129

Série
Tecnologia
Ambiental

Exhausted fluorescent bulbs: an important target for urban mining of rare earth elements

Fernanda Veronesi Marinho Pontes
Jéssica Frontino Paulino
Manuel Castro Carneiro

CETEM 45 anos
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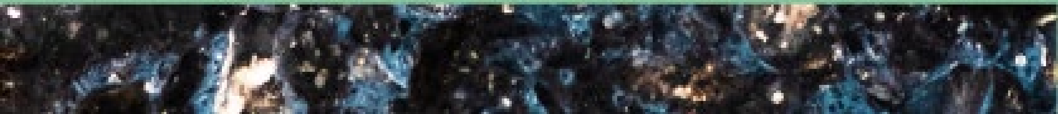

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Exhausted fluorescent bulbs: an important target for urban mining of rare earth elements

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Fernanda Veronesi Marinho Pontes
DSc. Instituto de Química - UFRJ. Professora Adjunta do Instituto de Química - UFRJ.

Jéssica Frontino Paulino
DQA - IQ - UFRJ.

Manuel Castro Carneiro
PhD Universidade de Barcelona. Pesquisador Titular CETEM/MCTI.

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There is no single definition that fits the wide diversity that the theme “Environmental Technologies” covers. First, the field of Environmental Technologies is characterized by a high degree of diversity and heterogeneity. In general, the term is used to include technologies and applications that are supposed to help reduce the negative impact of industrial activities and services, by private or public users, on the environment. The concept usually refers to technologies "at the end of the process" (end-of-pipe) integrated with clean technologies and recovery of contaminated areas. However, it can also cover broader issues such as monitoring, measuring, changing products or managing environmental systems. Environmental technologies are, therefore, of an interdisciplinary nature and can be applied at any stage of the production-consumption chain. Bearing this in mind, the “Environmental Technology Series” aims at bringing together specialists, such as: researchers, technologists, professors etc., from CETEM in particular, to disseminate their research in such diverse areas to serve as a stimulus for new and future researchers.

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Informações:
CETEM – Centro de Tecnologia Mineral
Av. Pedro Calmon, 900 – Cidade Universitária
21941-908 – Rio de Janeiro – RJ
Homepage: www.cetem.gov.br

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SUMMARY

ABSTRACT

Electronic and Electrical Equipment Waste (WEEE), also known as e-waste, encompasses a wide range of damaged, discarded, and outdated devices. Included in this category are the exhausted fluorescent lamp. Due to the increasing production of these wastes, coupled with delays in developing policies for collecting systems and environmental management, it is extremely necessary to ramp up the adoption of proactive measures to reduce illegal dumping. Improper disposal of fluorescent lamps (FLs) has become a significant environmental issue due to the implementation of the ban on incandescent lamps and the emergence of new LED (Light Emitting Diode) lighting technologies. In Brazil, the National Solid Waste Policy has established reverse logistics for various products, including FLs, due to the environmental risk associated with their improper disposal. Because of the presence of mercury and other potentially toxic elements in their composition, FLs have become an environmental liability. Reuse and recycling processes are crucial but face challenges, especially in component separation and the recovery of rare earth elements (REEs). The issue of REEs in FL waste is also addressed, highlighting the need to develop technologies for their recovery to mitigate impacts on human health and the environment. The management of electronic waste, including FLs, has become a global concern. Countries like those in European Union have established specific legislation for the recycling and recovery of these wastes. Brazil faces challenges in electronic waste recycling, with low technological potential and a lack of investments. The presence of REEs in FLs is of interest due to their importance in advanced technological applications. Consequently, urban mining emerges as a sustainable alternative to recover these elements from waste, reducing dependence on conventional mining, particularly in light of the scarcity of primary sources. Recycling FLs involves physical, chemical, and biological processes to separate and recover materials such as phosphor powder and mercury. The recovery of REEs from FL waste through hydrometallurgical methods has been studied, aiming at searching for more environmentally friendly processes. Recovering materials from FLs, including REEs, requires processes that align with green chemistry, aiming at reducing the environmental impacts to promote conscious recycling. Despite existing legislation, electronic waste recycling, including FLs, is not a priority in many countries, including Brazil. Considering environmental issues, REEs can be considered contaminants of emerging concern (CECs) due to their widespread global exploitation in the high-tech industrial sector. The presence of anthropogenic REEs in the environment poses an additional challenge to public health and the ecosystem, emphasizing the importance of conscientious recycling of these materials.

Keywords: Fluorescent bulbs, rare earth and mercury.

1 | INTRODUCTION

The growth of the global waste management market is driven by increased adoption of proactive government measures to reduce illegal dumping. Furthermore, the increase in population and globalization has led to the growth of the overall volume of waste around the world. The urban population produced about 1.3 billion tons of urban solid waste (MSW) in 2012, which is expected to grow to 2.2 billion tons by 2025. Allied to this growing regulation, the environmental awareness of consumers and the emergence of stricter environmental regulations have led industries to think about environmental management through the implementation of reverse logistics (RL) (BOUZON et al., 2016). In Brazil, reverse logistics was established as an instrument of economic and social development, by the National Solid Waste Policy in 2010, comprising a set of actions, procedures and means to enable the collection and return of solid waste to the business sector after consumption.

In the classification of urban solid waste, electronic waste, also known as e-waste, stands out. In 2014, the global generation of e-waste reached 44.4 million tons, and after five years, this number reached 53.6 million tons. With this rapidly growing waste stream, the projection is that by 2030, this number will reach 74.7 million tons (AWASTHI et al., 2022). Electronic waste encompasses a wide range of damaged, discarded, and outdated devices. Fluorescent bulbs waste is included in this category. Post-consumer fluorescent bulbs are required to return to their manufacturers, so that they can provide the proper final destination. These lamps are classified as hazardous waste and their inappropriate disposal can cause problems for the health of the population and the environment (DE FARIAS et al., 2020).

The history of fluorescent lamps begins with the invention of the mercury vapour lamp, patented in 1901 by Peter Cooper Hewitt, which worked by passing an electrical current through mercury vapour at low pressure. Operating at low voltage, these were more energy efficient than the incandescent lamps produced by General Electric (HULL, 1931). Mercury vapour lamps continued to be developed at a slow pace, especially in Europe, and by the early 1930s they were used to a limited extent for large-scale lighting. Some employed fluorescent coatings, but these were mainly used for colour correction rather than for actual lighting. Finally, sales of "Lumiline" fluorescent bulbs began in 1938, when four different sizes of tubes were placed on the North American market.

According to Gensch et al. (2015), there are two basic types of fluorescent lamps (FL): compact fluorescent lamps (CFLs) and linear fluorescent lamps (LFLs). Both are energy-saving lamps, available in a wide range of wattages, types and sizes, and work identically. CFLs are regularly used for both commercial (*i.e.*, retail, office, hospitals and leisure) and residential use as energy-saving alternatives, while LFLs are preferably used in commercial facilities.

In a fluorescent lamp, the transfer of electrical energy to visible light takes place in two stages. First, electrical energy is partially transformed into invisible ultraviolet radiation, mainly with a wavelength of 253.7 nm. The amount of visible radiation produced by the discharge itself is small compared to ultraviolet radiation. In a second step, the ultraviolet radiation (UV), produced by the discharge, focuses on the fluorescent powder (phosphorus), which is located on the inner wall of the glass tube and this, in turn, emits visible light. Figure 1 shows a lamp connected to a direct current (DC) source; when the lamp is energized, a potential difference (V = V2 - IR) appears between the electrodes. On average, electrons travel from the negative electrode (cathode) to the positive electrode (anode), while positive ions, whose average displacement speed is much slower than that of electrons, travel from the anode to the cathode.

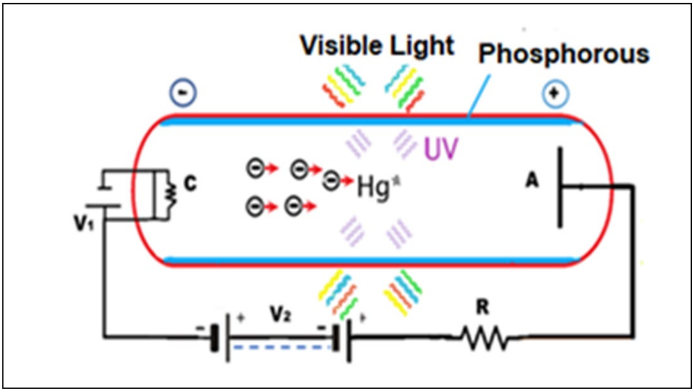

Figura 1. Gas discharge tube in series with a resistor R and a direct current (DC) source V2. Voltage V1 serves to heat the cathode, C is the cathode and A is the anode.

