

Analysis by ICP-MS spectrometry and ICP-OES spectrometry techniques for elements contained in residual powder of fluorescent lamps

Análise pelas técnicas de espectrômetria de ICP-MS e espectrômetria de ICP-OES para elementos contidos em pó residual de lâmpadas fluorescentes

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ABSTRACT

The process of lighting public and private areas was an important point for the well-being of humanity and guarantee of public and residential safety. The advances of luminous devices achieved by illustrious scientists such as Thomas Edson, Nicola Tesla, among others, were fundamental to the advancement of the progress of humanity. However, one must take into account the harmful effect on the environment generated by the residue of these new luminous devices. Environmental engineering, above all, has an important role to analyze and evaluate the degree of this waste and ensure a sustainable industrial process, especially in this twenty-first century in which it is increasingly necessary to measure the possible environmental impacts. Fluorescent lamps are one of the luminous devices that must be studied constantly. The present research focused on evaluating through the subsidy of ICP spectrometry techniques the chemical compounds present in fluorescent lamp residues, in order to have a view of the elements contained and their impact on the leaching process. Such research, carried out with the support of UNICAMP's chemical center, was vital for the implementation of this study as an initial starting point for future discussions in other future projects. The values obtained from the chemical elements were tabulated and showed the reflection of these residues present.

Keywords: Luminous effect, Rare earths, Environmental chemistry, Mercury.



1 INTRODUCTION

Civilization has gone through a whirlwind of scientific advances that have culminated in the current evolutionary stage in which it finds itself. Certainly, artificial lighting was one of those advances that provided a better quality of life to humanity. Among those responsible for the implementation of artificial lighting one could not fail to highlight the advances developed in the year 1879, by the inventor and American scientist Thomas Alva Edison. Its electric lamp was undoubtedly for a long time, the most used technology in the world, but with technological advances it came, gradually, losing space for more efficient lighting alternatives, which do not waste energy in the form of heat.

During the twentieth century there was undoubtedly an awakening of humanity to the problem of reconciling development and the advantages of modern life with the conservation of the environment.

The balance between technological development and sustainability, especially at the point of artificial lighting, has brought important adverse effects, such as increased pollution and a series of climate imbalances.

The advancement of engineering, especially the new academic trends variants of engineering such as chemical engineering and environmental engineering have come as important actors in the study of the harmful reflections of waste from artificial lighting. Such studies from the impacts generated called attention to the urgency of reviewing the relationship of man with the environment and consequently to the creation of a new productive logic that takes into account the sustainability of life on planet earth. To achieve the goal of sustainable development, it is necessary to adopt a series of actions that must be carried out in an integrated manner, among them, several related to the energy issue. Considering that the energy sector is responsible for important negative impacts on the environment, policies that aim to contain and mitigate these damages are increasingly common, either through the search for energy efficiency, the result of technological development, or through population awareness and the consequent more rational and efficient electrical consumption.

In 2008, with the estimate that primary energy demand could increase by 55% from 2005 to 2030, bringing serious risks to energy security and environmental sustainability, the *International Energy Agency* (IEA), developed a document recommending 25 energy policies that, if implemented, could reduce the emission of carbon dioxide (CO2) on the planet by 20% per year by 2030, the equivalent of 8.2 Gigatons of CO2 per year (IEA, 2011).



Such recommendations raised by the IEA cover the sectors of energy-consuming equipment, lighting, transportation and industry. Among the recommendations related to lighting is the policy of banning incandescent light bulbs by governments as soon as it is economically and commercially viable. To achieve this goal it was recommended that the establishment of goals and an implementation schedule, and also that government actions should be coordinated internationally in conjunction with the industrial sector, thus ensuring the supply of efficient and good quality lamps. After the publication of such recommendations, some countries, such as the United States, Argentina, Australia, and the member countries of the European Union, established schedules for the withdrawal of incandescent lamps from their markets (BASTOS, 2011). Following this sustainability policy, on December 31, 2010, the Brazilian government published an interministerial ordinance, establishing a schedule for the implementation of minimum energy performance indices for incandescent lamps produced and/or marketed in the country, with the focus of gradually banning these products from the Brazilian market (MME, 2010).

