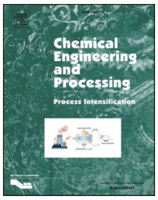
Chemical Engineering & Processing: Process Intensification 204 (2024) 109937

Contents lists available at ScienceDirect

Chemical Engineering and Processing - Process Intensification

journal homepage: www.elsevier.com/locate/cep



Process intensification in metal recovery from solid waste: Challenges, opportunities and recent advances

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A R T I C L E I N F O

Keywords:
Metal recovery
Process intensification
E-waste
Sustainable development
Green technologies
Precious metals

A B S T R A C T

The increasing production of electric and electronic devices corresponds to the significant increase of e-waste. These solid wastes contain a great amount of metals, thus representing a secondary source of precious elements, within a circular economy context. The recovery of metals from waste thus provides a great opportunity to decrease the energy consumption and the environmental impact associated with the typical processes for metal extraction. Along with the conventional recovery methods (i.e., pyrometallurgy and hydrometallurgy), some emerging technologies are being developed with a particular emphasis on the process intensification (PI). Greener leaching agents, lower temperatures and the combination of different approaches are the most reported methods to obtain a more sustainable metal recovery. In this perspective article, the recent advances in metal recovery technologies are critically reviewed, focusing the attention PI strategies adopted to improve the recovery efficiency and reduce the environmental impact of the whole process. Some tolls, such as the design of experiments (DoE), life cycle assessment (LCA), and machine learning are proposed to address the challenges and improve the dissemination of innovative solutions.

1. Introduction

The growing demand for electrical and electronic equipment (EEE) implies consequently the increase of the e-waste production, which is accelerating faster than the other waste streams [1–3]. By 2030, EEE is indeed projected to rise to 74.7 million metric tons due to the unprecedented rate at which electronic devices are being replaced [4].

As reported by Nithya et al. [5], all electrical or electronic equipment discarded as waste are the as called *e-waste* (also called as WEEE). The interest in these solid wastes arises from the presence of different valuable materials, such as precious metals, that can be recovered in the view of a circular economy. Typical WEEE is mainly composed of a metallic part (about 60 %), plastic components (about 20 %) and other materials (<20 %) such as glass, ceramics etc. [6] (Fig. 1). Hence, several authors considered e-waste as a non-natural ore [5], highlighting the possibility of extracting precious elements from waste as urban mining [7].

Based on the existing technologies, more economically feasible, sustainable and eco-friendly solutions for metal recycling should be developed, with the aim of reducing the environmental impact and the costs associated to these processes [2].

An extended literature survey is present about the most consolidated techniques for metal recovery from WEEE, and some indications are reported on the environmental impact of such techniques [8–10]. As reported by Liu et al. [11], even the currently advanced technologies employed for the recycling of WEEE produce large concentrations of pollutants (i.e., heavy metals or persistent organic compounds) which are then detected in the atmosphere.

The emerging methods, which are directed forward the application of more environmentally friendly approaches should get a comprehensive view both on the efficiency in the recovery of the desired elements and on the possible release of pollutants in the environment. As possible solutions, the utilization of greener leaching agents, as well as lower temperatures are proposed [12–14]. Along with the development of new technologies, some authors have proposed the possibility of modifying or combine the existing ones, despite the largest part of them is still on a laboratory scale [1].

In the present perspective, an overview on the recent advances in the metal recovery techniques, and the most interesting solutions for a process intensification (PI) will be discussed, highlighting the existing challenges and drawbacks of this field. The environmental and techno-economic analyses will be integrated with the circular economic framework, highlighting the different technologies' shortcomings and limitations, and possible future viewpoints.

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https://doi.org/10.1016/j.cep.2024.109937
Received 30 July 2024; Accepted 6 August 2024

2. Metal recovery from WEEE – Existing technologies, recent advances and main drawbacks

Generally, the recycling process involves different steps, as depicted in the Fig. 1.

The process generally begins with a pre-treatment (i.e., dismantling, size reduction and physical separation), through which non-ferrous metals are separated from the other components [15,16].

Dismantling, size reduction and physical separation are consolidated approaches, and can be modified based on the successive steps to perform [17,18]. However, the formation of fine dust, as well as the high energy consumption accompanying the crushing step are among the obstacles involved in this process [19]. Despite the pretreatment being widely employed on industrial scale, some authors have recently proposed the possibility of treating the printed circuit board as obtained after the dismantling [20,21]. This approach can certainly reduce the costs of the pre-treatment but can negatively impact on the dissolution efficiency, due to diffusive phenomena occurring in the system when the successive steps are performed [22–24].

After the pretreatment, the common techniques for the effective metal recovery are pyrometallurgical, hydro-metallurgical and bio-hydrometallurgical routes.

