curve of reliability for the elements in the range of interest of the study.

Figure 11: Relative sensitivity curve (S_{Ri}) for K series



The sensitivity curve shown in Figure 11 initially allowed the use of certified samples with known values.

This made it possible that if when using the same energy beam used in the sensitivity curve for the certified samples, the values were close to the pre-defined ones, then the samples collected and analyzed, their values were really as expected.

The adjustment was made with a standard certified by the *National Institute of Standards and Technology* (NIST).

Table 3 shows the samples certified by NIST for the standard called by NIST as Drinking Water Pollutants, *with the values certified by NIST and those measured in the LNLS*.

Table 3 - Comparison of values measured and certified by NIST.

Element	Measured Value and Confidence Interval (mg. L-1)	Certified Value and Confidence Interval (mg. L-1)
Cr	**8.91 ± 0.18**	**8.89 ± 0.45**
As	**8.66 ± 0.17**	**8.77 ± 0.45**
Se	**4.87 ± 0.07**	**4.69 ± 0.23**
Cd	**4.55 ± 0.64**	**4.54 ± 0.23**
Ba	**89.99 ± 0.76**	**91.89 ± 4.55**
Pb	**10.03 ± 1.01**	**10.09 ± 0.45**

Only after this validation of the system was then that the dried samples were used in the collected lucicite plates and their elemental chemical values were determined, according to Table

4, for the five weeks of the study, in relation to the input concentrations (tributary) of the chemical elements.

Table 4 - Concentration (mg. L-1) of the incoming chemical elements (tributary).

	Chemical element							
Weeks	P	S	Cl	Towards	Cr	Minutes	Faith	Zn
1	9,99	49,79	97,8	391	0,010	0,62	10,6	0,38
2	8,36	38,91	93,5	389	0,011	0,59	10,9	0,37
3	8,80	42,20	89,6	378	0,010	0,53	10,3	0,30
4	9,51	39,90	91,5	369	0,009	0,51	10,6	0,32
5	9,16	41,57	89,4	383	0,010	0,49	10,9	0,31
Average	9,16	42,47	92,4	382	0,010	0,55	10,7	0,34

Analyzing the results it can be noted by Table 4 that the elemental concentration for the samples maintained the same level, and there were no large fluctuations in chemical concentrations (Table 4), however it should be noted that the concentrations were high before the pre-treatment when analyzing the requirements of CONAMA (National Environmental Council).

After the pre-treatment by the compartmentalized anaerobic reactor system (RAC), there was a considerable reduction in the levels of elemental contamination, which are presented in Table 5, below.

Table 5 - Concentration (mg. L-1) of the output chemical elements (effluent)

	Chemical element							
Weeks	P	S	Cl	Towards	Cr	Minutes	Faith	Zn
1	4,99	19,79	47,8	191	0,009	0,42	6,6	0,18
2	3,36	18,91	43,5	189	0,008	0,39	6,9	0,17
3	3,8	22,2	39,6	178	0,009	0,33	7,3	0,1
4	4,51	19,9	41,5	169	0,009	0,41	6,6	0,12
5	4,16	21,57	39,4	183	0,008	0,39	6,9	0,11
Average	4,16	20,47	42,4	182	0,009	0,39	6,9	0,14

Analyzing the results for tributary and effluent in relation to the residual samples during the five weeks of study, we have that by the average values of the chemical elements detected: phosphorus (P); sulfur (S); chlorine (Cl); potassium (K); chromium (Cr); manganese (Mn); iron (Fe) and zinc (Zn). The percentage relationship of removal by the ANAC anaerobic reactor system can be evaluated. Table 6 presents the percentage values of removal of chemical compounds.

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Table 6 - Percentage of removal over the course of the five-week study.

Chemical element	Percentage removed (%)
P	50,05
S	60,25
Cl	51,12
Towards	51,15
Cr	10,00
Minutes	32,26
Faith	37,74
Zn	52,63

Table 6 shows that the percentage values of removal of chemical elements and heavy metals were significant, especially sulfur, which had a removal efficiency of 60.25%.

Such relevance is important since according to Oga (2003) compounds such as sulfur (S) widely used in the detergents and derivatives industries, have high toxicity to the human body, causing from skin irritation to ulcers that can cause malignant stomach tumors.

5 CONCLUSION

The scientific research of alternative treatment systems such as the anaerobic reactor, in the case of the study the compartmentalized RAC, comes to join efforts to obtain a wastewater of lower environmental impact and possible reuse, for example, in industrial and agricultural activities.

Being an environmental tool of relevance in association with processes such as in pig farming.

The period of monitoring and collection of samples that went through the process of detection by synchrotron radiation showed promising results, however it is still necessary to continue studies for a longer period of at least 2 years to observe the functioning of the RAC with a greater sludge stabilization over time.

The best initial results were undoubtedly for the compounds: phosphorus (50.05%); sulfur (already commented); chlorine (51.12%); potassium (51.15%) and zinc (52.63%).

It should also be noted that the RAC system if implemented with another treatment system can raise the metal removal system much more, in such a way as to generate a reuse wastewater of greater potential for application, for example, agricultural.

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