Since electrons are emitted from the cathode and travel to the anode, there must be a continuous supply of electrons from the cathode. The process of delivering electrons from the cathode is known as electron emission. Also, positive ions and electrons are created throughout the discharge. When electrons collide with mercury atoms, they ionize this element, resulting in the emission of electromagnetic radiation in the ultraviolet region. The phosphorus compound that coats the glass tube internally absorbs UV radiation, turning it into visible radiation. The color of visible light depends on the chemical composition of the phosphorus deposited on the inner surface of the glass tube. The glass tube is coated with alumina and phosphorus powder. According to Anand and Singh (2021), phosphorus compounds, which contain rare earth elements (ETR), are composed of about 34% of yttrium, 2% of europium, 12% of lanthanum, 4.5% of cerium and 2.7% of terbium.

Phosphorus powder is basically composed of a mixture of calcium halophosphate [$Ca_5(F,Cl)(PO_4)_3$], or strontium, doped with Sb^{3+} and Mn^{2+}, with compounds containing rare earth elements, which are classified as red, green or blue phosphorus, according to their chemical composition (TUNSU et al., 2016; ANAND; SINGH, 2021). Red phosphorus is composed of $Y_2O_3{:}Eu^{3+}$ (YOX). Green and blue phosphorus are composed of phosphates, aluminates or borates bearing REE, such as: $CeMgAl_{10}O_{17}{:}Tb^{3+}$, $LaPO_4{:}Ce^{3+}$, Tb^{3+}(LAP), $(Ce,Tb)MgAl_{11}O_{19}$(CAT) or $(Ce,Gd,Tb)MgB_5O_{10}$, in the case of green phosphorus, and $BaMgAl_{10}O_{17}{:}Eu^{2+}$ (BAM) and $(Sr,Ca,Ba)_5(PO_4)_3Cl{:}Eu^{2+}$, making up the blue phosphors (TUNSU et al., 2016; ANAND; SINGH, 2021; SHUKLA et al., 2021). More commonly, luminescent powder contains a mixture of these three types of phosphorus, where red phosphorus corresponds to about 50% (TUNSU et al., 2016).

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2 | WASTE MANAGEMENT OF FLUORESCENT LAMPS DISCARDED IN DIFFERENT COUNTRIES

Due to the presence of mercury in its composition and taking into account the volume of waste generated, whose use has grown dramatically due to the policy of banning incandescent lamps, fluorescent lamps ended up becoming an environmental liability. The generation of FL waste is expected to become increasingly larger due to Brazil's commitment made at the fourth meeting of the Conference of the Parties to the Minamata Convention (2022) to ban compact fluorescent lighting by 2025 (CLEAN LIGHTING COALITION, 2022).

According to Binnemans and Jones (2014), useless fluorescent lamps are being collected separately from other municipal waste streams, not because of their REE content, but because of the presence of mercury, which is an environmental hazard. In addition to mercury, other potentially toxic elements present in FB residues can pose an environmental and human health risk. A study was recently carried out on the environmental mobility of the elements Cd, Cu, Hg, Mn, Ni, Pb and Zn present in FB residues using a sequential extraction scheme (SES) and mathematical modeling of potential non-carcinogenic risks to human health, considering direct disposal of waste in soil. The results showed that Cd, Cu, Hg, Ni and Zn are the elements with the greatest mobility and bioavailability under natural soil conditions and that Cd and Hg showed a potential risk for the ingestion of vegetables by humans (DE FARIAS et al., 2020).

Over the years, the mercury content in fluorescent lamps has steadily declined, but each fluorescent lamp still contains a few micrograms of Hg. In the European Union alone, more than 100 million light lamps are sold annually - so the amount of mercury, around 50 mg kg^{-1}, in lighting waste, is very large. This problem has stimulated initiatives for the management of used lamps in Europe. Thus, stimulated by sustainable environmental legislation, in 1985 the Belgian government, together with 15 private companies, founded Indaver (INDAVER, 2023) starting the destination of chemical industry waste on European soil. Indaver's first location was near the Port of Antwerp. Continuing the company's expansion plans, in 1994 the Indaver unit for the treatment of mercury-bearing waste was inaugurated in Belgium (Beveren, Doel). With the IVKA installation in Doel, fluorescent lamps started to be treated with a thermal process to remove mercury. The facility also included a unit for mercury distillation.

Over the past few decades, the increasing volume of waste electrical and electronic equipment (WEEE) has become a matter of great concern around the world. In the European case, specific legislation has been developed in order to address the environmental problems associated with the proper management of this particular form of hazardous waste (Directive 2012/19/EU). Thus, the new legislation introduced specific targets for the reuse and recycling and recovery of WEEE, which European countries must include in their national policies. European legislation has established 10 categories of electrical and electronic equipment (EEE) and an indicative list of EEE covered by the categories. The "Lighting Equipment" category included the following products: fluorescent lamps (FL); high intensity discharge lamps including pressure sodium lamps and metal halide lamps; low pressure sodium lamps and light emitting diode (LED) lamps.

In accordance with DIRECTIVE 2008/98/EC, the hierarchy presented below is applicable as a general principle of legislation and policy for waste prevention and management: 1) Prevention and reduction; 2) Preparation for reuse; 3) Recycling; 4) Other types of recovery, for example energy recovery and 5) Disposal. Also according to this Directive, some terms received a definition: I) Reuse, any operation whereby products or components that are not waste are used again for the same purpose for which they were designed; II) Treatment, any recovery or disposal operation, including preparation prior to recovery or disposal; III) Recovery, any operation whose main result is the transformation of waste to serve a useful purpose, replacing other materials that would otherwise have been used for a specific purpose, or the preparation of waste for that purpose, at the facility or in the economy as a whole; IV) Preparation for reuse, recovery operations consisting of control, cleaning or repair, whereby products or product components that have become waste are prepared to be reused, without any other type of pre-processing; V) Recycling, any recovery operation through which the materials constituting the waste are again transformed into products, materials or substances for their original purpose or for other purposes. It includes the reprocessing of organic materials but does not include energy recovery or reprocessing into materials that are to be used as fuel or in filling operations and VI) Elimination, any operation other than recovery, even if it has recovery as a secondary consequence of substances or of energy.

Several countries already impose national limits on mercury content in CFLs that go beyond the requirements of the Minamata Convention (including the EU, Russian Federation and India). The average mercury content in CFLs has decreased by at least 20% in recent years (US EPA, 2019b). In the US, the typical amount of mercury in CFLs varies from 0.9 to 4 mg per lamp (COWI and ICF, 2017). Canada has also implemented national regulations for mercury-bearing products. For CFLs for general lighting purposes with a maximum power of up to 25 W, a maximum amount of mercury of 4 mg per lamp has been stipulated. The US imposes standards for the manufacture of lighting lamps through the Energy Star program, introducing a limit of 2.5 mg for CFLs up to 23 W and 3 mg for CFLs above 23 W (UNEP, 2013b).

In Russia and the Eurasian Economic Union (EEU), the EAEU Technical Standard 037/2016 on the restriction of the use of hazardous substances in electronic, electrical and radio products placed restrictions on CFLs that go beyond what was agreed in the Minamata Treaty and are equivalent to those established by the European Directive ROHS 2008/98/EC. That is, 2.5 mg for CFL < 23 W, 3.5 mg for 23-50 W, 5 mg for 50-150 W, 15 mg for >150 W, 7 mg for circular or square lamps and 5 mg for lamps for special purposes.

According to Panwar et al. (2018), the e-waste rules that came into effect in India in 2016 also apply to fluorescent lamps. The rules include specific guidelines that extend producer responsibility and random sampling testing to assess hazardous waste parameters.

In the United States, the Resource Conservation and Recovery Act (RCRA) of 1976 put in place rules for the storage, handling and disposal of household hazardous waste (HHW). Mercury (Hg) has been classified as a Level 1 chemical in the Toxic Substances Control Act's Inventory by the United States Environmental Protection Agency (USEPA). To comply with US legislation, several private companies have launched recycling programs, including by mail. This is the case with LampRecycling.com. Through this site, users can receive EasyPak recycling containers, an easy and safe way to recycle fluorescent lamps, batteries, ballasts and electronics. This is a service where the user fills

EasyPak containers at his own pace and then mails them via FedEx prepaid when ready. Other companies provide similar services, such as TerraCycle Regulated Waste, whose history begins in 1978 in the Chicago metropolitan area under the name AirCycle. As the lighting industry evolved, AirCycle identified the need to package used fluorescent lamps easily and efficiently. At the same time, to comply with legislation and reduce pollution arising from the irresponsible disposal of light lamps, the company designed a machine to meet recycling needs, the 55 VRS Bulb Eater® model (EARTH911, 2021).

In Brazil, according to the 18th Urban Solid Waste Management Diagnosis, published in 2020, the rate of occurrence of selective collection of household waste (HW) of municipalities participating in the National Sanitation Information System - NSIS, from 2017 to 2019, increased by about 14% in municipalities with this service. The results were based on the answer YES or NOT to the question "Is there selective collection in the city?" regardless of the method adopted (door-to-door, voluntary delivery points or other modality). In addition, the study by the United Nations Environment Program (2009) cites barriers to the transfer of electronic waste recycling technology in South Africa, Morocco, Colombia, Mexico and Brazil, which are: (i) low potential technology and (ii) lack of investment and business models. In Brazil, the Waste Management Policy was enacted to regulate waste management in public and private areas. This law establishes the criteria for the reverse logistics of electronic waste, including fluorescent lamps, and other categories of hazardous waste, such as herbicides, lubricating oil, batteries, tires and product packaging. Regarding the technological barrier, the collection and recycling processes are concentrated on components and materials with high added value (such as circuit boards and stainless steel), but other components are underestimated and, consequently, discarded in inappropriate places. Recycling e-waste does not appear to be a high priority issue in the aforementioned countries; furthermore, an additional fee for recycling e-waste seems very unpopular (KHAN et al. 2020).

