Facile method for the selective recover of Gd and Pr from LCD screen wastes using ultrasound-assisted leaching

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Abstract
Rare earth elements (REE) are essential for the production of technological devices. However, their high demand and low availability, together with an increase in electronic waste generation, compel the development of efficient, economic and green methods for recovering these elements from electronic waste. In this work, a facile method for selective recovering of REE from Liquid Crystal Display (LCD) screen wastes, employing ultrasound assisted leaching is presented. The screen wastes were milled and sieved to pass through – 325 mesh sieve. The milled powder was summited to ultrasound-assisted leaching in aqueous medium, at room temperature (25 °C) and pH 6 for 60 minutes. Subsequently, a magnetic separation was applied into the leach residue. ICP was employed to quantitatively analyze the composition of the LCD powders and the effectiveness of the extraction process. SEM-EDS allowed qualitatively analyzing the chemical composition of the solid materials. The results shown that the LCD screen wastes are formed, mainly, by amorphous oxides of Si, Fe, In, Sn and REE. The amount of Gd and Pr in the wastes were 93 mg/kg and 24 mg/kg, respectively; It represents enough reason to recover it. X-ray diffraction analysis of the magnetic portion of the leaching residue, confirmed the presence of an amorphous phase together with crystalline metallic iron alloy. The magnetic behavior, obtained by Vibration Sample Magnetometry, helped to understand the nature of the residues. The formation of this metallic alloy is attributed to the effect of high power ultrasonic during the leaching. It was confirmed that the magnetic residues concentrates and recovers 87 wt. % of Gd and 85 wt. % of Pr of the total content of these REE in the magnetic residue. Therefore, ultrasound-assisted leaching is a selective and facile method for recovering Gd and Pr from a screen wastes.

1. Introduction
Currently, electronic waste has become one of the major contributors to environmental pollution, mainly due to the politics of programmed obsolescence of many electronic devices over the past 20 years [1]. Now-a-days, the average life of a computer or a mobile phone is between 4 and 5 years [2], considerably increasing the amount of electronic waste generated [3]. Since the end of the 20th century, LCD screens have been used in various electronic products due to the advantages of light
quality, small volume and low energy consumption; therefore, more than 700 million LCD panels have been produced worldwide in recent years. Considering the average lifespan of 3 – 8 years of these panels, large quantities of LCD panels will be reaching their end of life in the coming years, tremendously contributing to the generation of electronic waste [4]. Despite the many disadvantages, these wastes contain important amounts of different elements with high commercial value [5, 6]. Among these, the REE [7] are considered critical raw materials [8], due to the difficulty of their separation, acquisition and the uncertainty of their disposition [9]. Recent studies have shown the quantities of REE (yttrium, europium, cerium, principally) in LCD screens are economically attractive for recovery if, however, the proposed methods have focused mainly on the backlight or LED´s of the LCD screens [10, 11]. In contrast, this investigation studies the recovery of Gd and Pr, among others, contained in the LCD panels, a subject that has not been reported. Besides, the importance of the recovery these REE is implicit not only in its use in new technologies, but also in their current high prices, which are among the REE with the greatest economic value [12].

The development of REE recovery strategies has been trending in recent years; most of the proposed processes employ inorganic acids such as HCl, H₂SO₄ or HNO₃ for leaching [13, 14, 15], which can cause damage to the environment and human health, if they are not adequately controlled. Additionally, more environmentally friendly reagents, such as sulfate-roasting followed by water leaching [16] and ionic liquids [17] have also been studied, to leach the REE. However, the chemicals and conditions employed in both proposals are costly and involve complicated downstream separation processes.

In the present investigation, the pyrophosphate ion (PPI) is used as a less hazardous alternative to inorganic acids, since it has been employed as a selective ligand for the REE ions in other studies [18]. It is also utilized for the preparation of medicines and the preservation of different foods, proving to be safe for the environment and for the human [19, 20]. A thermodynamic study, in the form of species distribution diagrams using the Hydra-Medusa software suite [21] was performed to determine the appropriate leaching conditions. In addition, ultrasound was employed during the leach as complementary method. Recent studies have shown that the use of ultrasound accelerates the
dissolution of metals [22] and rare earth elements, achieving nearly 100% in 1 to 3 hours [23–25]. In sonochemistry, molecules undergo a chemical reaction promoted by the application of ultrasound radiation (20 kHz – 10 MHz) in solid-liquid systems; ultrasound enhances the diffusion of soluble species in the liquid phase and increases the rate of penetration into the solid principally by the cavitation effect, which leads to the creation of many microcracks on the solid surface. Furthermore, if the raw material is a powder, ultrasound energy can cause particle rupture, with a consequent increase in surface area available for reaction [26].

