Original Article

Chemical analysis of LED bulb components: strategies for efficient recycling

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Abstract

Although there are several types of LED lamps on the market, the bulb type is one of the most sold in Brazil. A LED lighting system comprises some components such as base, heat sink, driver, and electronic board that contains the LED packages and a reflector. Each of these, made up of specific materials, involves polymers, metals, and ceramics. With the growth in the use of LED lamps in various sectors (industrial, commercial, and residential) there are several movements towards recycling them. However, it is necessary to know the materials most used in the LED lamp system to create recycling strategies for all components. In this context, the current study aimed to chemically characterize most of the components of bulb-type LED lamps by scanning electron microscopy (SEM) coupled with energy dispersive spectroscopy (EDS), in addition to X-ray fluorescence (FRX) and micro-Raman spectroscopy. The presence of strategic metals such as Ce and Y (rare earths), Si, In and Ga used in semiconductor components could be detected, as well as Cu, whose demand has doubled the price of this metal in recent years. No less important, the presence of Na, Mg, Al, Fe, Ni and Zn was also determined. Recycling these metals, from LED lamps, can reintegrate them into their productive chains.

Keywords: LED; Lamps; Recycling; Chemical characterization

Análise química de componentes de Lâmpadas LED: estratégias para reciclagem eficiente

Resumo

Embora existam vários tipos de lâmpadas LED no mercado, a do tipo bulbo é uma das mais vendidas no Brasil. Um sistema de iluminação LED é composto por alguns componentes, tais como: base, dissipador de calor, *driver* e placa eletrônica que contém os pacotes de LED e um refletor. Cada um desses componentes, formados por materiais específicos, envolve polímeros, metais e cerâmicas. Com o crescimento do uso de lâmpadas desse tipo em diversos setores (industrial, comercial e residencial), existem vários movimentos no sentido de reciclá-las. No entanto, é necessário conhecer os materiais mais utilizados no sistema dessas lâmpadas para criar estratégias de reciclagem de todos os componentes. Nesse contexto, o presente estudo teve como objetivo caracterizar quimicamente a maioria dos componentes de lâmpadas LED, do tipo bulbo, por meio de microscopia eletrônica de varredura (MEV) acoplada à espectroscopia de energia dispersiva (EDS), além de fluorescência de raios X (FRX) e espectroscopia Raman. Pôde ser detectada a presença de metais estratégicos como Ce e Y (terras raras), Si, In e Ga utilizados em componentes semicondutores, assim como Cu cuja demanda dobrou o preço desse metal nos últimos anos. Não menos importante, também foi determinada a presença de Na, Mg, Al, Fe, Ni e Zn. A reciclagem desses metais, a partir das lâmpadas LED, podem reintegra-los em suas cadeias produtivas.

Palavras-chave: LED; Lâmpada, Reciclagem; Caracterização química.

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1 Introduction

According to the JRC Technical Report - Update on Status of LED-Lightning world market since 2018 - sales of LED lamps have exceeded that of fluorescent ones. The International Energy Agency reported in 2019 record sales of over 10 billion units of LED light sources (bulbs, tubes, and modules) as well as luminaires [1]. In 2007, the International Energy Agency published a document recommending policies to reduce CO_2 emissions, mainly in the building, energy-consuming equipment, lighting, transport, and industry sectors. With regard to lighting, it was recommended that governments withdraw incandescent light bulbs from the market as quickly as possible. Based on these recommendations, the Brazilian government published Interministerial Ordinance No. 1007 (December 31, 2010), which establishes a schedule for the implementation of energy performance indexes for incandescent lamps with the aim of gradually banning them from the Brazilian market [2,3].