The minimum recovery targets for gas discharge lamps established by Directive 2012/19 / EU is 80%. According to BOUZON et al. (2016) there are 36 barriers that prevent or hinder the development of reverse logistics in Brazil, which the authors classified into seven categories: (i) technology and infrastructure, (ii) governance and supply chain process, (iii) economic, (iv) knowledge, (v) politics, (vi) market and competitors and (vii) management.

Although Japan is a model country because it has comprehensive laws to control the use of Hg and its disposal, according to Asari et al. (2011), only 25% of fluorescent lamps are recycled. Taiwan, on the other hand, has its own comprehensive policy for managing mercury and has adequate equipment to handle mercury-bearing waste. Additionally, Taiwan has one of the highest recycling rates in the world.

According to Huang et al. (2019), in Taiwan, recycling rates for fluorescent lamps and dry batteries are 88% and 45%, respectively, and in Japan, they are approximately 30% and 26%, respectively. This is despite the fact that Japan has good management systems and technologies to process this waste. Taiwan's recycling rates are unattainable in many countries and show that Taiwan has efficient waste processing technologies and a robust waste management system.

In Japan, the collected fluorescent lamps are crushed and then undergo a washing process, while in Taiwan, crushing is performed with thermal desorption. The methods adopted in Japan have better processing efficiency and are not only focused on fluorescent lamps, but also on other wastes bearing mercury and its constituent compounds. On the other hand, the methods followed in Taiwan are applicable on a smaller scale and are only suitable for fluorescent lamps. As both technologies have their own advantages and disadvantages, countries should consider national conditions and assess them before adopting them. With regard to mercury, Taiwan's front-end recycling system is relatively better managed, while Japan has a relatively complete solidification technology and specification management system in the back-end. In response to international trends, Taiwan and other countries must apply more regulated and environmentally sound storage methods for recovered mercury (KUANG-WEI et al. 2019).

The rare earth elements (REE), a group of 17 elements composed of the lanthanides, scandium and yttrium, are attracting world attention due to their importance in a wide variety of advanced technological applications. Demand for REE has been increasing, particularly due to REE applications in the renewable energy and defence sectors, with a huge expectation of worldwide REE supply shortages in the future. The production of consumer goods to meet the demands of contemporary society requires the exploration of natural resources, often non-renewable (gas, oil, for example). The speed with which products become obsolete further aggravates problems with the environment, which becomes the destination of waste generated and improperly disposed of in most cases.

In this context, based on the concept of urban mining, where waste generated by cities becomes an important source for the recovery of components and/or elements with high economic added value (EAV), REE could be reintroduced into the production chain using urban prospecting techniques. This could serve as a means to mitigate the negative environmental impacts caused by anthropogenic products. Given that REEs are considered critical elements for the future and are classified as strategic resources in several countries, there is a need for the exploration of new and alternative sources of REEs."

It is worth noting that the residues of fluorescent lamps have significant levels of rare earth elements (REE) when compared to the respective ores (JHA et al., 2016). Luminescent phosphorus can contain up to 8% w/w of these metals (LEITE et al., 2020). They have unique optical, electrical and magnetic characteristics, which are essential for the development of advanced technologies. At the end of the life of a fluorescent lamp, the phosphorus can be reused, or even recycled to obtain products rich in REE. Obtaining these metals from secondary sources is a sustainable alternative to primary production processes, contributing to the correct management of this type of waste.

China holds one of the world's largest reserves of REE ore, followed by Vietnam and Brazil (U.S. GEOLOGICAL SURVEY, 2021). In addition, China, which is also the world's production leader, controls prices, directly influencing the development of projects in other countries (BRASIL, 2019), making the exploration of new reserves economically unattractive, which must also take into account the responsibilities socio-environmental REE extraction through conventional mining affects ecosystems, altering landscapes and dispersing potential pollutants in the soil, atmosphere and water bodies, compromising fauna and flora. In this way, urban mining, that is, obtaining these metals from secondary sources, is shown as a parallel alternative to primary production processes, contributing, in addition, to the solution of one of the biggest environmental problems, installed in large Brazilian cities, which are sanitary landfills.

According to Corrêa and De Figueiredo (2020), in Brazil, over three decades, an open-air landfill operated almost out of control. Located in the metropolitan region of Rio de Janeiro, the Gramacho landfill is an example of this problem. The population of the capitals and surroundings has generated a large amount of waste. Due to frequent garbage collection, issues related to waste in urban centers are barely noticed.

The reuse and recycling processes require strict control in order to guarantee the quality of the products obtained, for which chemical and technological characterizations are necessary. The following sections present some techniques used for characterization and some methods used to valorize these residues as raw material.

3 | CHEMICAL AND TECHNOLOGICAL CHARACTERIZATION

Chemical and technological characterization is a powerful tool that has contributed with relevant information for the sustainable development of processes that involve the recovery/recycling of rare earth elements (REE) in fluorescent lamp waste. In the literature, few works address characterization as a main topic. Generally, the characterization of this matrix is briefly described at the beginning of works that study the REE recovery processes (BELARDI et al., 2014). The analytical procedures used in the chemical and technological characterization of fluorescent lamp residues encompass the following topics, (i) particle size determination, (ii) elemental chemical composition, (iii) identification of crystalline phases and morphology, (iv) thermal analysis and (v) chemical analyzes (SHIMIZU et al., 2005; URNIEZAITE et al., 2010; DE MICHAELIS et al., 2011; BELARDI et al., 2014; TUNSU et al., 2014; COSKUN; CIVELEKOGLU, 2014; TUNSU et al., 2016; HOPFE et al., 2017; GIJSEMANS et al., 2018; INNOCENZI et al., 2018; YIN et al., 2018; ANAND et al., 2019; DE FARIAS et al., 2020; SHUKLA et al., 2021).

3.1 | Determination of Particle Size

Grain size classification is the preliminary procedure performed to determine the size of the particles that make up a given sample. In this procedure, sieves with different mesh opening diameters are used for such classification (DE MICHELIS et al., 2011; BELARDI et al., 2014; TUNSU et al., 2016). The scanning electron microscopy (SEM) technique and laser source have also been used to determine the size distribution of particles containing REE by obtaining digital images (BELARDI et al., 2014; HOPFE et al., 2017). Determining the particle size is important, as a large surface area in the phosphorus powder is critical to the success of analytical procedures for leaching the REE (HOPFE et al., 2017).

Studies have shown that 80% of the material is characterized by a particle size below 25 µm (BELARDI et al., 2014; TUNSU et al., 2016). This fraction contains the majority of phosphorus powder and a high content of fine glass particles. Larger class fractions contain glass fragments and other impurities, such as plastics and electrode parts, which are generated during the breaking of the lamp into small pieces (TUNSU et al., 2016). According to Belardi et al. (2014), REE recovery procedures must be applied in classes below 7 µm, as the size distribution of all REE particles is smaller than this value. A laser diffractometer was used for particle size analysis and the results indicated that the particles are evenly scattered in the fractions of approximately 0.7 µm and 4 – 5 µm (HOPFE et al., 2017). The presence of REEs in the fine fraction is a limitation for recovery methods that involve physical separation processes. In this case, methods involving chemical processes are more suitable (BELARDI et al., 2014). Previous particle size determination supports REE chemical leaching studies.

3.2 | Elementary Chemical Composition

The X-ray fluorescence (XRF) technique has been commonly used to determine the elementary chemical composition of the fluorescent lamp matrix (URNIEZAITE et al., 2010; DE MICHELIS et al., 2011; HOPFE et al., 2017, GIJSEMANS et al., 2018; INNOCENZI et al., 2018; YIN et al., 2018; DE FARIAS et al., 2020). This technique for analyzing solid samples is frequently used in different matrices due to its practicality in preparing the samples, the possibility of determining several elements at the same time and because it is a technique with a lower cost per analysis. However, XRF has lower precision and accuracy, when compared to atomic spectrometry techniques, and low sensitivity for some elements (MARGUÍ; QUERALT; HIDALGO, 2009), making it inadequate for the determination of minority elements, especially for some rare earth elements present in the residue of fluorescent lamps. Furthermore, the usual calibration procedure for XRF instrumentation depends on the availability of certified reference materials and not on analytical curves used in quantitative instrumental techniques (POTTS; WEBB, 1992; SIEBER, 2002).