Pyrometallurgical approach can be considered as the most consolidated technique for metal recovery, in which the main advantage is represented by the possibility of minimizing mechanical pretreatment of the solid waste. The separation of the desired metals from WEEE occurs through the use of high temperatures. Specifically, the waste materials are subjected to incineration, sintering, and melting at high temperatures, with smelting furnace or plasma processes among the most used incinerator employed [4]. Among the disadvantages, high energy requirement and costs, low selectivity, and the emission of hazardous gases are the most important and highlight the necessity of developing possible different solutions [17,4,25]. **Hydrometallurgy is the second most reported technique for metal recovery**, involving different steps such as leaching, purification and recovery. The process is carried out in aqueous solution, with advantages as the lower costs and environmental impact with respect to the pyrometallurgical ones, as well as the good control of impurities. The leaching step is typically performed in the presence of strong acids (i.e., H_2SO_4, HNO_3, HCl, etc.), thiourea, cyanides, thiosulphate, ferric chloride or aqua-regia [4]. Despite the better characteristics with respect to the pyrometallurgy, these processes have low selectivity for the valuable metals (i.e., precious metals), and the production of large amounts of wastewater and emissions of chlorine gas [1]. Recently, there has been a growing interest in the **bio-hydrometallurgical pathways as a possible alternative option** for the dissolution of metals from e-waste, due to their cost-effectiveness and eco friendliness [26]. With reduced greenhouse gas emissions, the mechanism leads the selective recovery of some metals from WEEE, through the use of bacteria, which are able to secreting acids, ligands, and lixiviants for solubilization. However, this technology is still at a pilot scale, and several researchers are currently devoted to *(i)* the possibility of using bacteria with high resistance to different environments and *(ii)* the optimization of the factors affecting the leaching efficiency [27].

As is evident, and widely reported in the literature findings [4, 28–30], each of the above-mentioned technologies presents some disadvantages, and the design of new strategies or the combination of different low-cost and eco-friendly approaches in the field of metal recovery is urgent. Moreover, the heterogeneous nature of the WEEE leads the necessity of controlling process parameters based on the solid waste [31].

In this context, PI can *(i)* enhance the performance of the metal recovery, *(ii)* reduce the environmental impact of the adopted processes and *(iii)* reduce the costs associated to the whole process [4,6]. In the next section, some considerations on possible PI will be discussed, focusing the attention on the current challenges and opportunities.

3. Process intensification in metal recovery

PI in metal recovery has gained great attention due to the possibility of increase the profit. As a result of the PI, process plants are smaller, the environmental impact and the energy consumption are reduced, and some disadvantages of the existing technologies are overcome. Some years ago, some authors proposed **the combination of extraction and stripping (single stage)** in the liquid emulsion membrane process as a PI for the extraction of Ru, avoiding the stripping stage commonly adopted in these processes [32]. The possibility of reducing the number of stages in the extraction process is clearly able to reduce the energy

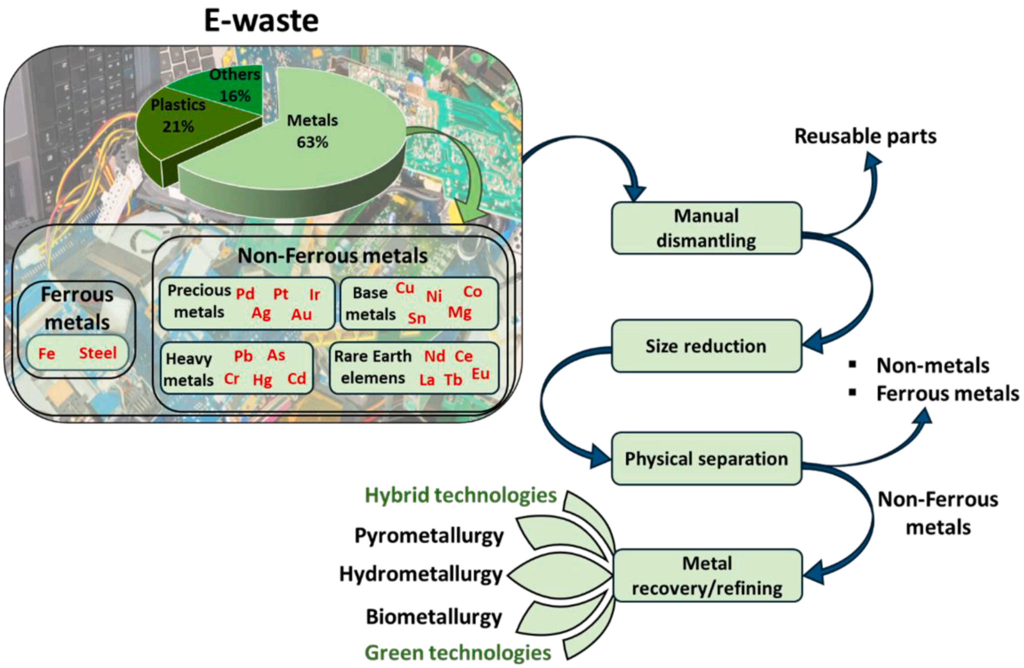

Fig. 1. Schematization of the main steps involved in the metal recovery process.

requirement, which is in accordance with the PI approach.