According to data from the Brazilian Lighting Industry Association (Abilux) consumption of LED lamps jumped from 27 million in 2014 to 81 million in 2015 [4]. Taking as an example the consumption of lamps in Brazil in 2014, the ratio was as follows: 85 million halogen, 150 million incandescent, 250 million compact fluorescent, 100 million tubes, 11 million sodium and sodium vapor, and 20 million LED lamps [5]. So, it is possible to identify the great potential for replacing traditional light bulbs with LED lamps. A document published in Market Research.com [6] indicates a 10% annual increase in the LED lighting market in Brazil according to Lumière Electric Magazine. With the growing increase in the use of LED lamps, it is undeniable the need for appropriate management end of life LED lamps, mainly because these types of lamps contain rare earth metals and excessive contents of copper, iron, aluminum, nickel, gold, phosphorus, silver and zinc [7]. Rahman et al. [8] used Google Scholar and Scopus database to identify existing LED lamp searches topics from 2008 to 2016 and report that only two works on Google Scholar and three on Scopus were related to LED lamp recycling. In a search from the Web of Science Date Bank, choosing the period of 2016 to 2023 and the words LED, recycling and lamps many works related to the theme were found. However, most of them refer to rare earth recycling [9], metal recycling [10,11] plastic recycling [12], and economic feasibility [13] on LED devices. One of them is about an innovative method for the recycling of end-of-life LED bulbs by mechanical processing [14]. Because LED lamps are composed of three types of engineering materials (polymer, metals, and ceramics) there is a difficulty in the separation process of each of them offering challenges in developing an efficient recycling process. In 2021 a process route for preparing and separating the components of LED lamps was proposed through an International Application Published under the Patent Treaty (PCT) Number WO 2021/113944 A1 [15]. The method involves grinding the LED lamps in autogenous mills. The purpose of this work is to present the chemical characterization of bulb-type LED lamps performed during the development of a project in partnership between IPT and Tramppo supported by Empresa Brasileira de Pesquisa e Inovação (Embrapii) that resulted in the referred application. The technology developed from this partnership seeks to fill a gap that is the individual separation of each of the types of materials present in the lamp (metals, ceramics and polymers) sending them to their appropriate recycling processes.

2 Materials and methods

2.1 Dismantling, separation, milling and characterization methods of LED lamp

Approximately 176 kg of bulb-type LED lamps were stored at TRAMPPO - Sustainable Lamp Management, packaged in drums, boxes, and big bags.

Due to the small amount of lamps stored, instead of carrying out a sampling procedure, it was decided to use all available material. Consequently, was performed a procedure aiming at the quartering of the lamps, in order to obtain aliquots for the tests in laboratories of the Institute for Technological Research (IPT). After weighing, occurred the transfer of all stored bulb-type LED lumps to a single big bag, which was lifted with the aid of a forklift. After that, a technician cut a part of the bottom out, in order to release the light bulbs and form an approximately conical pile on the floor. The pile formed was divided into four quadrants, which were stored separately in boxes identified as aliquots 1 to 4. It is important to point out that any type of LED lamp other than the bulb type was removed at this stage of the process.

At IPT, one of the aliquots was quartered again, concerning to generate a sub-aliquot of about 2 kg that was dry comminuted using a laboratory Willye knife mill (model Star FT 80, Fortinox). Subsequently it was pulverized in an oscillating disc mill for chemical analysis (X-Ray Fluorescence) and for solid waste classification (NBR 10.004: 2004) [16].

The chemical analysis of the milled aliquot of bulb-type LED lamps was carried out by X-ray fluorescence (Malvern Panalytical, Zetium), using a pressed sample without a calibration standard for the chemical elements comprised between fluorine (F) and uranium (U). It is, therefore, a semi-quantitative analysis. For a better understanding of the composition of individual components obtained from the disassembly of bulb-type LED lamps, scanning electron microscopy (SEM, JEOL JSM 6300) coupled with an energy dispersive spectrometer (EDS, Noran System) was performed. The components were covered with Au and were analyzed through an acceleration voltage of 20 kV in secondary electron mode. One electronic board containing the LED packages was cut in a diamond disc in a transverse section and was polished with diamond paste. The purpose of this procedure was to analyze the multi-layer structure of the LED component. Raman spectroscopy was used to identify some components of the bulb-type LED lump. The spectra were obtained from a WiTec Confocal Raman spectrophotometer, model alpha 300R (WiTec GmbH, Germany). Excitation line was used in the visible region at 633 nm of an argon laser (WiTec brand, S/N 100-1665-154). Laser power was kept below 10 mW to avoid sample degradation. Each spectrum corresponds to the average of 100 accumulations, acquired with an integration time of 50 s.

2.2 Solid waste classification

The milled aliquot of bulb-type LED lamps was analyzed according to ABNT NBR 10.004: 2004 - Solid Waste – Classification [16]. This Standard classifies solid waste according to its potential risks to the environment and public health so that it can be properly managed.

3 Results and discussion

3.1 Chemical composition by X-Ray fluorescence

Table 1 presents the results obtained for the milled sub-aliquot samples of bulb-type LED lamps. The results are presented as elements. The high value of loss on ignition (LOI) of about 58% obtained in the analysis indicates that most of their components are made up of organic materials, mainly polymers.

