



Recent advances in the recovery of precious metals (Au, Ag, Pd) from acidic and WEEE solutions by solvent extraction and polymer inclusion membrane processes – a mini-review

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ABSTRACT

The growing demand for precious metals, such as gold, silver and palladium, and the depletion of primary metallic resources contribute to the search for new solutions that enable the recovery of these valuable materials from waste electrical and electronic equipment (WEEE), which is now treated as their secondary source. Conventional methods based on high-temperature pyrometallurgical routes are now giving way to hydrometallurgical processes due to their greater accuracy, ease of control and the possibility of planning the metal recovery processes at various scales. Scientists are particularly interested in the precious metal recovery methods based on the utilization of solvent extraction (SE) and polymer inclusion membranes (PIMs) processes, mainly because of their high efficiency and the possibility of using various chemical compounds as extractants and carriers. The article reviews the latest achievements in the applications of SE and PIMs for the recovery of Au(III), Ag(I) and Pd(II) from acidic solutions, including those obtained as a result of leaching WEEE. The limitations and advantages of SE and PIMs processes based on the use of both commercial and non-commercial chemicals are described.

Keywords: Electronic scrap; Metals recovery; Solvent extraction; Membrane separation

1. Introduction

In recent years, there has been a rapid increase in the production of various electrical and electronic equipment (EEE), with its increased miniaturization and affordability leading to the shortening of its lifespan [1–3]. Operational problems faced by the electrical and electronic equipment repair sector also have a major impact on the decreasing lifespan of EEE [4]. This has led to the generation of significant amounts of waste electrical and electronic equipment (WEEE) [5,6]. WEEE includes but is not limited to worn-out integrated circuit (IC) manufacturing and computer industry products, printed circuit boards (PCBs), connectors,

wiring, metal housings, as well as batteries and fluorescent lamps [7–10].

WEEE contains many metals, both hazardous ones that can be released in case of improper storage or processing (e.g., mercury) and may pose a threat to human health and the environment, as well as precious ones such as gold, silver and palladium, whose recovery is beneficial for economic and environmental reasons alike [11–14]. Due to the significant and systematically increasing amount of EEE waste, as well as the limited quantities of precious metals in ores and problems related to their extraction, new solutions and methods enabling proper e-waste management and fast and efficient recovery of valuable metals from such waste

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are intensively sought in recent years [15,16]. WEEE management is relatively well performed across the European Union mainly thanks to the WEEE Directive 2012/19/EU [17], which sets high targets for the Member States for the collection, recycling and recovery of valuable materials from e-waste. For many other countries outside the EU, collection, recycling and recovery rates are lower (e.g., in African countries) and WEEE legislation is constantly evolving (e.g., in India) [18,19].

Technologies for recovering valuable metals from e-waste used today are primarily based on physical processes that separate metal and non-metal fractions based on differences in their properties (e.g., magnetic, electrical conductivity, density, etc.) and chemical, such as pyrometallurgical, hydrometallurgical and biotechnological methods [16,20]. While pyrometallurgical processes in which EEE waste is subjected to high temperatures (often exceeding 1,000°C), usually in specially adapted furnaces, have been of great interest to scientists for many years, their use is systematically declining due to the high costs and the large scale of such operations (typically large amounts of WEEE are introduced into the furnaces) [10,16]. Hydrometallurgical methods involve the use of leaching

agents (e.g., strong bases, acids, chlorine systems, etc.) and transfer of the desired components into a solution. Today, they are much more frequently used and modified than pyrometallurgical methods. Many researchers believe that hydrometallurgical methods will play a key role in future waste electronic and electrical equipment management processes due to their high accuracy, predictability, ease of control, and the possibility of planning the metal recovery process at different scales [21,22].