Recently, our research group carried out the semi-quantitative characterization of the elements present in the lamp dust residue, provided by a Brazilian recycling company, using the X-ray fluorescence (XRF) technique. The analysis was performed after a thermal treatment (350°C for 24h) of the residue to remove mercury. For the analysis, 2.0 g of lamp powder and 0.4 g of boric acid, as a binder, were used, mixed and pressed to form a tablet. The results showed that the major elements were Ca (200,590 ppm), P (115,942 ppm), Si (114,374 ppm), Na (47,732 ppm) and Al (29,462 ppm). The elements of the rare earth group identified in this residue were Y (17,639 ppm), La (1279 ppm), Tb (937 ppm) and Ce (868 ppm) (DE FARIAS et al., 2020).

The XRF technique has also been used for the chemical characterization of each fraction obtained in the particle size classification. The results obtained by Michaelis et al. (2011)

showed that silicon is the main element and that higher concentrations of calcium and yttrium can be found in the fraction below 40 μm.

Scanning electron microscopy with energy dispersion detector (SEM-EDS) is a semi-quantitative technique for elemental analysis. This technique has been used to estimate the content of elements present in fluorescent lamp matrices (SHIMIZU et al., 2005; BELARDI, at al., 2014; TUNSU, et al., 2014). SEM-EDS have also been used to assess the appearance (aspect) of the material, to obtain data on the elements present and to determine the occurrence and distribution of components in the matrix (TUNSU, et al., 2014). However, the data obtained by the EDS technique are not as accurate as those obtained in chemical analyses that use spectrometric analytical techniques for quantification.

Results from the EDS analysis on phosphorus powder showed that oxygen, yttrium and aluminum are the most abundant elements. In relation to rare earth elements, yttrium stands out in the highest content, followed by La, Ce, Tb and Eu. Rare earth elements contribute about 8% of the total mass of phosphorus powder (LEITE et al, 2020), and among the different components present in phosphorus, red phosphorus is considered the most abundant (ANAND et al, 2019). The typical content of red phosphorus (Y_2O_3:Eu^{3+}) is around 55% of the triphosphorous mixture used in fluorescent lamps (TUNSU, et al., 2016).

SEM-EDS analysis also revealed the concomitant presence of yttrium and europium in distinct particles. In some cases, europium has also been found to be independent of yttrium. The elements lanthanum, cerium and terbium were found together in the particles (TUNSU et al., 2014). Shimizu et al.

(2005) also used the SEM-EDS technique to obtain data on the REE present in fluorescent lamp residues. Particle size ranged from 5 to 10 µm. Lanthanum and cerium were found concomitantly in the same particle and yttrium was found alone in different particles.

3.3 | Identification of Crystalline Phases and Morphology

X-ray diffraction (XRD) has been used to determine the mineral phases of the sample and the structure in which REEs may be present in fluorescent lamps (SHIMIZU et al., 2005; TUNSU et al., 2014; SHUKLA et al., 2021). Results indicate that the matrix is mainly composed of fluor-apatite ($Ca_5(PO_4)_3F$) and hydroxyapatite ($Ca_5(PO_4)_3(OH)$) as the predominant crystalline phase. The presence of strontium phosphate ($Sr_5(PO_4)_3$) and potassium pyrophosphate tetra-hydrate ($K_4P_2O_7.4H_2O$) in the phosphorus powder residue was also observed (COSKUN; CIVELEKOGLU, 2014). Regarding REE, XRD analyses confirmed the presence of yttrium and europium as oxides. Terbium oxide and gadolinium oxide were also identified (TUNSU et al., 2014). Results from XRD also revealed the presence of wakefieldite (YVO_4) and monazite (La, Ce, Tb, Gd)PO_4. The glass present in the residues is not detectable by X-ray diffraction due to its amorphous state (IPPOLITO et al., 2017a).

Recently, our research group used the XRD technique to identify the crystalline phases present in a fluorescent lamp residue given away by a Brazilian recycler. The X-ray diffractogram, obtained by the powder method, was generated in a Bruker-D4 Endeavor equipment, under the following operating conditions: Co Kα radiation (40 kV/40 mA), goniometer speed of 0.02° 2θ per step, with a counting time of 0.5 s per step and collected from 4 to 80° 2θ, with a position-sensitive detector (LynxEye). The results confirmed the presence of fluorapatite, indicated the presence of collusite, aluminium oxide, barium sulphate and calcium carbonate. It was not possible to identify the structure of the REEs in this sample, probably due to their low content or because they are present in the amorphous phase. Figure 2 shows the diffractogram obtained for the FB residue sample.

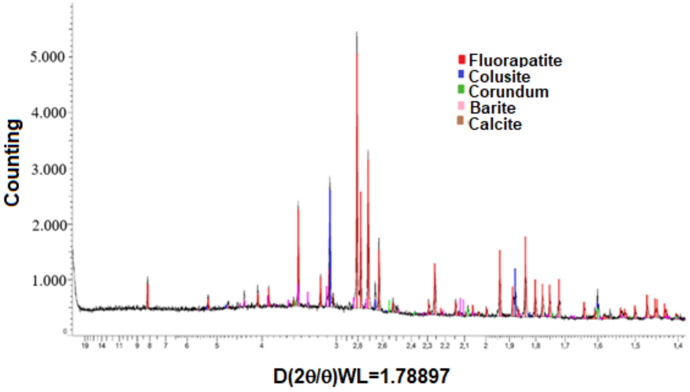

Figure 2. Fluorescent lamps residue sample diffractogram.

The elements Y, Eu, La and Ce are the main REE identified in luminescent materials from waste fluorescent lamps. Yttrium and Eu are present as oxides and La and Ce are present as phosphates. It was not possible to identify the structure of Tb due to the low content of this element (SHIMIZU et al., 2005). In the work developed by Shukla et al. (2021), the semi-quantitative XRD analysis of phosphorus powder indicated the occurrence of the components, $Y_2O_3:Eu^{3+}$ (about 47%), $CeMgAl_{11}O_{19}:Tb^{3+}$ (about 11%), $LaPO_4:Ce^{3+}Tb^{3+}$ (22%), and $BaMgAl_{10}O_{17}:Eu^{2+}$ (18%). The components SiO_2 and Al_2O_3 were also found in smaller amounts in the phases. In addition to XRD, the X-ray excited photoelectron spectroscopy (XPS) technique was used to investigate the valence state of the elements present in the phosphorus powder.

3.4 | Thermal Analysis

Techniques based on thermal analysis can also be used to complement the chemical and technological characterization studies of fluorescent lamp matrices (BELARDI et al., 2014.) These techniques assess changes in the properties of a sample when submitted to controlled temperature variation. Thermogravimetric analysis (TGA) consists of measuring the loss or gain in mass of a sample as a function of temperature and/or time in a controlled environment and with temperature programming. The technique allows simulating thermal degradation under different conditions, such as pyrolysis and combustion in different matrices. Different physical and chemical properties of the compounds can be evaluated (DÍAZ et al., 2021).

The behaviour of fractions obtained in the particle size classification, from results obtained with the TGA curves, was evaluated and the main results indicated that almost all fractions, and to a lesser extent the fraction mainly constituted by SiO_2, present mass loss up to approximately 650° C. Carbonate decomposition (fractions between 0.150 and 0.038 mm) starts at a temperature of 650°C and ends around 730°C. The presence of calcite was also confirmed (BELARDI et al., 2014). In the study of the hydrometallurgical process for recovering rare earth elements from fractions of fluorescent lamp waste, thermogravimetric analysis was used as a tool to determine the ideal temperature needed to convert the oxalate precipitate from REEs into oxide (TUNSU et al., 2016).

3.5 | Chemical Analysis by Spectroscopic Techniques

Chemical analysis by spectroscopic techniques is a widely used quantitative analytical procedure for the characterization of fluorescent lamp residues (SHIMIZU et al., 2005; TUNSU et al., 2014; TUNSU et al., 2016; GIJSEMANS et al., 2018; ANAND et al., 2019; PATELI et al., 2020). This procedure is important to determine the total concentration of analytes, especially the minority elements present in this matrix. Generally speaking, chemical analysis can be divided into two steps. In the first one, the matrix is subjected to a specific treatment to make the analytes available and the second one involves the determination of the total concentration of the species using an instrumental analytical technique. Dissolving solid samples is one of the most important steps in sample preparation (GÜNGOR; ELIK, 2007). Successful decomposition of a sample depends on employing a simple, less labour-intensive analytical procedure using small amounts of reagents to promote matrix dissolution and provide reliable results. The selection of a decomposition procedure should consider important characteristics such as the nature of the matrix and analyte/species to be quantified, the concentration of the species of interest and possible interferers, the available quantification technique, and the required precision and accuracy. When possible, leaching of species of interest is encouraged, as the resulting solution is less complex, minimizing matrix interferences. However, in some cases the complete dissolution of sample is indicated (PRIEGO-CAPOTE; LUQUE DE CASTRO, 2007).

In works developed with fluorescent lamp residues, generally, the first step of the chemical analysis consists of the decomposition/digestion of the matrix (dry or wet) to promote the dissolution of the material and the second step includes the quantification of the species of interest using analytical spectroscopic techniques. For the chemical characterization of this material, the method of dry decomposition from matrix fusion, with subsequent dissolution in acid medium and quantification of the elements using the technique of optical emission spectrometry with inductively coupled plasma (ICP OES) or spectrometry Inductively Coupled Plasma Mass spectrometry (ICP-MS) has been proposed in the literature (GOYNE et al., 2010; BELARDI at al., 2014; LIU et al., 2014; GIJSEMANS et al., 2018; ANAND et al., 2019).