Of the remaining, almost 5% is aluminum, 5% iron, 3% silicon, 2% copper, 3% bromine, 2% zinc and 2% calcium. Other elements are present in smaller quantities, with values below 1.5%, such as sodium, magnesium, phosphor, sulfur, chlorine, potassium, titanium, chromium, manganese, nickel, strontium, yttrium, zirconium, niobium, tin, antimony, barium, lead, and bismuth. Most of these elements were observed by SEM/EDS analyses as will be presented in the next section. Br and Sb were not observed by energy dispersive spectroscopy.

This is probably due to the presence of these elements in the polymer as flame retardant, but the polymer was not analyzed by SEM/EDS in this work. Charitopoulou et al. [17] reported that tetrabromobisphenol A (TBBPA) is one of the most material used as flame retardants in the polymer or polymer blend in electric and electronic devices, besides polybrominated biphenyls (PBB), for instance. Niu et al. [18] studied flame retardancy of a nano-Sb_2O_3–brominated epoxy resin (BEO)–poly (butylene terephthalate) (PBT) composite.

3.2 SEM/EDS and Raman Spectroscopy Analyses

3.2.1 Base (Socket)

Figure 1 shows scanning electron micrographs and EDS spectra from a sample of a socket (Edson screw type) of one type of bulb LED lamp. Cenci et al. [19] studied the chemical composition of bulb-type LED lumps and reported that sockets are constituted of aluminum. Dodbiba et al. [20] related the physical properties of various components of bulb LED lamps indicating that their Edson screws are composed of Cu-Zn-alloys, Ni, and black glass. Martins et al. [14] reported a composition of 81.78 wt% of Fe and 17.44 wt% of Ni for a type of Edison screw used in a type of bulb LED lump. In our work, the major constituents of the socket are Ni and Fe, showing that different materials could be used to produce this component in the LED lighting system design.

Table 1. Chemical composition (wt%) of the milled aliquot of bulb-type LED lamps

	Na	Mg	Al	Si	P	S	Cl	K	Ca
%	0.09	1.47	4.57	2.80	0.04	0.08	0.13	0.04	2.44
	Ti	**Cr**	**Mn**	**Fe**	**Ni**	**Cu**	**Zn**	**Br**	**Sr**
%	1.37	0.01	0.96	4.79	0.12	2.28	1.70	3.13	0.03
	Y	**Zr**	**Nb**	**Sn**	**Sb**	**Ba**	**Pb**	**Bi**	**LOI**
%	<0.01	0.04	<0.01	1.05	0.36	0.13	0.14	<0.01	57.6

Figure 1. (a) Photography of a socket of one type of bulb LED lamp, (b) micrograph of one piece of the socket, obtained by SEM, and (c) EDS spectrum of the region indicated in orange in figure b.

3.2.2 Polymer housing

Figure 2 presents the Raman spectra of the polymers used as reflector (polymeric cover) and as polymeric housing. The reflector was identified as polycarbonate according to Zimmerer et al. [21] and Resta et al. [22]. Some references indicate that polycarbonate (PC) and polybutylene terephthalate (PBT) are the main materials being used as reflector in bulb-type LED lumps [14,19,20]. The polymeric housing seems to be constituted of a polycarbonate blend [23].

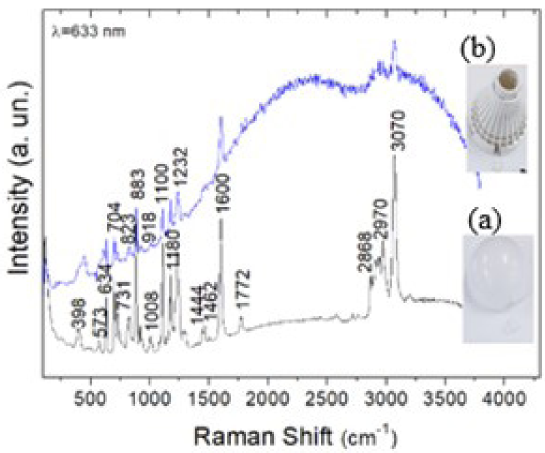

Figure 2. Raman spectrum of the reflector (a) and polymer housing (b) of on type of bulb LED lamp.

3.2.3 LED Driver

Figure 3 presents a photography of PCB (printed circuit board) within the emphasis of one capacitor. The micrograph obtained by scanning electron microscopy, of the surface of the capacitor indicates the presence of Si (Figure 3b, area 2), probable due a silicone rubber. The wire that connect it to the board is composed of Sn. Figures 3e, 3g and 3h show that the foils inside the capacitor are composed of one material rich in C and O that can be attributed to a capacitor paper (separator) and a second is rich in Al (cathode).