Nonetheless, the recovery of valuable metals from WEEE using hydrometallurgical methods is a complex process (Fig. 1), involving multiple unit operations ranging from disassembly, comminution, and physical separation of components to leaching, which enables the transfer of the desired substances to aqueous solutions [21,22]. The subsequent recovery of valuable metal ions from aqueous solutions is made possible by using solvent extraction (SE) processes or polymer inclusion membranes (PIMs), among others.

This paper aims to present recent developments regarding the feasibility of using SE and PIM processes to recover such precious metals as gold, silver, and palladium from acidic model solutions and solutions obtained by leaching waste electrical and electronic equipment (using

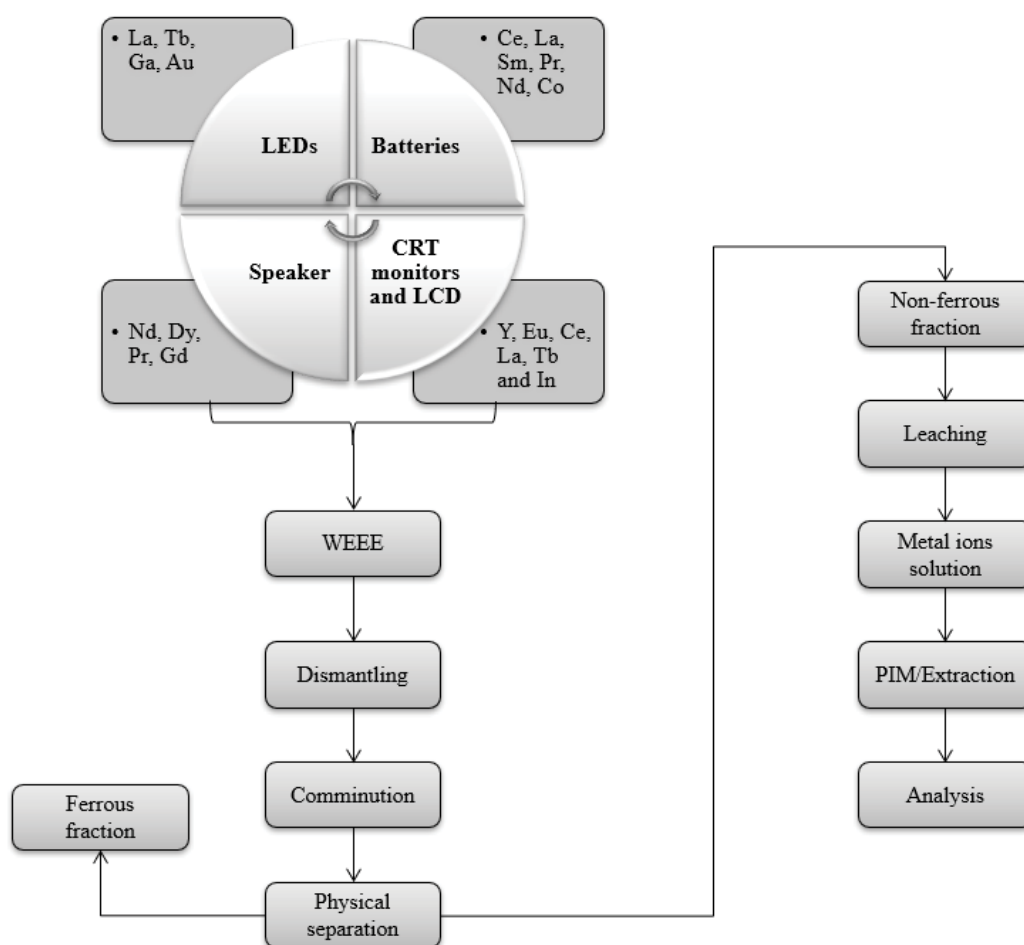


Fig. 1. Stages of WEEE proceeding for the recovery of valuable metals. Source: own elaboration based on [21,22].

acids or their mixtures) and to discuss the limitations and challenges of implementing such solutions on a larger scale.