The fluxes most used for dry decomposition of fluorescent lamp residues are meta and lithium tetra-borate, a mixture of sodium carbonate and boric acid and sodium hydroxide. The decomposition time can vary from 4.5 min (in an induction oven) to 60 min and the melting temperature from 850°C to 1200°C. The molten mixture can be solubilized in dilute nitric or hydrochloric acid solutions or adding concentrated hydrochloric acid. Then, the resulting solution can be analysed by different spectroscopic techniques, such as aforementioned (BELARDI et al., 2014; LIU et al., 2014; GIJSEMANS et. al., 2018; ANAND et al., 2019).

Beraldi et al. (2014) performed the digestion of a fluorescent lamp residue by fusion with meta/lithium tetra-borate in an induction oven followed by solubilisation in dilute nitric acid. To ensure successful melting, the residues were first dried at 60°C overnight and ground manually to obtain a particle size below 0.053 mm. The results of the chemical characterization indicated the presence of the elements Y (49260 mg kg^{-1}), La (4583 mg kg^{-1}), Eu (3957 mg kg^{-1}), Ce (3383 mg kg^{-1}) and Tb (1320 mg kg^{-1}) at higher concentrations, followed by the presence of Gd (114 mg kg^{-1}), Lu (8.8 mg kg^{-1}) and Sc (3.0 mg kg^{-1}). Nd, Yb, Pm and Pr were found in concentrations in the order of 1 mg kg^{-1} and Sm, Er and Ho in concentrations ranging from 0.3 to 0.1 mg kg^{-1}. Dry decomposition methods present as disadvantages possible sample contamination by impurities present in the flux that can interfere with the analysis accuracy, the loss of elements by volatilization (SANDRONI; SMITH, 2002) and the sample dilution.

Wet decomposition methods have also been used for the determination of REE in fluorescent lamp waste. The lamp residue can be digested in closed flasks with aqua-regia solution (volume ratio of 1 HNO_3:4 HCl) under heating at around 90°C (INNOCENZI et al., 2016, IPPOLITO et al., 2017a; IPPOLITO et al., 2017b, INNOCENZI et al., 2018). Decomposition time can vary from 2 to 3 h (TUNSU et. al, 2014; TUNSU et al., 2016). After the wet matrix decomposition, the quantification of the analytes can be performed in the solution obtained using ICP OES techniques (IPPOLITO et al., 2017b, INNOCENZI et al., 2018), ICP-MS (TUNSU et. al, 2016, IPPOLITO et al., 2017a) or atomic absorption spectrometry (AAS) (DE MICHELIS et al., 2011). The undissolved glass residue, from the procedure involving the digestion of fluorescent lamp residue with aqua-regia, was analysed using the SEM/EDS technique and the results indicated a good dissolution of the phosphorus particles (TUNSU et. al., 2014). Concentrated sulphuric acid was also employed for the wet decomposition of the fluorescent powder. After dissolving the material, the REE were quantified using the ICP OES as a quantification technique (SHIMIZU et al., 2005).

Tunsu et al. (2016) used the wet procedure based on aqua-regia digestion to determine the concentration of metals in fluorescent lamp residues. Three different fractions of fluorescent lamp residues were studied and the results of the chemical analysis showed that Ca (93 - 135 g kg^{-1}), Al (7.5 - 9.3 g kg^{-1}), Ba (4.5 - 7.1 g kg^{-1}) and Fe (4.5 - 7.2 g kg^{-1}) are present in higher concentrations. As evidenced in previous results in the literature, yttrium is the predominant REE in all samples, with a content ranging from 162 to 175 g kg^{-1} and Eu is the second most relevant REE in the residue with an approximate content of 11 g kg^{-1}.

Microwave radiation has also been used to promote acid digestion of waste from fluorescent lamps. The microwave-assisted digestion procedure consisted of mixing approximately 50 mg of the material with 10 ml of hydrochloric acid and a total digestion time of one hour and 10 minutes. The warm-up program was carried out in 4 stages, varying the dwell time and the heating ramp. The solutions obtained after the digestion process were analyzed using the ICP OES technique (PATELI et al., 2020). The digestion method proposed by Pateli et al. (2020) is about 2.6 times faster compared to methods that use the heating plate.

Methods that use melting and heating plate have some disadvantages, such as a large amount of reagents used and longer digestion times. Furthermore, the analytical procedures are laborious and present a risk of sample contamination (SANDRONI; SMITH, 2002). Due to these factors, techniques for the direct introduction of solid samples have been increasingly investigated (DURRANT, 1999). Techniques for direct introduction of solid samples, such as laser ablation, are also being studied, with the objective of reducing the number of analysis steps and, consequently, reducing the time and probability of contamination.

Laser ablation (Laser Ablation, LA) has been studied as a technique for introducing solid samples, with several applications for both LA-ICP-MS (hyphenated laser ablation for inductively coupled plasma mass spectrometry) and for the LA-ICP OES (laser ablation hyphenated to optical emission spectrometry with inductively coupled plasma), in geology, mineralogy, biological and environmental research, materials science, among many other areas (LEITE et al., 2011).

The LA-ICP-MS is a multi-element analytical technique used for direct determination of traces and isotopic analysis in solid samples (PICKHARDT et al., 2000) due to its high spatial resolution and low detection limits, in addition to having the potential to quantify more than 70 elements simultaneously (GUILLONG et al., 2011). However, in practice, there are limitations regarding the availability of calibration standards with the same matrix composition as the sample of interest or regarding the correction of spectral interference (LEITE et al., 2011), which impairs quantification. This technique can be promising to corroborate the results of chemical and technological characterization of fluorescent lamp matrices.

4 | REUSE AND RECYCLING OF USED FLUORESCENT LAMP COMPONENTS

Processes for reusing fluorescent lamps at the end of their useful life occur through the application of physical processes to separate the components. Recycling to obtain new products includes chemical and biological processes. After disassembly, the phosphor powder and other parts of the lamp can be reused, or the phosphorus powder can be recycled to obtain a product rich in REE.

Phosphorus powders can be reused to manufacture other lamps; however, there are some limitations to this form of reuse. Processed lamps would have to be selected and grouped by types and manufacturers, as there are variations in their compositions, which may have combinations of types of phosphorus (BINNEMANS et al., 2013; BINNEMANS; JONES, 2014).

Another important point is that the process of recycling fluorescent lamps, at the end of their useful life, depends on the type of lamp. For linear tube lamps, the aluminium end caps are cut off; the phosphorus powder is expelled by a pressurized air stream and collected. Mercury is recovered by passing the gas through activated carbon. Metallic lids, as well as other non-metallic materials, can be reused after the mercury decontamination process (BINNEMANS; JONES, 2014; WU et al., 2014; LEE et al., 2015). By this method, about 99% of a lamp can be used (WU et al., 2014).

Non-linear tube fluorescent lamps undergo a crushing process, which can be dry or wet, followed by physical separation of the fraction that contains the phosphorus powder (BINNEMANS et al., 2013; BINNEMANS; JONES, 2014; WU et al., 2014). In this step it is possible to separate the phosphorus powder from the thicker fraction of the glass. In wet ways, the solvents used to prevent the diffusion of mercury vapor are ethanol and acetone (WU et al., 2014). The dry process is carried out in a closed environment and with controlled pressure to collect the gaseous fraction that contains mercury. In this process, the recovered phosphorus powder will be mixed with a very fine glass powder (BINNEMANS; JONES, 2014; WU et al., 2014). Importantly, the recovered phosphorus powder will also contain the alumina from the coating.

Another inconvenience in reusing fresh phosphorus is the fact that this material can deteriorate during the lifetime of the lamp, due to exposure to high-energy ultraviolet radiation and the impact suffered by ions and molecules present in the gas phase (BINNEMANS et al., 2013; BINNEMANS; JONES, 2014; WU et al., 2014). Furthermore, most mercury accumulates in the powder over time of use (JANG et al., 2005; REY-RAAP; GALLARDO, 2012; WU et al., 2014). Thus, the reuse of phosphorus powder becomes very limited and the process for recovering powder with high purity has a high cost (IPPOLITO et al., 2021). However, this material becomes attractive as a secondary source for REE recovery.

The amount of mercury present in the solid phase of fluorescent lamp residues (glass, terminals and phosphorus powder) varies due to different types of lamps, different operating conditions, lifetime of lamps and operating conditions of the recycling plant. Rey-Raap and Gallardo (2012) found mean mercury content equal to 204.2 ± 8.9 ng g^{-1} in spent compact fluorescent lamp powders. The mercury content values found by Lecler et al. (2018) for powder residues from 18 W linear tube lamps, obtained by the crushing operation, ranged from 6.50 to 114.9 μg g^{-1}. In their study they report that in a spent lamp about 33% of all mercury is present in the solid phase (8% in glasses, 13% in terminals and 13% in luminescent powder) while for new lamps this value is about 20 %. Lee et al. (2020) analysed a residue (containing glass, terminals and luminescent powder), obtained from a wet recycling plant, whose mass fraction of Hg was 485 mg kg^{-1} of residue. Our research group analysed a luminescent powder residue from a Brazilian recycler, before and after a heat treatment for mercury recovery, the Hg contents found were 230 ± 10 mg kg^{-1} and 40 ± 2 mg kg^{-1}, respectively.