Furthermore, some authors directed their efforts in the **minimization of carbon footprint and atmospheric emissions** [33] in the pyrometallurgical route. Zhou and co-workers [34] critically reviewed some challenges and evolutions in the pyrometallurgic recycling of spent lithium-ion battery, highlighting how using lower temperatures can avoid the damage of the electrolyte structures as well as reducing pollution, but involves additional energy and costs consumption.

In the hydrometallurgical methodology, as alternative to the strong acids commonly employed, some authors have reported the use of **bio-based organic acids** such as acetic, lactic, formic or citric acids. These substances drive towards more sustainable strategies, reducing the environmental impact, the costs and the operating conditions of the process [35]. In some cases, the use of ligands in the leaching solution is proposed in combination with greener oxidizing agents (i.e., O_2 or H_2O_2) [36].

Moreover, some authors have recently proposed the combination of pyrometallurgical processes with bio/hydro-metallurgical routes for PI purposes. With specifical focus on lithium-ion batteries for instance, He et al. [37] highlighted how the **combination of different techniques** can reduce the environmental impact due to the application of milder conditions in terms of temperatures (below 1000 °C) and pH.

In this context, the **combination of different sustainable technologies** was recently proposed for the recovery of precious or semi-precious metals from a variety of electronic waste. As an example, the different sustainable stages can be adopted for the recovery of nickel from exhausted nickel-containing multilayer ceramic capacitors (MLCCs) [38]. Specifically, after the dismantling of MLCCs from the e-waste and the crushing with a pestle and a mortar, a leaching under mildly acidic conditions in the presence of NaCl and $CuCl_2$ can be carried out for the oxidation of the metallic Ni. Subsequent photodeposition and pH adjustment stages are useful to effectively separate the metals present in the system (i.e., Ni and Cu). Similarly, the recycling of palladium from spent catalysts can be achieved, noticing the effective recovery of the metal by adopting the above-mentioned leaching procedure in association with a photodeposition stage through which the formation of zero-valent palladium deposited on ZnO is obtained [39]. The use of ZnO allows dissolving it at pH lower than 6.30, thus obtaining the pure metal (i.e., Pd).

Furthermore, as a PI of the common acid-based leaching processes, **subcritical water extraction** and **microwave-assisted extraction** were employed for the recovery of Ni, Co, La, Nd, and Ce from spent NiMH batteries [40]. Subcritical water extraction resulted in the highest recovery efficiency, while microwave-assisted extraction demonstrated great potential in terms of energy-effectiveness in comparison with the other techniques. Conversely, the longest reaction time and the highest energy consumption were required for the common leaching process. Moreover, as a possible PI for metal recovery, some authors have identified the **adoption of microfluidics devices** [6], which are able to improve the mass and heat transfer and, consequently, the efficiency of the process.

However, all these proposed solutions are still far from the large-scale application, and more studies are needed. In this regard, for the development of new solutions on a lab scale it is mandatory a **design of experiment (DoE)**. Sujatha et al. [41] for instance, used the statistical approach for the optimization of a Ni extraction process by green emulsion liquid membrane. Firstly, they evaluated the most important parameters affecting Ni extraction through a parameter screening using Plackett Barman design. Then, the identified parameters were analysed and optimized for PI purposes. Similarly, Hemmati et al. [42] investigated indium extraction from WEEE through flat sheet supported liquid membrane via response surface methodology (RSM), and artificial neural networks (ANN). Through a low number of experiments, the effect of different parameters was explored and modelled, and the optimal conditions were identified. The use of RSM was also reported by others for the selective recovery of Cu [43,44], Cu and Au [45], Ag and Au [46], demonstrating the potential of this approach in the process optimization, as well as in the development of new processes.

Furthermore, **combination of different bio-based processes** (i.e., biosorption, bioleaching, biomineralization) was identified as an interesting methodology for the efficient and selective recovery of metals from WEEE [47]. However, some challenges to the practical applications are still limiting this solution. Indeed, along with the environmental interests, the economic aspects are crucial for the effective development of new commercial-available technologies [48].

In this context, a **techno-economic assessment** of an integrated recovery system based on bio and hydrometallurgy was reported by Van Yken et al. [49]. They identified, even on a lab scale, the parameters (i.e., pH and pulp density), able to optimize the metal leaching and reduce the operating costs of the process. Similarly, a techno-economic evaluation of a miniaturized Cu-solvent extraction plant (MSXP) was recently described by some authors [50]. They demonstrated a great reduction in experimental costs using MSXP with respect to the most employed devices, along with a reduction of the start-up time, etc. The proposed set-up has proven to be a great ally in the PI approaches, due to the possibility of improving the efficiency of the experiments on a lab and pilot scale, which represent the first steps for the development of new processes for metal recovery.