2. Fundamentals of solvent extraction and polymer inclusion membrane processes

2.1. Solvent extraction

Based on the samples' physical properties, extraction can be divided into liquid and solid extraction. Liquid extraction involves transferring selected components from a sample solution (feed solution) to another solution (receiving solution). In contrast, solid sample extraction involves dissolving the desired components in an appropriate solvent and then separating the solution from the insoluble matrix components [23,24].

The main parameters describing the extraction process are the partition coefficient, distribution ratio and extraction percentage [25,26].

Partition coefficient K_D [Eq. (1)], where: $[A_{\text{org}}]$ is the ratio of activities of a solute in an aqueous/organic system will remain constant and independent of the total quantity of $[A_{\text{aq}}]$.

$$K_D = \frac{[A_{\text{org}}]}{[A_{\text{aq}}]} \quad (1)$$

Since the solute exists in different forms due to such processes as ionization, protonation, complexation, or polymerization, the value of the K_D partition coefficient does not depend on the solute's total concentration. Thus, in practice, the so-called D_M distribution ratio is used, as this value is determined by the experimental conditions [Eq. (2)] [25].

$$D_M = \frac{[M]_{\text{org}}}{[M]_{\text{aq}}} = \frac{[M]_{\text{feed}} - [M]_{\text{aq}}}{[M]_{\text{aq}}} \quad (2)$$

where $[M]_{\text{org}}$ – the concentration of metal ions in an organic phase (mol/dm³), $[M]_{\text{aq}}$ – the concentration of metal ions in an aqueous phase (mol/dm³), $[M]_{\text{feed}}$ – the concentration on metal ions in feed solution (mol/dm³).

The most vital parameter in terms of process efficiency is the extraction percentage – % E_M [Eq. (3)]. It depends on the distribution ratio and the volume of the phases and is expressed as follows [26]:

$$\%E_M = \frac{D_M}{D_M + \frac{V_{\text{aq}}}{V_{\text{org}}}} \times 100\% \quad (3)$$

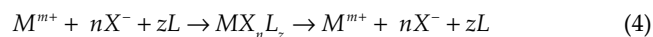
where V_{aq} – volume of an aqueous phase (dm³), V_{org} – volume of an organic phase (dm³) ($V_{\text{aq}} = V_{\text{org}} = 1$).

2.2. Polymer inclusion membrane process

Polymer inclusion membranes (PIMs) contain a polymer, carrier, and typically also a plasticizer, which are dissolved in an appropriate organic solvent; once they

are poured onto a glass ring and the solvent evaporates, they form thin, stable and flexible films. The plasticizer is responsible for improving the membrane's mechanical properties. Membranes are usually conditioned in distilled water to ensure their homogeneity [27]. Numerous studies have shown that polymer inclusion membranes can serve as an alternative to traditional solvent extraction during metal ions recovery processes [28,29]. For PIM, metal ion transport depends primarily on the carrier contained in the membrane. The carrier is most often an ion exchanger or a complexing compound [30]. On the other hand, the rate of metal ion transport by PIM is a more complex process and depends on many parameters, for example, the polymer matrix type, carrier concentration, plasticizer amount, thickness and surface morphology of the membrane, as well as the composition of the feed and receiving solution [31]. The transport of metal ions by PIM occurs through the facilitated transport mechanism, which involves the ion exchange of metal ions and counterions on the membrane surface. A distinction is made between co-transport, in which both the metal ion and the counterion move in the same direction, and counter-transport, in which the metal ion moves in the opposite direction to the counterion [32] (Fig. 2). Counterions (X^-) are ions that can de-diffuse and be exchanged for another ion. The diagram below shows the mechanisms of metal ion (M^{m+}) transport across a polymer membrane.

The metal ion (M^{m+}) and the counterion (X^-) are extracted with an inert carrier (L) into an organic phase, that is, the polymer inclusion membrane, forming a complex compound (MX_nL_z) that diffuses through the PIM. This is followed by a re-extraction process during which the metal ion and counterion are transferred to the receiving phase [Eq. (4)].