Heat treatment is commonly used for the recovery and/or elimination of mercury from the residues of lamp phosphors (LEE et al., 2015; LEE et al., 2020; LECLER et al., 2018). The boiling point of mercury is 356°C under normal pressure conditions. The luminescent powder residues are treated by heating, at a temperature ranging between 400 and 600°C, under vacuum (BINNEMANS et al., 2013; BINNEMANS.; JONES, 2014; WU et al., 2014; LEE et al., 2020). Mercury can be recovered as an element by vapor condensation or retained in adsorbent solid phases, such as activated carbon or iron oxide nanoparticles, among others (BINNEMANS et al., 2013; BINNEMANS.; JONES, 2014; LEE et al., 2015; HU et al., 2016; CHEN et al., 2020). The removal of mercury by thermal desorption can achieve yields higher than 70% (LECLER et al., 2018; LEE et al., 2015; HU et al., 2016; CHEN et al., 2020). Temperatures from 800°C (CHANG et al., 2009; 2010) are necessary to volatilize all of the mercury, because during the discharge process, elemental mercury is oxidized to Hg^{2+} ions, which bind more strongly to the dust phosphorus and glass (CHANG et al., 2009; DANG et al., 2002; BINNEMANS et al., 2013; BINNEMANS.; JONES, 2014).

Such high temperatures require high energy expenditure, and the removal of mercury by wet processes may be an option. Over time, some of these processes were studied: leaching with sodium hypochlorite solutions, removing more than 99% of Hg from the residue (SOBRAL et al., 2004); removal of Hg from aqueous solution by photocatalytic reduction with yields higher than 99% (BUSSI et al., 2010); removal of Hg using iodine and potassium iodide solutions with 97% yield, followed by metal recovery with organic extractants (TUNSU et al., 2015); methods that use plant residues as low-cost adsorbents, with yields higher than 99% (AL-GHOUTI et al., 2019). Chemically, leaching with mineral acids (such as nitric and hydrochloric acids) is also an alternative (TUNSU et al., 2014); however, depending on the experimental conditions, they can promote the solubilisation of a fraction of the lanthanides, which would compromise a possible recycling of the luminescent powder.

The other parts that consist of metallic materials and glass can also be reused through a decontamination process, as in the case of phosphorus powders. Glass is a 100% recyclable material; however, there are difficulties in the complete removal of phosphorus dust adhered to the surface and mercury. For glass decontamination, among the processes already mentioned above, many authors cite thermal desorption and acid leaching (JANG et al., 2005; REY-RAAP; GALLARDO, 2013; DRZYMALA et al., 2020). Due to the difficulty of completely removing contaminants from the glass matrix, co-processing becomes an environmentally correct alternative for final disposal, with the incorporation of this residue in other industrial segments, such as civil construction: in the manufacture of geo-polymers and ceramic materials (NOVAIS et al., 2016; ROSSON et al., 2021); in the cement industry (DRZYMALA et al., 2020; PITARCH et al., 2021); in paving (GEDIK et al., 2021).

Figure 3 shows the flowchart of a simplified process for reusing and recycling fluorescent lamp components. The blocks highlighted in orange represent the process of dismantling and separating the components (phosphorus powder, glass and plastic and metallic constituents) and the blocks in blue show the operations of crushing and separating by particle size.

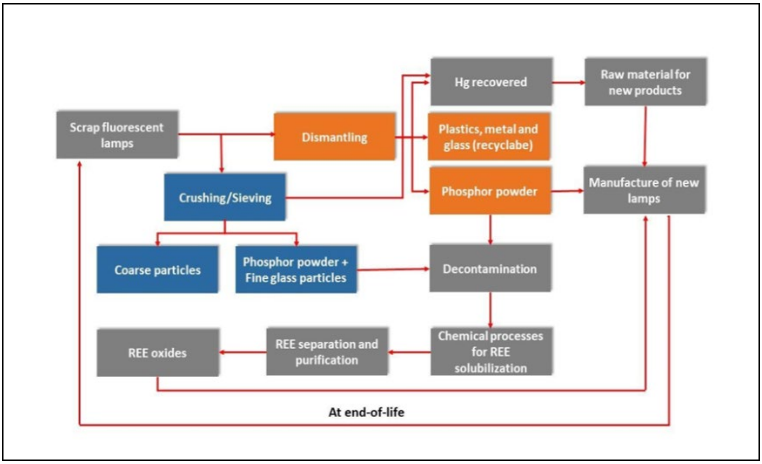

Figure 3. Simplified flowchart of reuse and recycling of fluorescent lamps.

4.1 | Chemical Processing of Phosphorus Powder to Recover Rare Earth Elements

The most traditional methodologies employed for recycling and recovering REE in decontaminated powder mostly involve hydrometallurgical processes, where the processing steps are similar to those used in the extraction of rare earths from minerals (JHA et al., 2016; ALVES et al., 2021). The hydrometallurgical processes employ wet chemical decomposition methods using mineral acids (such as sulphuric, hydrochloric or nitric acids) or even alkaline solutions (such as NaOH or Na_2CO_3) to dissolve the materials. Other methods involve dry decomposition by melting with suitable inorganic fluxes (such as NaOH or Na_2CO_3). After solubilisation, recovery of REE from the aqueous phase can be carried out by precipitation, fractional crystallization and extraction with organic solvents.

4.1.1 | Conventional methods

Numerous studies described in the literature show the efficiency of leaching with inorganic acids in hydrometallurgical processes. The most traditional methods employ the aforementioned sulphuric, hydrochloric or nitric acids with varying parameters such as acid concentration, solid/liquid ratio, temperature and reaction time. The solubilisation of REE will also depend on how the element is found in this matrix, as lamps can contain different types of phosphorus. In red phosphorus, yttrium and europium are present in the form of oxides and are easily solubilized in acid medium under moderate conditions, as generically represented in Reaction 1, where RE represents Y and the lanthanides:

$$RE_2O_3 + 6H^+ \rightleftarrows 2RE^{3+} + 3H_2O \qquad (1)$$

Yttrium and europium are predominant in the mixtures, with an emphasis on yttrium (TUNSU et al., 2016; LEE et al., 2017). Several authors have developed methods for leaching these metals with diluted solutions (0.5 – 4.0 mol L^{-1}) of HCl, HNO_3 and H_2SO_4, testing different solid/liquid ratios. Temperatures generally vary between 20 and 90°C, with a reaction time that can take from a few minutes to 24 hours, depending on the combination of all experimental conditions. Under optimized experimental conditions, recoveries of yttrium and europium are high, reaching yields between 80 and 100% (DE MICHELIS et al., 2011; TUNSU et al., 2014; LEE et al., 2017; VU et al., 2017; EDUAFO; MISHRA, 2018; WU et al., 2019; HUYNH; HA; VU, 2022; SUMAN; RAJAKI; ANSARI, 2023).

Calcium (as well as barium and strontium) from halo-phosphates increases acid consumption during leaching, and, in addition, with its solubilisation, the amount of metallic cations in the solution increases, which can be inconvenient for the final separation step and purification of ETR. The use of sulphuric acid as a leaching agent has the advantage of decreasing the solubility of alkaline earth metals, due to the formation of poorly soluble sulphates. According to Tunsu et al. (2016) calcium reacts more quickly with acid than REE, and based on this difference it is possible to selectively solubilize it with a pre-treatment with dilute acids and reduced contact time. In their work, Tunsu et al. (2016) obtained a calcium removal efficiency of up to 90% by treating the phosphorus powder with nitric acid 1 mol L^{-1} for 10 to 15 min, with losses of yttrium and europium below 3%.

Terbium and europium are the most economically valued REE (IPPOLITO et al., 2021). Terbium, as well as cerium, lanthanum and gadolinium are present in green phosphorus, in the form of phosphates and spinel, which are chemically more refractory (ANAND; SINGH, 2021; IPPOLITO et al., 2021). As with blue phosphorus, the solubilisation of green phosphorus is favoured under more drastic operating conditions: higher acid concentrations, higher temperatures, ultrasound (TUNSU et al. 2014) and microwave decompositions (TANVAR et al. 2020; SHUKLA; DHAWAN, 2020; SHUKLA et al. 2021; YANG et al., 2023). SHUKLA et al. (2021) considered the decomposition of a phosphorus powder sample by baking with microwave-assisted concentrated sulphuric acid. Terbium recovery reached about 94%.