Moreover, a PI of the process [39] for the recovery of Pd from spent materials (mentioned in the previous section) was reported recently [51]. Specifically, the possibility of recovering the streams coming from the different units (i.e., leaching and photodeposition) was evaluated. Furthermore, the photocatalyst employed in the photodeposition unit was prepared different times starting from exhaust effluents generated during the recovery process. Then, to assess the environmental impact of the procedure, a **life cycle assessment** (LCA) was performed on a laboratory scale, which highlighted the critical points and guided the exploration of different configurations able to enhance the beneficial effects of the process [10]. LCA can be a useful tool to evaluate possible improvements for the metal recovery processes, allowing the environmental assessment of the whole methods in accordance with ISO 14, 040/14,044 [52]. However, a few LCAs studies are present on these technologies, particularly on lab scale [53]. The lack in this topic was also found by Kouloumpis and Yan [54], that conversely analysed the environmental impact of co-processing of coal mine and electronic wastes, demonstrating a great reduction in the toxicity and an enhancement of the climate change impact with respect to common treatment. Despite the growing number of studies following this approach, some obstacles such as data availability and ambiguity still limit its diffusion; however, it is desirable to overcome these problems, and associate LCA with other approaches such as machine learning and simulation to assist the development of interesting solutions for metal recovery.

4. Conclusions

Due to the growing WEEE production, the development of alternative sustainable and cost-effective solutions for metal recovery is urgent. The conventional methods, despite well-established on industrial scale, have different limitations such as the high environmental impact, the high costs and the low selectivity. Among the emerging strategies, the use of greener leaching agents and the development of bio-based systems are the most interesting.

Nevertheless, irrespective from the adopted methodologies, a rational approach should be applied when a new alternative solution is proposed, as well as when a PI is suggested. As briefly described in the Fig. 2, a proper design of experiment (DoE) is crucial to recognise the most affecting parameters and to optimize the process starting from a lab scale. Moreover, life cycle assessment (LCA) should be employed to evaluate the environmental feasibility of the new proposed technologies, identifying the critical points and possible modifications. Finally, the techno-economic evaluation, even on a lab and pilot scale, is decisive

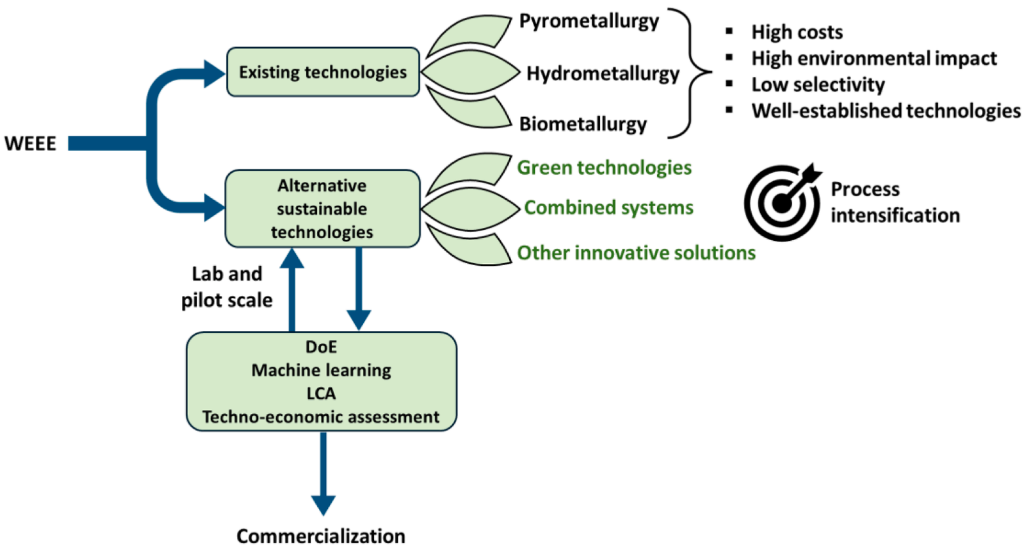

Fig. 2. Schematic approach for the development of new technologies for metal recovery.

to understand the applicability of the system on commercial levels. Through the use of these tools, future research can direct their efforts in overcoming current limitations in the metal recovery field, starting from laboratory scale up to industrial levels, by offering suitable solutions both from economic and environmental point of view.

CRediT authorship contribution statement

Marica Muscetta: Writing – original draft, Writing – review & editing.

Declaration of competing interest

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.

Data availability

No data was used for the research described in the article.

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