The carrier molecule (L) released in this way diffuses back into the feed phase to extract further ions from the solution. The extraction and re-extraction processes occur in succession until the system attains chemical equilibrium.

Co-transport (A) involves the simultaneous transport of a metal ion (M^{m+}) and a counterion (X^-) across the membrane from the feed phase to the receiving phase. When an inert carrier is used, the process's driving force is the variation of the partition coefficient of the transported substance and its complex formed with the carrier at the feed phase/membrane and membrane/receiving phase interfaces. The released applied carrier molecule diffuses back toward the feed phase to extract more metal ions until a complete chemical equilibrium is attained. The facilitated transport (B) of metal ions involves the formation of a hydrophobic complex between a carrier molecule (L) and a metal ion (M^{m+}), which then diffuses through the membrane phase into the receiving phase, thereby releasing the carrier molecule (L). When acidic carriers are used, the transport of metal ions occurs according to the counter-transport mechanism (C). The counter-transport mechanism involves the transport of metal ions from the feed phase to the receiving phase across the membrane while hydrogen ion transport occurs in the opposite direction. At the feed phase/membrane interface, the metal ion forms a complex compound – ML_n with the deprotonated form

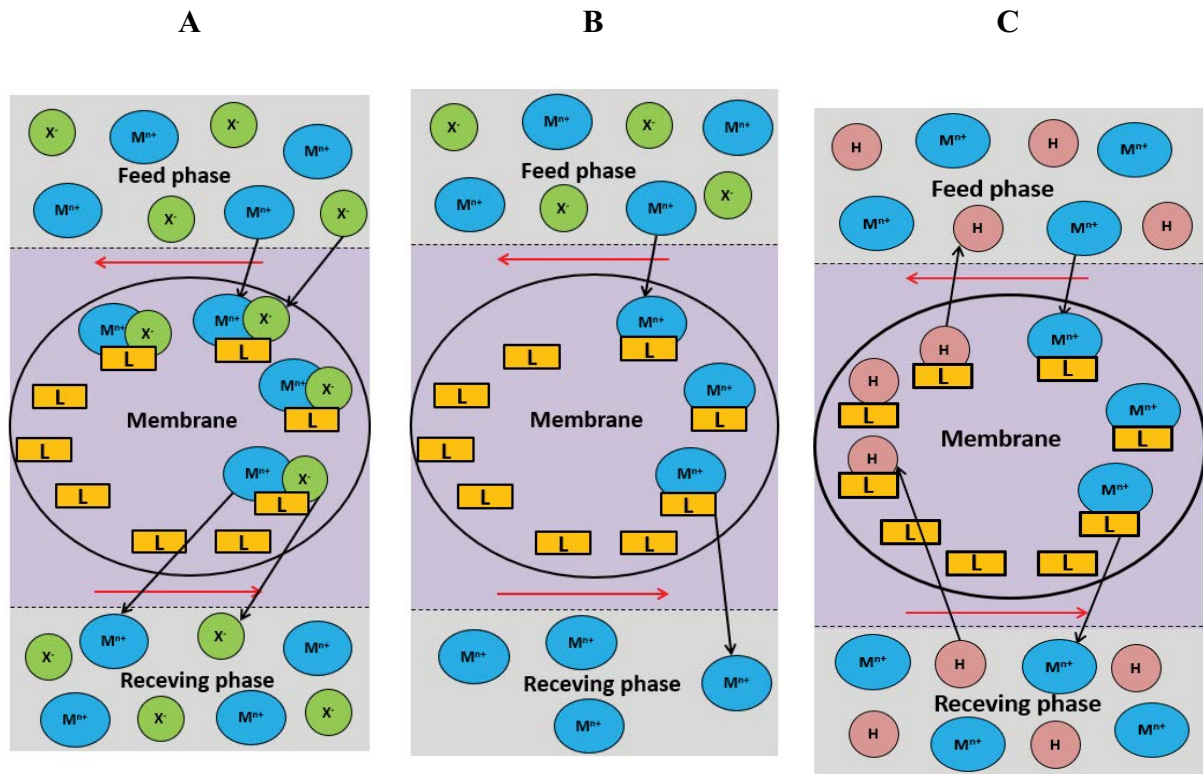


Fig. 2. Mechanisms of metal ion transport across polymer inclusion membranes, where: A – co-transport, B – facilitated transport, C – counter-transport, L – carrier, X⁻ – counterions, Mⁿ⁺ – metal ions, H – hydrogen ion. Source: own elaboration based on [20,33–35].

of the carrier, and after the diffusion of the complex compound at the membrane/receiving phase interface, a process of re-extraction of the metal ions into the receiving phase occurs with simultaneous uptake of hydrogen ions present in this phase. The counter-transport mechanism's driving force is the difference in the values of the partition coefficients, formed by maintaining an appropriate pH gradient between the feed and receiving phases [20].

The primary parameters describing the process of transporting ions through the polymer membrane are the initial flux of metal ions – J_0 , the $S_{M1/M2}$ selectivity coefficients represented by Eq. (8) (where $J_{0,M1}$ and $J_{0,M2}$ represent the initial flux for M1 and M2 metal ions, respectively).