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Hence, the present study explores the recovery of the REE compounds (Pr and Gd) from waste LCD screens by ultrasound-assisted leaching, using pyrophosphate ion. This proposal introduces a quick, easy and inexpensive method to recover these valuable elements from wastes.

2. Materials And Methods
The LCD screens were collected from electronic wastes. They were cleaned by separating the plastics films and subjected to the experimental procedure shown in Fig. 1, where the leaching assisted by
sonochemistry is shown. As may be observed in Fig. 1, the LCD screen wastes were milled for 30 minutes, using an automatic mortar grinder. The milled powder was sieved to pass through − 325 mesh sieve, since the largest quantity of RE elements is recovered by leaching at this particle size [27]. The powder obtained was characterized by X-ray diffraction, XRD, (Equinox 2000 diffractometer), and by scanning electron microscopy with Energy-dispersive X-ray spectroscopy (SEM-EDS-Joel, model IT 300). In addition, the LCD powders were digested in aqua regia (HCL:HNO₃, 3:1) and analyzed by inductively coupled plasma (ICP-OES, Perkin Elmer, Optima 3000 XL) in order to quantify the chemical composition.

For the leaching tests, three grams of the milled and sieved powder were immersed in 150 mL beaker, containing a solution with 0.05 M PPI agent (pyrophosphate ion, P₂O₇⁴⁻). The solution pH was adjusted and maintained at 6, using sulfuric acid. These experimental conditions were selected in accordance to the results of previous studies [18]. The leaching solution was sonicated for 60 minutes, using an Ultrasonic Homogenizer 300VT, equipped with a piezoelectric transducer at a frequency of 90 kHz and a solid titanium tip of 9.5 mm. The experiments were performed at a sonication output power of 120 W. The beaker was placed into an ice bucket, to guarantee that the temperature of the solution in all cases did not exceed room temperature (25 °C) after 1 h of ultrasonic irradiation. The leaching solution was filtered, obtaining a liquor and a solid phase (leach residue). The solid was air-dried at 80 °C and magnetically separated into non-magnetic and magnetic powders. The leach liquor, as well as the magnetic and non-magnetic solids, were characterized by XRD, SEM-EDS and ICP.

3. Results And Discussion

In Fig. 2, the XRD pattern of the milled LCD screen powder is presented. As may be observed, no specific diffraction peaks are exhibited, indicating an amorphous material. This result is expected, since the main component of the LCD screens is silicon [28], combined with small amounts of different metallic oxides, such as indium, REE and tin (not detectable by XRD due to the detection limit of the diffractometer).

To confirm the presence of rare earth elements (REE) in the milled and sieved LCD powder, SEM-EDS
qualitative elemental analysis were carried out. The results are shown in Fig. 3, where the qualitative chemical distributions of different elements are shown. silicon, aluminum, some REE, indium, tin and iron may be observed. In addition, all the RE elements are uniformly distributed in the small particles. As can be appreciated, the elements with the highest concentrations are silicon, aluminum and oxygen, probably as oxides compounds (SiO₂ and Al₂O₃), whereas the REE are concentrated in the smallest particles. Moreover, the fine particle size ensures the homogeneity of the sample and the percentage of the rare earth elements that can be recovered [29]. For this reason, the powder sieved at − 325 mesh (44 µm) was selected for the leaching study.

The results of the chemical analysis obtained by Inductively Coupled Plasma (ICP-OES) show the presence of rare earths, such as Pr (24 mg/kg), Gd (93 mg/kg), Er (477 mg/kg) and others elements, such as In (2422 mg/kg), Sn (835 mg/kg), Fe (2827 mg/kg) and Zn (9 mg/kg). According to the structural and chemical characterization (SEM-EDS and ICP), the LCD screen waste is composed of a mixture of oxides of Si, Al, Fe, and small amounts of oxides of Gd, In, Pr and Er. It is important to note that these materials are in sufficient quantities to justify their separation [30].

To select the adequate experimental conditions for selectively separating Gd and Pr from the other elements, a thermodynamic analysis using the Hydra-Medusa software [21] was performed, this analysis shows that Gd(III) and Fe(III) form soluble species Gd₂(PO₄)²⁺ and Fe(PO₄)⁻ with the pyrophosphate ion (PO₄)⁴⁻ up to pH 8. Furthermore, these ions precipitate as hydroxides in alkaline solutions (above pH 8). Species distribution diagrams are constructed from the logarithm of the reaction equilibrium constant (k) of the reagents [31]. However, praseodymium ion (Pr(III)) has not reported its value of k, but considering that the log k values for the REE close to Pr are similar (Nd³⁺ = 20, Sm³⁺ = 20.2, Eu³⁺ = 20.3, Gd³⁺ =20.5) [32], it is possible to infer that the behavior of this element with PPI is similar. This analysis helped to establish the adequate leaching conditions: pH values between 4 and 6, room temperature (25 °C), assisted with ultrasound in order to improve the dissolution process [25, 33].