Figure 4a presents a photography of PCB of one bulb-type LED lamp, and Figures 4b and 4c shows EDS spectra of the points indicated in Figure 4a. The EDS spectra show that the major constituent of the surface of BP3125 is silicon. According to the data sheet [24] of this device it is a high primary-side feedback and regulation controller for LED lighting and integrates power MOSFET (metal oxide semiconductor field effect). It is connected with Cu and Zn as show in Figure 4c.

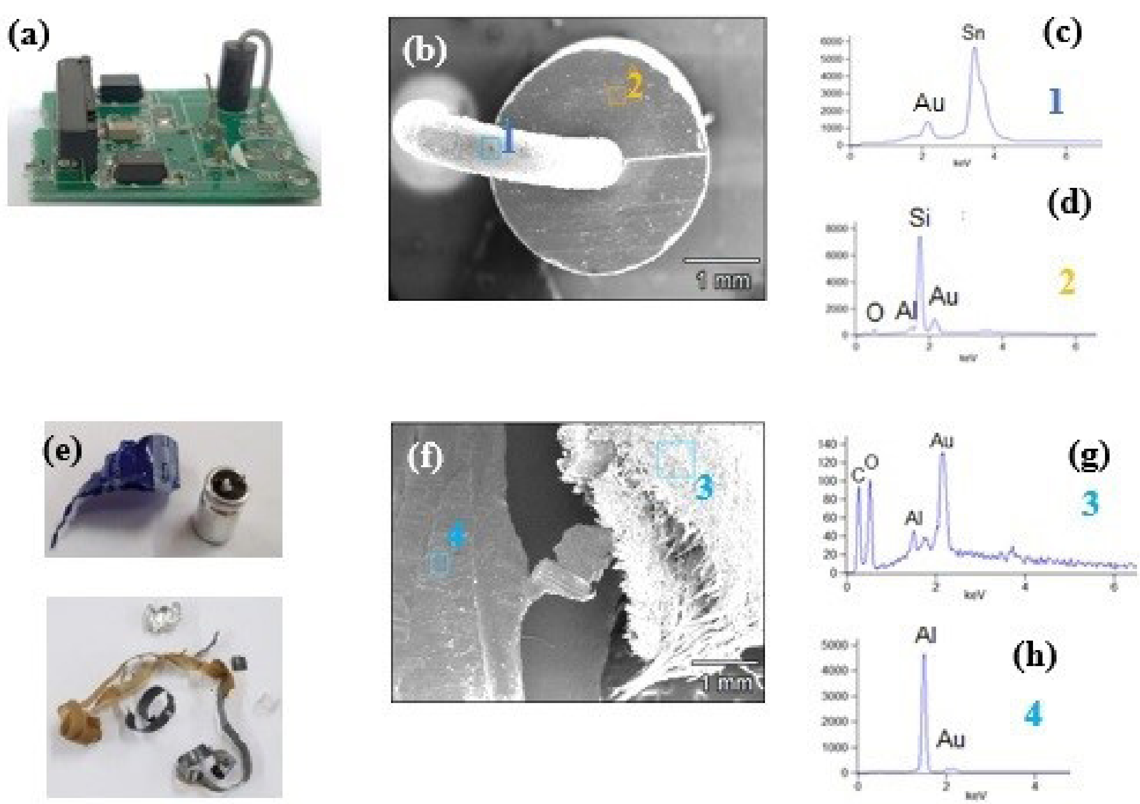

Figure 3. (a) Photography of the PCB of the one type of LED lamp, (b) micrograph by SEM of a capacitor, (c) and (d) EDS spectra of the regions 1 and 2, (e) photography of an opened capacitor and their insider components, (f) micrograph of the insider components, (g) and (h) EDS spectra of region 3 and 4 (figure f).

Figures 5 shows other regions of the printed circuit board, indicating the presence of Sn, Pb, Cu, Si, Ti and Ba. Illés and Kékesi [25] reported that Sn-Pb is used as solder although it has been moderately replaced by Sn-Ag-Cu in the European Union and USA. Mir et al. [26] reported that Pb are employed in electrical contacts and solders, while metals such as Cu, Sn, and Al are intended to be used for mechanical, thermal, and electrical purposes. In our work, Ag was not found by SEM/EDS analyses. It can be seen from Figure 5c a region rich in Ti and Ba, indicating the presence of barium titanate that is widely used in various electro-optic devices [27].