Alternatively, a pre-treatment can be applied to convert these compounds into species that can be more easily solubilised in an acidic medium. Alkaline fusion is able to convert the most refractory compounds into REE oxides, which can later be submitted to acid leaching, as represented by Reaction 1. The most used alkalis are NaOH or KOH, $Ba(OH)_2$ and Na_2CO_3. Reactions 2 to 6 show simplified representations for some conversions of different compounds present in green and blue phosphorus (INNOCENZI et al., 2016; IPPOLITO et al., 2017b; YURRAMENDI et al., 2019; TUNSU et al., 2016), where TR represents Y and the lanthanides:

$$2REPO_4 + 6KOH \rightarrow RE_2O_3 + 2K_3PO_4 + 3H_2O \qquad (2)$$

$$2REPO_4 + 3Ba(OH)_2 \rightarrow RE_2O_3 + Ba_3(PO_4)_2 + 3H_2O \qquad (3)$$

$$2REPO_4 + 3Na_2CO_3 \rightarrow RE_2O_3 + 2Na_3PO_4 + 3CO_2 \qquad (4)$$

$$Ce_{0.67}Tb_{0.33}MgAl_{11}O_{19} + NaOH + O_2 \rightarrow NaAlO_2 + MgO + CeO_2 + Tb_4O_7 + H_2O \qquad (5)$$

$$BaMgAl_{10}O_{17}{:}Eu^{2+} + NaOH + O_2 + CO_2 \rightarrow NaAlO_2 + BaCO_3 + MgO + Eu_2O_3 + +H_2O \qquad (6)$$

Temperatures are high, varying between 600 and 950°C, depending on the alkali melting point, with time varying between 1 and 3 h. Another important parameter to be considered is the mass ratio between phosphorus powder and alkali, which in many cases can be less than or equal to 1. After acid leaching, it is possible to recover terbium, as with other REE, with high yields, ranging from 60 and 100% (INNOCENZI et al., 2016; IPPOLITO et al., 2017b; LIANG et al., 2016; LIU et al., 2019; YURRAMENDI et al., 2019; XIE et al., 2023).

The processes described so far are the most widely cited in the literature. Several studies are periodically published based on more traditional methods. Recent studies propose combinations of the treatments mentioned above with the replacement of conventional heating by the use of microwave radiation, in a reduced time, for converting refractory compounds into oxides (TANVAR et al., 2020; SHUKLA et al., 2020; SHUKLA; DHAWAN 2020; LIU et al. 2022).

One option to heat pre-treatment is mechanical activation. Mechanical activation produces a change in the physicochemical properties of the matrix due to the effects caused by the application of impact, compression and shear forces. The defects caused in the crystal lattice and the increase in the surface area favour leaching, eliminating the need for heat treatment and the use of reagents, thus providing greater economic and environmental attractiveness to the method (TAN et al., 2017; VAN LOY et al., 2018; IPPOLITO et al., 2021). Activation is usually performed in a ball mill and some of the studied parameters are rotation speed, activation time, ball size and ratio between the number of balls and the luminescent powder mass (HASEGAWA et al., 2018; HE et al., 2018; VAN LOY et al., 2018). Milling can be performed dry, followed by acid leaching, significantly increasing leaching when compared to untreated phosphorus powder (SONG et al., 2017; TAN et al., 2017; VAN LOY et al., 2018; IPPOLITO et al., 2021). Mechanical activation and leaching of metals can also be combined in a wet grinding, with solutions of the aforementioned acids (VAN LOY et al., 2018) or even with chelating agents, such as ethylene-diamine-tetra-acetic acid (HASEGAWA et al., 2018).

4.1.2 | Environmentally friendly methods

With the advancement of green chemistry (ARMENTA et al., 2008; GALUSZKA et al., 2013) there is a need for a more conscious use of chemical reagents and the development of more environmentally friendly methods. The indiscriminate use of these reagents and the inappropriate disposal of final effluents can cause adverse environmental impacts. In these processes there is a high consumption of water resources, increased corrosion of equipment in industrial plants and the large generation of toxic liquid and gaseous effluents, such as Cl_2, SO_x and NO_x.

Organic acids have been used in the recycling of waste electrical and electronic equipment as an alternative to the use of mineral acids (ZEBA et al., 2022; GISMONTI et al., 2021; BANERJEE et al., 2022). Some of these acids are recognized as eco-friendly lixiviants, as they are a low environmental impact option in hydrometallurgical processes due to characteristics such as easy natural degradation and a reduction in the release of toxic gases during the process (BANERJEE et al., 2022, BINNEMANS; JONES, 2023).

Solvo-metallurgical processes using organosulphonic acids have been developed, such as those studied by Gijsemans et al. (2018) and Rodriguez et al. (2021). It was possible to quantitatively solubilize green phosphorus REEs (LAP) with pure methane-sulphonic acid at temperatures close to 200°C.

Goyne et al. (2010) studied the effects of low molecular weight organic acids such as citric, phthalic, oxalic and salicylic acid on the release of REE from phosphate minerals such as apatite and xenotime. In these cases, the leaching of these metals is influenced by the chemical balance of complexation of the metals with the anions of the respective organic acids, the greater the complexation constant, the greater the leaching efficiency. Organic acids were classified according to leaching efficiency accordingly to the complexation capacity of each ligand: citrate > oxalate > phthalate > salicylate ≈ non-binding anions.

The stability of the complex formed between the REE and the binding anion of the organic acid has a direct impact on the leaching yield, as well as on the other steps of REE recovery from the aqueous solution. For those cases, in which unstable complexes are formed, less favourable experimental conditions (*i.e.*, higher temperatures or reaction times) may be necessary to achieve satisfactory yields, as observed by Tunsu et al. (2014). The leaching with acetic acid 25% v/v at 20°C lasted 168 h, to reach the solubilisation of 50 and 75% of Eu and Y, respectively, being little significant for the other REE (between 2 and 10%).

The efficiency of REE and other metals leaching by the action of low molecular weight organic acids corroborates bioleaching studies, which are based on the dissolution process of REE carriers by the action of a specific group of microorganisms. Studies report the use of heterotrophic microorganisms that produce these acids (such as acetic, citric, oxalic and gluconic acid) in the bioleaching of waste electrical and electronic equipment (ZHUANG et al., 2015; HOPFE et al., 2017; PRYIA; HAIT, 2017; HOPFE et al., 2018). Bioleaching is an environmentally friendly alternative when compared to conventional methods; however, the optimization and strict control of biotic and abiotic factors such as microbial species, pH, temperature, nutrients and system aeration rate can increase process costs (PRYIA;

HAIT, 2017). A disadvantage is the fact that bioleaching is a longer process compared to other methods (HOPFE et al., 2017; PRYIA; HAIT, 2017). Furthermore, the yield is relatively lower in metal recovery. Reed et al. (2016) obtained an overall REE recovery of 2% in phosphorus powder leaching employing a *Gluconobacter oxydans* culture. In their studies HOPFE et al. (2017, 2018) achieved variable yields between 5.0 and 12.6% with different cultures (*tea fungus Kombucha, Lactobacillus casei and Komagataeibacter xylinus*). The authors observed a selectivity trend for Y and Eu in all cases, as observed by Castro et al. (2023) a yield of 13.43% and 11.99% for Y and Eu, with bioleaching using *Aspergillus niger* culture.

4.2 | Separation and Purification Methods

The step of separating REE from aqueous solutions after leaching is essential to obtain products with high purity. The main methods are based on selective precipitation and liquid-liquid extraction.

4.2.1 | Precipitation separation

Precipitation separations depend on the difference in solubility between the species of interest and the others. REEs form poorly soluble compounds with various inorganic ions such as hydroxides, oxalates, sulphates, carbonates, phosphates, fluorides and sulphides. Solubility is influenced by parameters such as pH, temperature, concentration, ionic strength and the presence of other species in solution. The efficiency and selectivity of precipitation separation depend on strict control of these parameters.

Han (2020) studied the physicochemical principles of REE precipitation with some precipitating agents: oxalate, carbonate, sulphate, fluoride and phosphate. The study corroborated the strong influence of pH. The role of ions from mineral acids (*i.e.*, nitrate, sulphate and chloride) used in the leaching step was also evaluated, with nitrate offering the most favourable environment for precipitation (followed by sulphate and chloride), quite possibly due to its weak complexing capacity.

Depending on the composition of the solutions after leaching, separation and purification strategies can combine precipitation with liquid-liquid extraction. Precipitation can be applied to remove contaminating species, such as alkaline earth metals, for example, followed by liquid-liquid extraction of REEs, or even, REEs can be precipitated, with subsequent solubilisation and liquid-liquid extraction, amplifying the purification of the final product (ANAND; SINGH, 2021).