After applying the ultrasonic assisted leaching process for 60 min at room temperature, a solid
residue was obtained (leach residue), which was analyzed by means of XRD (Fig. 4). As can be appreciated in Fig. 4 (a), the leach residue consisted of an amorphous material, together with small amount of crystalline Fe, which is identified as a peak near to 2-theta of 44°. Due to the presence of metallic iron, the residue was subjected to a magnetic separation, obtaining a magnetic and a non-magnetic solid. Both solids were independently analyzed by XRD (Figs. 4b and 4c). As can be observed, the non-magnetic residue (Fig. 4b) shows an XRD pattern typical of an amorphous material, attributed to silica base material, which was not affected by the leach. In contrast, the magnetic residue (Fig. 4c) is a crystalline iron matrix, probably with small amounts of other metals (gadolinium, praseodymium or similar elements), since a slight displacement of the diffraction peak is detected from its theoretical position at 2-theta of 44°. At the same Fig. 4, the three residues (combined leach, magnetic and non-magnetic), were qualitatively characterized by SEM, using back-scattered electrons (BSE). As can be observed, the powders are composed by irregular and polygonal particles. In addition, there are no differences in contrast in each residue, which indicates that the residues contain a homogenous distribution of atoms along the particles. However, comparing the different residues, the magnetic residue (Fig. 4c) appears brighter, which may be ascribed to the presence of compounds that contain atoms with greater atomic number, such as REE.

To characterize their physical behavior, the magnetic hysteresis loops of each residue were acquired and are presented in Fig. 5. In this figure, it can be observed that the magnetic residue presents a saturation magnetization of 120 emu/g, attributed to the presence of an iron alloy with undefined composition, in good agreement with the XRD pattern show in Fig. 4(c). It is known that, pure iron shows a specific saturation magnetization near to 217 emu/g, therefore, the reduced magnetization value corresponds to iron, containing very low concentrations of materials that possesses slight magnetization, in accordance with the XRD patterns, since no other phases were detected. The non-magnetic residue shows ferrimagnetic behavior, with a very low specific saturation magnetization of approximately 0.08 emu/g, attributed to the presence of small amounts of ferrimagnetic materials as oxides, although these was not observed in XRD pattern due to the detection limit of the analysis equipment.
In addition, the magnetic hysteresis loop of the combined leach residue shows ferrimagnetic behavior, with a specific saturation magnetization around 0.19 emu/g. This confirms mostly amorphous silica and aluminum oxides, together with small quantities of ferrimagnetic materials, as iron and RE metals and/or oxides.

The chemical composition of the leach liquor and the solid residues (magnetic and non-magnetic) were quantified by ICP; the results are shown in Table 1. According to these results, the magnetic material (0.3 g) is composed mainly of Fe, Pr and Gd, which corresponds to 94.5%, 86.8% and 85.4%, respectively, of the total amount of each element in the LCD screens; this represents an important concentration of these elements, which is higher leaching efficiency comparing with conventional leaching [34]. On the other hand, 98.6% of the In, 73.9% of the Sn and 84.34% of the Er remained in the non-magnetic solid (2.58 g). As for the leach liquor, it contained appreciable percentages of Er (12.0%), Sn (24.6%) and Zn (91.2%).

| Element | Non-magnetic residue | Magnetic residue | Leach liquor |
|---------|----------------------|------------------|-------------|
|         | mg/Kg | % extrac. | mg/Kg | % extrac. | mg/Kg | % extrac. |
| Pr      | 3     | 13 | 21 | 87 | 0 | 0 |
| Gd      | 2     | 2 | 78 | 85 | 11 | 12 |
| Er      | 449   | 84 | 20 | 4 | 64 | 12 |
| In      | 1749  | 99 | 0 | 0 | 25 | 1 |
| Sn      | 542   | 74 | 11 | 2 | 180 | 25 |
| Fe      | 84    | 4 | 2089 | 94 | 37 | 2 |
| Zn      | 2     | 9 | 0 | 0 | 19 | 91 |

It is worth to mention that when the leaching process is carried out without PPI, the separation of Gd and Pr was not achieved, and these elements were not leached. In the same way, if the leaching is performed without ultrasound, a magnetic residue is not produced; therefore, the ultrasound radiation promotes the selective separation of Gd and Pr from others RRE, as magnetic materials and the leaching agent keep the soluble state of the REE.