3.2.4 LED module

Figure 6 shows a photography of the LED module and the micrographs of the bottom side and upside part (white) obtained by scanning electron microscopy. The bottom side is composed of aluminum and the upside is covered with TiO_2.

The first attempt to analyze the LED package was to detach it from the Al/TiO_2 plate (Figure 7). Figure 7b shows the enlarged region marked in white in Figure 7a. From the analysis of energy dispersive spectroscopy, elements such as C, O, Si, Ti, Al, Y and Ga were observed. Table 2 presents semi quantitative analyses of the region analyzed. Alim et al. [28] reported that yttrium aluminum garnet (YAG): Ce phosphor particle as well gallium nitride (GaN), or gallium phosphide (GaP), for example, are some of the commonly used materials in manufacturing LED chips.

In a second attempt the LED module was cut in cross section. It was polished with diamond paste and then covered with Au, to be analyzed by SEM/EDS. Figure 8 shows a micrograph and EDS spectra from the area in Figure 8c showing that one of the layers, of the cross section, is composed of spheres rich in Si, Ca, Al and O, possibly a glass phase (Figure 8e). Figure 8d shows that particles lower than 25 µm are constituted of O, Al, Y and Ce, whose composition is presented in Table 3. Using the

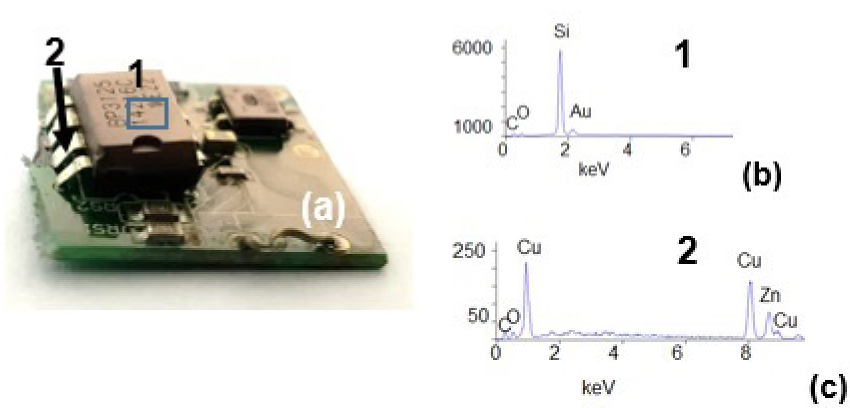

Figure 4. (a) Photography of the PCB of a type of bulb LED lamp circuit; (b) and (c) EDS spectra of the regions indicated in Figure 4a.

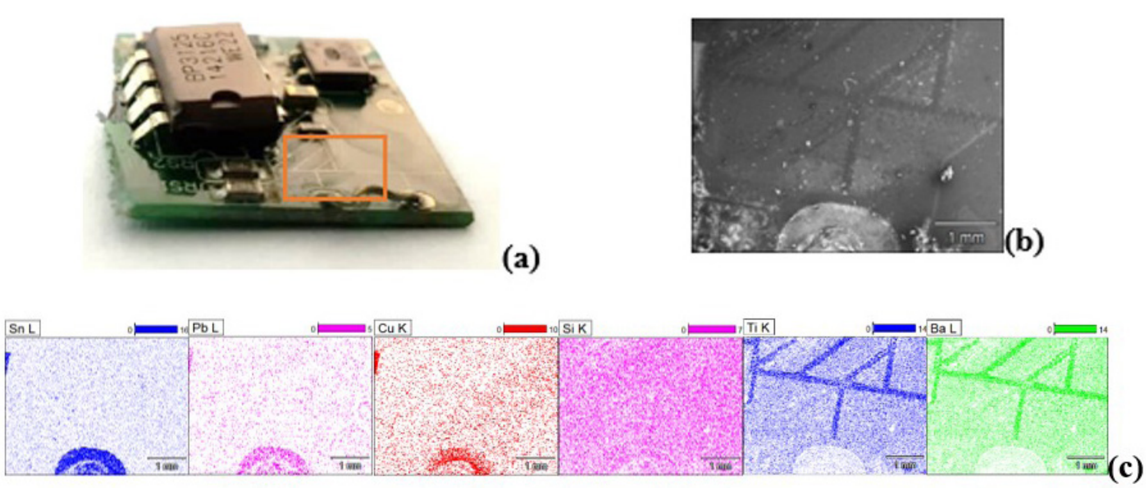

Figure 5. (a) Photography of the PCB of a type of LED bulb lamp, (b) micrograph by SEM of the region indicated in (a) and (c) mapping of the elements Sn, Pb, Cu, Si, Ti, and Ba in the region of the micrograph of PCB shown in figure b.