2 OBJECTIVE

The purpose of this research was to address by ICP spectrometry technologies the composition of chemical elements present in fluorescent lamp residues. The use of ICP spectrometry allows a chemical proof of high efficiency of the compounds present in order to estimate their presence in the residue of artificial lighting devices. Thus, it is possible to simulate in the laboratory the effect of acid rain in the leaching process of mercury present in lamps, in order to estimate the environmental damage of the improper disposal of this material in nature.

3 LITERATURE REVIEW

The topic of fluorescent lamps is an important focus of study and its vast academic and scientific study material to date. Thus, it is necessary at this point to raise a portion of this important theme for reflection and scientific sharing.

3.1 FLUORESCENT LAMPS

Despite the first tests of incandescent lamps (pioneers) developed by Thomas Edson, the advances of artificial lighting were undoubtedly realized with the adoption of fluorescent lamps created by Nicholas Tesla and commercialized in a timid way from 1938 (NAIME and GARCIA, 2004).



According to Júnior and Windmöller (2008) fluorescent lamps have been gaining increasing adherence of consumers to the incandescent lamp for its more energy efficient character. Despite the higher cost, its useful life can be up to 15 times farther, in addition to the reduction in electricity consumption by up to 80%.

3.1.1 Components, types and operation

According to Júnior and Windmöller (2008) the process of artificial lighting by the technique of fluorescent lamps comprises the principle of light emission from a transparent glass tube, with two electrodes, one at each end, a mixture of gases at low pressure (an inert noble gas, usually argon, added to mercury vapor) and a luminescent material that internally coats the tube, usually phosphorus powder. When the switch is turned on, the electrodes emit electrons, which collide with the mercury (Hg) atoms present inside the lamp, thus causing the emission of energy in the form of ultraviolet (UV) radiation. As reported by Júnior and Windmöller (2008), the emission of light occurs because the fluorescent layer, which coats the inner surface of the tube, converting this radiation into visible light. Figure 1 shows the luminescent device in detail.



Source: Profelectro

Bastos (2011) states that fluorescent lamps can be classified according to their format into: tubular, circular or compact. Because they are much larger, tubular-shaped fluorescent lamps cannot be easily used to replace traditional incandescent lamps. This finding due to this deficiency enabled the emergence of the first compact fluorescent lamps. Bastos (2011) comments that compact fluorescent lamps have the benefit of having the same type of nozzle, with similar dimension, and a luminous flux with lower power, which generates energy savings, and superior service life. The IEA (2006) estimates that about 25% of the energy consumed by compact fluorescent lamps is converted into visible light, while for conventional incandescent lamps the proportion in circular format, although it is still less usual in Brazil such lamps have a performance



similar to compact fluorescent lamp and also have Edson type thread (BASTOS, 2011). It is worth noting that the Edison-type screw is the name given to the thread-shaped fastening mechanism of incandescent bulbs. Figures 2, 3 and 4 presented below, show the different types of fluorescent lamps in the market.







Source: https://www.philips.com/global

3.1.2 Waste fluorescent lamps

As Bastos (2011) reports in relation to the NBR 10.004 standard of ABNT, where the waste generated by fluorescent lamps is classified. These according to their criteria of dangerousness, handling and destination. Fluorescent lamps are classified after their use as hazardous waste from non-specific sources, class I waste, under code F044. The NBR 10.004 of ABNT as pointed out by Bastos (2011) started to include lamps containing mercury in Annex A after a review in 2004, regarding the toxicity of mercury (Hg) present in the luminous devices.



3.1.2.1 Residual mercury (Hg): Potential risks and actual harm to human health

For NEMA (2005) the vision in relation to fluorescent lamps comes with the false idea of them being less harmful to the environment, since they are lamps that minimize the need for energy generation, in addition to causing less waste. However, as NEMA (2005) discussed, this is not such a trivial issue. This type of lamp represents an important risk to human health and the protection of ecosystems, mainly due to the fact that it has a high content of mercury (Hg) in its composition. The presence of mercury is necessary for the operation of fluorescent lamps because it is associated with their longevity and energy efficiency.