As the magnetic residue shown a selective separation of Gd and Pr, together with iron, an elemental mapping was performed by means of SEM-EDS analysis, which is shown in Fig. 6. In this figure, it can be observed the presence of a homogeneous distribution of Fe, Gd and Pr, confirming the concentration of these elements into the magnetic residue.

The formation of an iron base alloy containing rare earth elements, as Gd and Pr, is an interesting
result itself, and it can be ascribed to the effect ultrasound during the leaching process. It is well-known that the ultrasound manages to produce mechanical effects, such as micro jets and shock waves, which cause microscopic turbulence in the solution and high-speed collisions between the solids [35]. These effects are difficult to achieve with conventional mechanical agitation [26].

According to some authors [35–36], sonochemistry or ultrasonic irradiation of water produces the free radicals H· and OH· that can combine to produce H₂O₂, which is a strong oxidant. [35]:

\[
H_2O \overset{\text{H· + OH·}}{\longrightarrow} H_2O_2
\]

\[
\cdot OH + \cdot OH \rightarrow H_2O_2
\]

The presence of H₂ and H₂O₂ promote chemical and physical effects since they can act as strong reducing agents, as follows:

\[
\text{Fe}_2\text{O}_3 + 3\text{H}_2\text{O}_2 \overset{\text{p}_2\text{O}_5^{4-}}{\longrightarrow} 2\text{Fe}_{(s)} + 3\text{H}_2\text{O} + 3\text{O}_2
\]

In the ultrasonic leaching, the formation of these agents promote an iron ion reduction from Fe³⁺ and/or Fe²⁺ to Fe⁰, as shown in Eq. (4), which could incorporate Gd(III) and Pr(III) into its crystal structure or they could be also reduced to metallic phases. These solid products can be recovered by applying a magnetic field, obtaining a concentrated magnetic residue composed mainly of Fe, Gd and Pr, as was demonstrated previously. Therefore, the magnetic separation of the residue formed after the ultrasonic assisted leaching, is a facile and economic method for concentrating Gd and Pr elements.

4. Conclusions
It was determined that LCD screen wastes have 93 mg/kg and 24 mg/kg of Gd and Pr, respectively. In order to retrieve these RRE, a facile method for selective concentrating of some REE is proposed. In
particular, Gd and Pr from LCD screen wastes can be effectively recovered by ultrasonic assisted leaching, using pyrophosphate ion as complexing ligand. A retrieval of 85 wt. % and 87 wt. % of Gd and Pr, respectively, was achieved, using an ultrasound-assisted leaching for 60 minutes at room temperature. The combination of ultrasound and leaching at room temperature showed positive impacts on enhancing the separation of REEs; substantial physicochemical changes occurred during the leach assisted with ultrasound, including structural transformations, chemical radical formation, chemical reduction, and even, compound decomposition. Structural analysis and chemical decomposition, promoted by the formation of water radicals, could explain the effectiveness of the ultrasound leaching in improving the recovery of Gd and Pr from LCD screen waste. However, other valuable REE, such as In and Er, remain in the solid residue.

Declarations

Availability of data and materials

All data generated or analyzed during this study will be made available on request.

Competing interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Authors’ contributions

Conceptualization, G.T.L.L. and A.M.B.M.; methodology, A.D.T.P. and A.M.B.M.; software, A.D.T.P. and G.T.L.L.; validation, A.M.B.M., F.S.D. and G.T.L.L.; formal analysis, A.M.B.M. and G.T.L.L.; investigation, A.D.T.P.; resources, G.T.L.L. and A.M.B.M.; data curation, A.D.T.P.; writing original draft preparation, A.D.T.P. and A.M.B.M.; writing review and editing, A.M.B.M., G.T.L.L., F.S.D. and A.D.T.P.; visualization, A.D.T.P. and A.M.B.M.; supervision, G.T.L.L.; project administration, G.T.L.L. and A.M.B.M.; funding acquisition, G.T.L.L. and A.M.B.M. All authors have read and agreed to the published version of the manuscript. All authors read and approved the final manuscript.

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Figures

![Experimental procedure diagram](image-url)

Figure 1

Experimental procedure
The XRD pattern of the milled LCD screen powder.
Figure 3

The qualitative chemical distributions of different elements
A solid residue was obtained (leach residue), which was analyzed by means of XRD. (a), the leach residue consisted of an amorphous material, together with small amount of crystalline Fe, which is identified as a peak near to 2-theta of 44°. (b and c) the residue was subjected to a magnetic separation, obtaining a magnetic and a non-magnetic solid.
The magnetic residue presents a saturation magnetization of 120 emu/g

Figure 5
Figure 6

SEM-EDS analysis