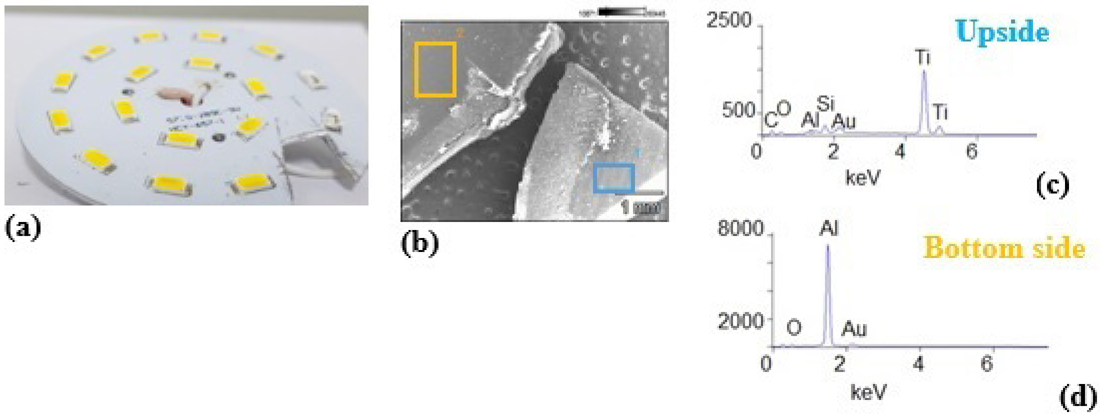

Figure 6. (a) Photography of the LED module, (b) micrograph by SEM of the bottom side (left) and upside part (right) of the module and (c) and (d) EDS spectra of the regions indicated in figure b.

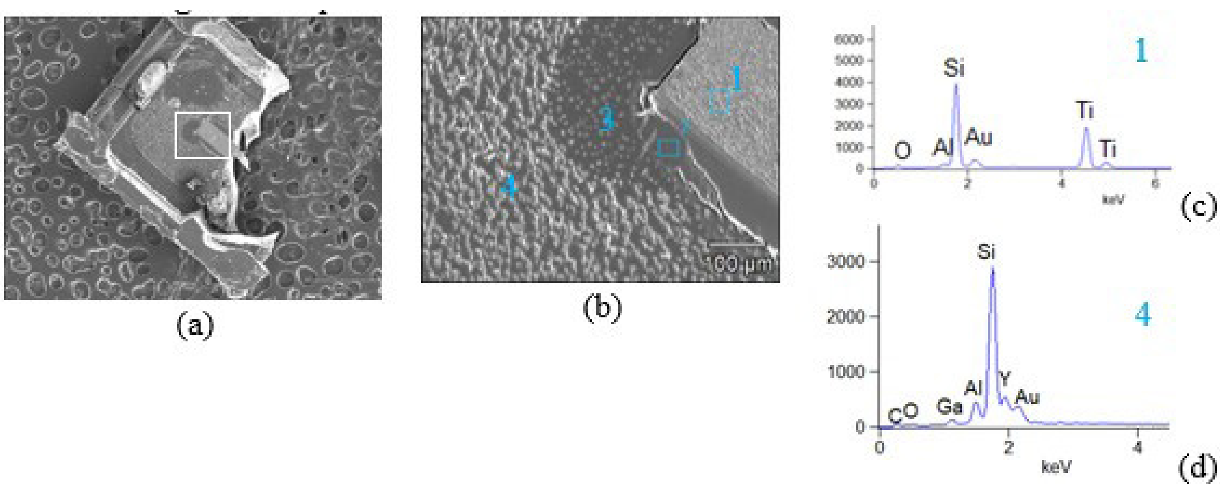

Figure 7. (a) and (b) Micrographs, obtained by SEM, of the LED package and (c) and (d) spectra of the regions 1 and 4 indicated in figure b.