As the MMA (2015) well remembers mercury is the most volatile of all metals, and by this characteristic, its main source of environmental contamination is through atmospheric emission. When released, it remains in the environment, circulating between: air, water, sediment, soil and biota in general, and may assume other chemical forms by chemical assimilation to other chemical elements, consequently having a prolonged toxic effect.

In relation to the toxicity of mercury one of the main channels of human contamination is in the exposure of the diet, especially through the consumption of contaminated fish, suffice it to recall the unfortunate episode of Minamata in Japan in the early twentieth century, around the 50s, in relation to the fishing village. According to Silva (2013) the village of Minamata had an intoxication that affected at least 2,252 people, being fatal to 1,043, a fatality percentage of 46.32%.

As Bastos (2011) and MMA (2015) report on Hg, it is known that once dispersed in nature, mercury is easily leached into aqueous systems, in which it naturally turns into methylmercury, one of the most toxic forms of Hg present in the environment. As a reflex as the UNEP (2002) exposes in relation to the toxicity of Hg (especially methylmercury), it has great chemical assimilarity to reach and compromise the central nervous system. As the first effects of contamination in adults there is the presence of non-specific symptoms such as: paresthesia, malaise, and blurred vision. In cases of persistent and cumulative exposures, the signs are: loss of visual field, deafness, dysarthria and ataxia culminating in coma and death (UNEP, 2002).

According to the WHO (2005) about 80% of the mercury vapor inhaled by the lungs is absorbed into the blood, and when exposed at high levels its effects are felt in the nervous systems: nervous, digestive, respiratory, immune and kidneys.

For Júnior and Windmöller (2008) when it comes to fluorescent lamps, studies show that the interaction of metal with phosphorus powder and glass also could convert mercury into other more dangerous chemical forms and with greater mobility in the environment. This highlights the



concern of the EPA (2014) that explains that when breaking a lamp that contains mercury, immediately a small part of this element is released in the form of vapor. Most of it, which is retained in the waste also releases the compost, but gradually, being something of great danger to the environment and to man.

The residual concern of fluorescent lamps is such that Johnson *et. al.* (2008) conducted a study in the USA with fluorescent lamps of 13 W power and demonstrated that after 4 days of monitoring, about 30% of the total mercury contained in the lamp was dispersed in the environment. Such results were so alarming that Pacyna *et al.* (2010) conducted a supplementary study in which they arrived statistically at estimates of global mercury emissions released into the atmosphere in 2005, by the lighting sector, around 1 to 2 tons, and according to these studies through a statistical mathematical model concluded estimated forecasts for 2025 of Hg emissions between 5 to 13 tons.

3.2 TECHNIQUES OF SPECTROMETRY BY ICP-OES AND ICP-MS

According to CAIQ (2023) the process comprised by the technology of optical emission spectrometry with inductively coupled plasma called ICP-OES, as well as the mass spectrometry with inductively coupled plasma known as ICP-MS are technological tools that allow a wide range of detection and quantification of chemical elements present in a given material under analysis.

According to Smith and Nordberg (2015) the point of similarity between the use of ICP-MS or ICP-OES comprises the use of inductive plasma. The purpose of the use of plasma is to ensure the ionization process and consequent detection of the elements present in the sample submitted to the process. The plasma source used is formed by ionization of an inert noble gas, usually argon (Ar), which flows in a torch wrapped in a copper coil (Cu) connected to a radiofrequency generator. When this gas is flowing, a spark is created, called an electron seed, which generates the beginning of the ionization of the argon and, with this, a frequency range between 27 and 40 MHz is applied to the charge coil to accelerate the electrons supplied. Regarding the transfer of energy from the ICP-OES and ICP-MS techniques that pass to the electrons through the coil according to Smith and Nordberg (2015) is called inductive coupling. In this coupling, the electrons are: highly energized inducing further ionization of the gas in which results in a plasma with temperatures ranging between 7000-10000 K, which are sufficient to allow atomization and ionization of most chemical elements.