In precipitation separations, oxalate precipitation is the main alternative. The selective precipitation of REE oxalates must occur at acidic pH, thus avoiding the precipitation of other more soluble metallic oxalates, such as calcium oxalate, for example. Due to the physicochemical similarities between the elements of this group, after precipitation a mixture of REE oxalates is obtained (Reaction 7). Precipitation occurs at pH around 1 and 2, at room temperature or with heating (60-80°C), at varying times (20-60 min). After calcination (600-900°C) (Reaction 8) it is possible to obtain oxide mixtures with a purity higher than 98% (INNOCENZI et al., 2016; IPPOLITO et al. 2017b; LIU et al., 2019; SHUKLA et al., 2020; TANVAR et al., 2020; IPPOLITO et al., 2021).

$$2RE^{3+} + 3H_2C_2O_4 + nH_2O \rightleftarrows RE_2(C_2O_4)_3.nH_2O + 6H^+ \quad (7)$$

$$RE_2(C_2O_4)_3.nH_2O \rightarrow RE_2O_3 + 3CO + 3CO_2 + nH_2O_{(vapor)} \quad (8)$$

Ion exchange and extraction with organic solvents are alternatives for separation between REE and purification. However, ion exchange is no longer used on a large scale and is mainly restricted to the purification of small amounts and analytical applications (XIE et al., 2014).

4.2.2 | Separation by liquid-liquid extraction

The solvent extraction process is quite advantageous mainly because of its large-scale applicability. Being widely used in extractive metallurgy, it is capable of selectively extracting the species of interest in aqueous solution. The organo-compounds of the extracted metals are preferably soluble in the organic phase and can then be re-extracted to the aqueous phase with a suitable solution, usually an aqueous solution of a dilute acid. The pure REE oxides are obtained by precipitating their hydroxides or oxalates, as described above.

Solvent extraction takes place due to the ability of organic extracting molecules to complex with metals in the aqueous phase. The efficiency depends on several factors such as the extraction mechanism, the type of organic diluent, the concentration of the extracting agent, the nature of the leachate, the pH of the aqueous phase, the ratio between the organic phase and the aqueous phase (OP/AP) and the number of stages in which the extraction takes place. Due to the physicochemical similarities between these elements, the extraction process usually requires multiple successive steps to obtain products with high purity.

Organophosphate extractants are not the only ones, but they are the most widely used for extracting REE from aqueous solutions. Several studies in the literature report the efficiency of D2EHPA (di-2-ethylhexylphosphoric acid) in separating REE. D2EHPA is an extracting acid and its extraction mechanism is through the exchange of cations, as generically illustrated in Reaction 9, where REE^{3+} represents any cation of REE and HX an extracting acid, which generally forms dimers or larger aggregates in nonpolar organic solutions (XIE et al., 2014).

$$REE^{3+} + H_2X_2 \rightleftarrows REE(HX_2)_3 + 3H^+ \quad (9)$$

It is possible to selectively extract the REE with the strict adjustment of the experimental conditions, as described by INNOCENZI et al. (2018) who determined the separation factors between REE in sulphuric leachates obtained from fluorescent lamp powders, using D2EHPA diluted in kerosene. The best optimized experimental conditions were defined with D2EHPA 15% v/v, pH = 0.5, OP/AP = 1, and room temperature. In six stages it was possible to obtain selective extraction of yttrium with yields of 47% Y, 7% Tb, 4% Ce, without extracting other ETR and other metals.

The presence of certain ions in the aqueous solution will directly influence the extraction process, so the nature of the leaching agent must be taken into account, as observed by YURRAMENDI et al. (2019). The authors verified that the hydrochloric medium was the most suitable for the selective extraction of terbium against cerium and lanthanum with D2EHPA diluted in kerosene, when compared to the sulphuric medium. In a medium containing 6 mol L^{-1} HCl with D2EHPA 60% v/v, OP/AP = 1, in a single stage Y, Gd and Eu were extracted. Ce and La were extracted with high yield with pure D2EHPA, OP/AP = 1, in two stages. Similar results were found by Gijsemans et al. (2018) for separating Tb from Ce and La after a solvo-metallurgical process with methane-sulphonic acid.

Nonpolar organic solvents used as diluents are aliphatic or aromatic hydrocarbons. Aromatics reduce the effect of emulsification and gel formation (BATCHU; BINNEMANS, 2018). Aliphatic extenders promote higher extraction efficiency for all REEs; however, aromatics promote higher separation between light and heavy REEs, being more selective for the latter, as tested by BATCHU; BINNEMANS (2018) for acid extractants.

The use of other cation exchange extractants for REE extraction is reported in the literature, such as: Cyanex 272 (di-2,4,4-trimethylpentylphosphinic acid), Cyanex 572 (structure not reported in the literature) and PC88A (2-ethylhexylphosphonic acid mono-2-ethylhexyl ester) (INNOCENZI et al., 2018; GIJSEMANS et al., 2018; PAULINO et al., 2018).

Another class of organic extractants are neutral molecules, which also form stable complexes with REEs. TUNSU et al. (2016) report the use of Cyanex 923 (a commercial blend of trialkylphosphine oxides) for the recovery of Y and Eu. Yields close to 100% were achieved with Cyanex 923 35% v/v in kerosene, OP/AP = 2, in three stages of extraction from a solution with HNO_3 2 mol L^{-1}.

Extractants mixtures are also evaluated for their synergistic effect. The mixture can be done with extractants of the same class or even a mixture between different classes. More recent researches studied the synergistic effects and the extraction mechanism for mixtures between: Cyanex 302 (bis-2,4,4 trimethylpentyl monothiophosphinic acid) and D2EHPA in kerosene

(ALLAHKARAMI et al., 2021); HEHHAP [(heptylaminomethylphosphonic acid mono-2-ethyl-hexyl) ester] and Cyanex 272 in heptane (WEI et al., 2020).

Padhan and Sarangi (2021) evaluated the effect of acidic, basic (anionic exchangers) and neutral extractants and their mixtures, diluted in kerosene. The extractants used in the study were: D2EHPA, PC88A, Cyanex 272, Cyanex 301 (bis(2,4,4-trimethylpentyl) dithiophosphinic acid), Cyanex 302, Cyanex 921 (tri-octyl phosphine oxide), Cyanex 923, LIX 84I (2-hydroxy-5-nonylacetophenone oxime), LIX 622N (mixture of 5-dodecylsalicylaldoxime with tridecanol), Alamine 336 (tri-octyl/dodecyl amine) and Aliquat 336 (tricaprylmethylammonium chloride).

The disadvantage in using solvent extraction is the use of a large amount of organic solvents, and the fact that the process commonly needs to be carried out in several stages to obtain products with high purity, greatly increasing this consumption. In addition, environmental aspects must also be taken into account, due to the generation of toxic waste and the evaporation losses that release these compounds into the atmosphere. According to Vahidi and Zhao (2017), solvent extraction corresponds, on average, to 30% of the environmental impacts associated with the production of rare earths. The search for alternative and more environmentally friendly methods that can replace traditional methods has been growing over the years.

Ionic liquids appear as an alternative to replacing conventional organic extractants. They are composed of organic salts with a low melting point (< 100°C), have a wide variety of chemical structures, but are essentially formed by a bulky organic cation (such as ammonium, phosphonium, imidazolium, pyridinium etc.)

in combination with an anion of smaller size, which can be inorganic (chloride, hexafluorophosphate, tetrafluoroborate etc.) or organic (acetate, triflate, bis(trifluoromethyl) sulphonyl imide etc.) (ARRACHART et al., 2021).

Ionic liquids belong to a class of solvents that have low flammability, volatility and toxicity and are, therefore, considered an environmentally friendly alternative. Furthermore, they have high chemical and thermal stability. The extraction mechanisms are based on ion exchange systems (cationic and anionic) and solvation (ARRACHART et al., 2021; PRUSTY et al., 2021).

Two-phase aqueous systems (TPAS) based on ionic liquids have been used for extracting REE from aqueous solutions. The aqueous system contains the ionic liquid, electrolytes (such as $NaNO_3$, Na_2SO_4 etc.) and the target species. After separation, the system consists of a phase enriched with ionic liquid and another rich in electrolytes. Parameters such as contact time, system pH, ionic liquid and electrolyte concentration influence the extraction efficiency. Target species can be re-extracted from the ionic liquid with dilute acid solutions. Studies under optimized conditions show yields that can reach ~100% in the application of TPAS for extracting REE from aqueous solutions (CHEN et al. 2018; SUN et al., 2018; LEITE et al., 2020).

5 | FINAL REMARKS

Although several countries, including Brazil, have adequate legislation for waste prevention and management, waste recycling, especially electronic waste, does not seem to be a high priority issue. In Brazil, there is a plethora of barriers that impede or hinder the development of reverse logistics. Fortunately, there are many successful initiatives in countries such as Japan, USA, Taiwan, Australia and the European Union.

Also related to environmental issues, REE can be considered contaminants of emerging concern (CECs) due to the large worldwide exploitation for use in the high-tech industry sector. Electronic waste stands out as one of the main anthropogenic sources of REEs. The determination of contaminants of emerging concern in environmental samples has become a critical and challenging problem in the development of analytical methods (PENA-PEREIRA et al., 2021). Recent studies show that REEs of anthropogenic origin are found in the environment, including aquatic systems, and can be bio-accumulated in the food chain (GWENZI et al, 2018; GU et al., 2020). Thus, the development of new technologies for the recovery/recycling of these elements from electronic waste, including the dust from fluorescent lamps, significantly contribute to the human health.

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CETEM – Centro de Tecnologia Mineral
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