Table 2. Chemical composition (wt%) of points 3 and 4 indicated in Figure 7b

Elements	C	O	Al	Si	Ga	Y
Area 3	20.9	12.5	10.5	33.4	2.16	20.5
Point 4	29.1	13.9	4.4	31.1	12.3	9.14

Table 3. Chemical composition (wt%) of the regions indicated in Figures 8d

Elements	O	Al	Si	Y	Ce
Area 1	-	31.22	-	65.58	3.20
Area 2	8.50	30.25	-	57.93	3.32
Area 3	-	-	100	-	-

optical microscope coupled to the Raman spectrometer, it was possible to find particle like those observed by SEM in the polished section of LED chip. By using the excitation line 633 nm in these particles, Raman spectra were obtained and one of them is shown in Figure 9. The bands present at 150, 220, 263, 340, 370, 406 and 777 cm^{-1} are attributed to YAG ($Y_3Al_5O_{12}$) according to Lukowiak et al. [29] and Abdullin et al. [30]. It was possible to observe the YAG:Ce particles in the polished sample. The Raman analysis in the surface of the LED chip (Figure 9) shows spectrum with bands at 620, 1000, 1030, 1140, 1161, 1195, 1573 and 1598 cm^{-1} characteristics of polystyrene [31].

With the exception of B, Sb, Na, P, K, Zr, Nb, and Bi, the other elements, determined by FRX, were detected in the SEM/EDS analyses. B and Sb are commonly used as flame-retardants in polymers and the polymeric components were not analyzed by this technique. From the XRF analysis, Na, P, K, Zr, Nb, Sr and Bi are present in the milled aliquot of the bulb-type LED lamps in small amounts (< 0.12 wt%).

Table 4 shows a summary of the results obtained by FRX, EDS and analyses of phase composition by micro-Raman spectroscopy in the different components of bulb-type LED lamps.