For Cazes (2005) a ICP-OES has as a process the electromagnetic radiation emitted by atoms or ions excited by the plasma in the regions of the visible and ultraviolet spectrum that



comprise the spectral range from 160 to 800 nm. In this case, the sample to be analyzed is used in the form of a solution and undergoes a nebulization process that results in its vaporization and, consequently, atomization and excitation, which can be measured in the emission spectrum. In this case, due to the particularity and individuality of each chemical element corresponding to a specific relative wavelength range. However, Cazes (2005) reports that there are limitations to the determination of atoms C, H, O and N due to the use of solvents that contain them in their composition and because they are normally present in the air, they cause large interferences in their records.

When analyzing the ICP-MS, Smith and Nordberg (2015) argue that the process is based on measuring the number of ions generated when the sample, which undergoes previous nebulization, is heated by the plasma and these are separated by a magnetic field according to their mass-versus-charge ratios. In ICP-MS the analysis is performed with low detection limit, usually in the order of picogram per liter (pg.mL⁻¹).

3.2.1 Applicability of ICP-OES and ICP-MS spectrometry

When the application of the spectrometry techniques of ICP-OES and ICP-MS is verified, according to Cazes (2005) both have the practicality of determining a multielemental analysis in a single measurement and because they use a heating source, such as plasma, it can be applied to the study of samples from the most diverse origins, such as: soil, metallurgy, agricultural, processed or natural foods, petroleum products, biological materials and pharmaceuticals.

For Cazes (2005) one of the primary objectives of the ICP-OES and ICP-MS techniques are undoubtedly to evaluate the residual compounds present as results of industrial processes.

4 METHODOLOGY

In this topic is presented the method of execution of scientific research based on methodologies already widespread for the tests.

4.1 COMPONENTS USED FOR ANALYSIS: MATERIALS, REAGENTS AND EQUIPMENT

For the evaluation process, some chemical materials, and reagents necessary for the elaboration of the chemical process were used according to the methodology of Saint'Pierre (2010). Which were: bidistilled nitric acid (HNO₃); sulphuric acid 96 % (H2SO4); hydrochloric acid 36 % (HCl); lithium metaborate P.A. (LiBO₂); variable volumetric pipettes; 15 mL and 50 mL polypropylene tubes; tweezers and spatula of metal; water dispenser; and metal crucible.



Regarding the necessary equipment used in the research, we have the use of: ICP-MS spectrometer (*NexIon 300 X, PerkinElmer, USA*); ICP-OES spectrometer (*Optima 4300 DV, PerkinElmer, USA*); heating plate; peagameter; and Meker's beak.

4.2 PREPARATION OF FLUORESCENT LAMP RESIDUES

Samples of inoperable fluorescent lamps were ground for the preparation of assays and analyses by ICP spectrometry techniques. Care was taken to grind the material in a *glove box* at the chemistry laboratory of Unicamp. It is important to be careful not to be exposed to the toxic material contained in the residue of the lamp represented, especially by volatile mercury. Figure 5 shows an overview of the *glove box* used in the experiment.



Figure 5: Glove box used in the laboratory.

Inside the glove box were placed petri dishes with 20 g of activated carbon in order to adsorb part of the mercury vapor released inside the glove box. The adsorption process is a chemical concept that is inserted in the assimilation of the chemical elements on the surface of the activated carbon for further analysis. The lamps were broken and ground internally *the glove box* with the aid of the inner gloves of the chamber, maintaining the protection against exposure of toxic elements. The powder generated was stored in clock glass in a 50 mL tube (SAINT'PIERRE, 2010).

4.2.1 Phase of acid dissolution of powder

Form collected 50 mg of the residual powder and allocated in polypopylene tubes of 15 mL with the addition of the acidified preparation. For such preparation the sample had the addition of 1 mL of bidistilled HNO 3, HCL in the stoichiometric ratio of 3:1 (3HCL : 1HNO3) respectively, and placed in heating plate at 900C until dissolution occurs (SAINT'PIERRE, 2010).



The samples were packed in glass in a volume of 10 mL for the analysis phase by the ICP-MS and ICP-OES techniques.

It should be noted that in this phase the preparation was carried out in a chapel with the use of a protective mask and waterproof gloves.