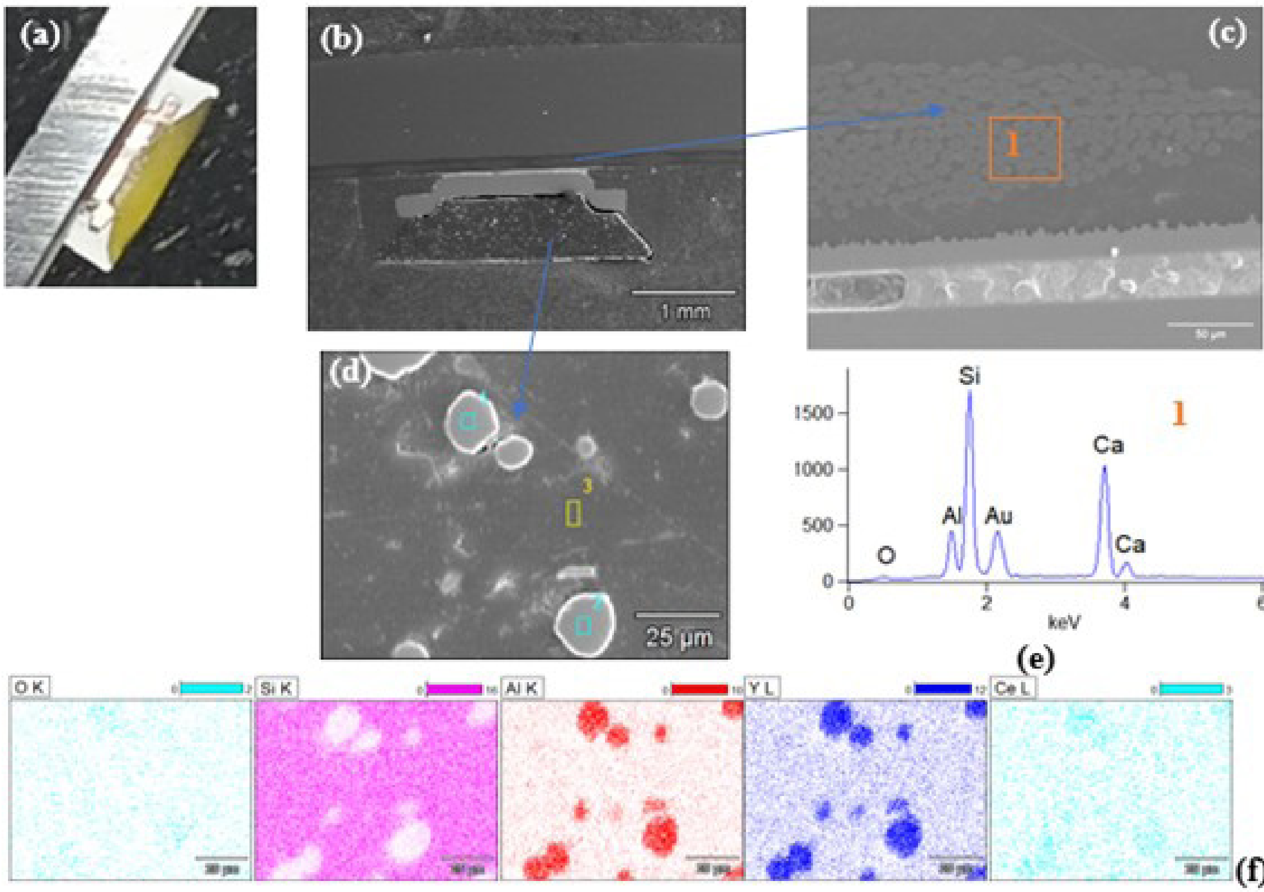

Figure 8. (a), (b), (c) and (d) micrographs by SEM of the cross section of LED module (e) EDS spectrum of the region 1 indicated in figure c and (f) elemental mapping of the total region in figure d.

Table 4. Distribution of the elements determined by FRX, EDS and analyses of phase composition by micro-Raman spectroscopy in the different components of bulb-type LED lamps

Component	Metal	Polymer	Ceramic
Socket	Ni and Fe		
Screw	Ni, Fe and Cu		
Diffuser		Polycarbonate	
Driver and PCB	Cu, Sn, Pb, Cr, Mn, Al, Zn, S, Cl, Si	Silicone rubber	Al_2O_3 and $BaTiO_3$
		Cellulose (paper)	
LED Module	Si, Cu, Sn, Pb, Ga(*)	Epoxy resin, polystyrene	TiO_2, YAG:Ce ,
			Ca-Al-Si-O
Heat Sink	Al	Polycarbonate blend (polymer housing)	

(*) It was not possible to confirm if Ga is present as GaN or GaP.

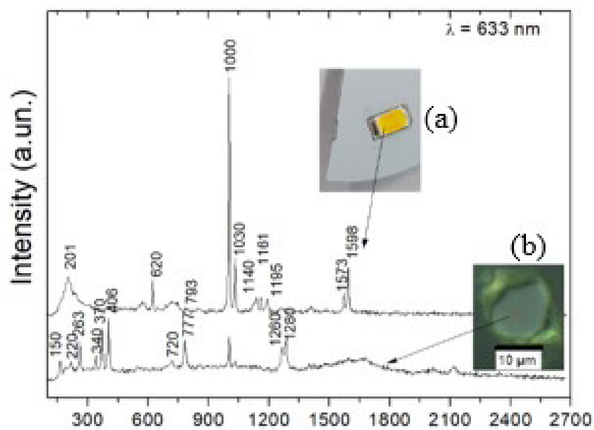

Figure 9. Raman spectrum: (a) surface of LED chip and (b) polished LED surface.

3.3 Solid waste - classification

The milled aliquot of bulb-type LED lamps was analyzed according to NBR 10.004:2004 – Solid Waste – Classification [16]. From the results presented in Table 5, it can be concluded that it refers to Class I – Hazardous, since the concentrations of lead (in the leached extract), aluminum, and total phenols (in the solubilized extract) are above the maximum value allowed by the cited norm. Therefore, if improperly managed, it poses a serious threat to human health and the environment.

Table 5. Analyzed and reference values of lead, aluminum, and total phenols according to NBR 10.004-2004 [16] for the milled aliquot of bulb-type LED lamps

Compound	Extract	Analysed value (mg/l)	Reference value (mg/l)
Lead	Leached	1.48	1.00
Aluminum	Solubilized	0.61	0.20
Total phenols	Solubilized	0.03	0.01

4 Conclusion

The bulb-type LED lamps, considered in this work, not only present strategic elements such as Ga, Ce, Y, Al, Cu, Ni, Si, and Cu but it is also considered a Class I (Hazardous) waste that justifies an effort to find suitable technological routes for its recycling, as they already have been presented in the literature. Ce and Y are rare earth metals used for the production of lasers and persistent phosphors, for example. In and Ga are scarce metals used as semiconductor in conjunction with Si. Nowadays Cu is used intensively in electric vehicles, and is becoming a rare and expensive metal. One of the challenges for a technological route is the adequate separation of the different components that compose it. In this sense, the work developed in partnership IPT/Tramppo/Embrapii resulted in a patent filling (BR 102019026715-1 A2) that has great potential for this purpose.

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