4.2.2 Alkaline fusion phase for ICP-OES analysis

In this phase, using the methodology of Saint'Pierre (2010), 45 mg of the ground powder was weighed in a metal crucible and mixed with 0.5 g of LiBO₂. The so-called alkaline melting phase is carried out by heating the mixture to 8000C (in a special oven) with a Meker nozzle until the complete melting of the crucible material. After this phase, the result of the Becker fusion containing 50 mL of 3 10% HNO is collected and after 10 minutes the resulting product was transferred to a 50 mL polypropylene tube for analysis by the ICP-OES technique.

4.2.3 Hg leaching test phase

The evaluation of the degree of impact on the environment by the leaching process was carried out by acid rain of mercury (Hg) present in the contaminated fluorescent lamp, in order to estimate the impact on the environment by the irregular disposal in landfills (JÚNIOR and WINDMÖLLER, 2008).

In the procedure as Saint'Pierre (2010) formulated, 50 mg of powder from the ground material and 2.5 g of powder-free glass were collected and transferred to a 50 mL polypropylene tube. The material was treated with pH solutions of acidic values: 6.1; 5.1 and 4.1. This addition was made with the addition of drops of 0.1 mol stock solution. ^{L-1} sulfuric acid (H_{2SO4}) in deionized water. The resulting solutions were left for 24 h in an agitator to reproduce the effect of water movement suffered by a residue in the environment. After the preparation phase, the samples were centrifuged and later analyzed by the ICP-OES technique.

4.3 ICP SPECTROMETRY ANALYSIS EQUIPMENT

The use of ICP spectrometry is widely used in analytical chemistry for multielemental determination through its high sensitivity and low limit of quantification that enables the detection of elements in trace concentrations (SAINT'PIERRE, 2010).



4.3.1 ICP-MS spectrometry

Second Saint'Pierre (2010) the use of ICP spectrometry has as a procedure the ionization and atomization of the analytes of the sample through a high energy source. The term aNalite refers to a substance or chemical component, in a sample, that is the subject of analysis in an assay, in the case of the scientific study in question are the elements contained in fluorescent lamps.

In the PCI process, a concentric nebulizer is used that introduces the liquid sample in aerosol form into the center of a generated plasma. Skoog and Holler (2006) report that plasma is an ionized gaseous mixture containing a high concentration of cations and electrons. In ICP equipment, plasma is normally produced by argon gas in a quartz torch, and the plasma reaches a temperature of around 5,000 K (4,727oC) which allows the almost complete atomization and ionization of the analytes present in the study sample.

Figure 6 shows the ICP-MS equipment used in the scientific study that is located in the chemical laboratory.



Figure 6: ICP-MS spectrometer, model NexIon 300 X from PerkinElmer used.

Table 1 (below) presents the adjustment of the calibration used in the ICP-MS equipment in order to observe the concern in the adjustment of the calibration developed according to Skoog and Holler (2006).

Parameter	Adjusted value
RF Power	1300 W
Argon (plasma) flow rate	15 L.min-1
Argon flow (auxiliary)	1 L.min-1
Argon flow (nebulizer)	0.92 L.min-1
Aspiration rate	1.50 L.min-1
Number of Reads	Triplicate

Table 1 – Values used in the calibration of the research in the ICP-MS.



4.3.2 ICP-OES spectrometry

The diffractionation between the ICP techniques is basically in the generation of signals, where in the ICP-MS the ions must be transferred to the mass spectrometer, in the ICP-OES it must be in the propagation of radiation.

Another factor is in the emission of radiation and the mass spectrum. Figure 7 (below) shows the ICP-OES equipment used in the study.



Figure 7: ICP-OES spectrometer, PerkinElmer model Optima 4300 used.

Regarding the calibration adjustment of the equipment, the calibration values can be observed in table 2, below.

Parameter	Adjusted value
Power	1400 W
Argon (plasma) flow rate	15 L.min-1
Argon flow (auxiliary)	0.5 L.min-1
Argon flow (nebulizer)	0.6 L.min-1
Aspiration rate	1.50 L.min-1
Number of Reads	Triplicate
Data Acquisition	Peak area

Table 2 – Values used in the calibration of the research in the ICP-OES.

5 RESULTS

5.1 DETECTED ELEMENTS

The results obtained by the chemical techniques and procedures are evaluated are presented below.

5.1.1 Elements detected by dissolutions in HNO3, HCl and Água Regia

The chemical elements recovered (detected) by the IPC techniques are presented in table 3



(next page) in relation to the powder samples of the lamps. It is important to highlight that according to the methodology used, the elements suffered dissolution by acid attack, according to the values obtained in table 3.

ICP-MS					ICP-OES		
Chemical element	HNO3	HCl	Regal Water	HNO3	HCl	Regal Water	
Y	-	-	-	50	50	51	
Eu	2,71	2,40	2,80	3,1	3,1	3,1	
Ce	2,04	2,10	2,31	-	-	-	
La	1.15	1.20	1,28	-	-	-	
Tb	0,130	0,140	0,138	-	-	-	

Table 3 – Values detected in μ g.g-1 of the lamp powder dissolved in HNO3, HCl and aqua regia by the different techniques of ICP spectrometry.

Table 3 shows that the chemical element obtained in the highest concentration was Yttrium (Y) in aqua regia in the value of 51 μ g.g-1. It is important to note that the ICP-MS and ICP-OES techniques should be used together to minimize the degree of technical limitation of each, since individually they have certain limitations, as shown in Table 3.

Table 3 shows that the chemical elements Ce (Cerium), La (Lanthanum) and Tb (Terbium) by the ICP-OES technique due to the fact that there is not enough sensitivity to determine low concentration chemical elements.

5.1.2 Leaching test of waste material

The tests for leachate powder and leachate glass are presented in table 4 below. Remembering that the test was performed by the process of natural leaching of the mercury present and measured by the ICP-OES technique.

	L	eached powdo	er	Leached glass		
Colution	pHs			pHs		
Solution	6,1	5,1	4,1	6,1	5,1	4,1
Concentration		(µg.g-1)		(µg.g-1)		
Hg	9,2	14	51	0,10	0,18	0,40

Table 4 – Concentration (μ g.g-1) quantified in relation to the acid pH range after simulation of leaching by acid rain measured by ICP-OES.

According to Jowitt *et al.* (2018) the presence of atmospheric carbon dioxide dissolved in rainwater causes the pH to be slightly acidic (pH value equal to 5.7) due to the production of carbonic acid. Still based on what it says Jowitt *et al.* (2018) The Presence of nitrogen (N) and



sulfur (S) oxides in the atmosphere, emitted by anthropic actions, usually further decrease the rainfall pH, by the formation of HNO3 and H2SO4.

Jowitt *et al.* (2018) comment that arain is considered acidic when its pH decreases more than 5.6. Thus, the tests performed and cataloged in table 4 (previous) were made based on these considerations.

Table 4 shows that the highest intensity concentrations were obtained for pH equal to 4.1, which corroborates the observations of Jowitt *et al.* (2018).

The values raised in tables 3 and 4 are below the limit established in the Brazilian standard for material considered hazardous waste for disposal in special landfills than by the ABNT standard (1987a). This is since in cases of hazardousness of residual material the permissible values are 100 ^{mg.kg-1}, as Junior and Windmoller (2008) point out.

6 CONCLUSION

ICP spectrometry techniques are technologies that come to contribute to the environmental chemical study to measure the degree of impact of chemical elements present. It is worth mentioning that only in table 4 it was possible to evaluate the degree of mercury by the leaching procedure. In the tests in table 3, due to the fact of the high degree of volatilization of Hg, it was not retained.

The research led to the detection of chemical elements in fluorescent lamps that are called rare earths that have high monetary value.

Elements such as Yttrium (Y) and Europium (I) according to Schuller *et al.* (2012) has international values around 63.00 and 800.00 US\$.^{kg-1}, respectively. Being attractive for the research of recovery of these elements for commercialization and elimination of this residue in the ecosystem.

When evaluating the leaching tests for the effect of acid rain of mercury present in fluorescent lamps, the values in tests found in table 4, show the high degree of risk to the environment since the decay of pH has in return a resurgence of the present concentrations of mercury (Hg) that contaminate the entire biota of planet Earth.



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