$$S_{M1/M2} = \frac{J_{0,M1}}{J_{0,M2}} \quad (8)$$

The most important parameter determining the efficiency of the process transporting metal ions across the membrane is the recovery of metal ions from the feed phase to the receiving phase Eq. (9):

$$RF = \frac{c_i - c}{c_i} \times 100\% \quad (9)$$

where c_i – initial concentration of metal ions in the feed phase (mol/dm³), c – metal ion concentration after the period of time (mol/dm³).

3. Recovery of gold(III) ions

Because of its value and excellent properties (non-corrosion, high electrical and thermal conductivity), gold is often recovered from WEEE [36,37]. Currently, Au is recovered from e-waste using such methods as leaching processes, as well as solvent extraction and membrane separation. Leaching processes are carried out using various solutions, such as aqua regia, which is a mixture of HCl

The initial metal ions flux J_0 is defined by Eq. (5), where P is the permeability coefficient and c_0 is the initial metal ions concentration in the feed phase (mol/dm³).

$$J_0 = P \cdot c_0 \quad (5)$$

$$P = \left(\frac{V}{A} \right) \cdot k \quad (6)$$

where A – the effective membrane area (m²), V – the volume of feed phase [m³].

The reaction rate constant k (s⁻¹), takes on different values depending on the order of the reaction; for example, for a first-order reaction, it is described by Eq. (7).

$$\ln \frac{c}{c_0} = -kt \quad (7)$$

and HNO_3 (3:1; Au(III) recovery over 99%) [38], sodium thiosulfate solutions (0.12 and 0.2 mol/dm³, gold recovery of 70% and 75%, respectively) [39], and 65% HNO_3 solution (recovery of ca. 82% Au(III) [40]. The solvents used in leaching processes are typically neither cheap nor environmentally inert, and as such, other methods are being developed to reduce the volumes of solvents used and limit pretreatment processes [41]. For example, gold can be easily recycled from the gold-plated layer of waste mobile phone (WMP) PCBs using a DMF-CuCl₂-CaCl₂ (DMF: dimethylformamide) system called “mild aqua regia” with no pretreatment or enrichment processes required; additionally, the reaction system can be used recurrently [42]. Wu et al. [43] utilized the process of spontaneous reduction of gold by electroactive aniline polymers during hydrometallurgical leaching (with the mixture of concentrated HCl and HNO_3) of e-waste. They reported that by coupling the metal reduction process with an increase in the intrinsic oxidation state of the polymer and the subsequent re-protonation and reduction of the intrinsically oxidized polymer to the protonated EM salt (emeraldine salt), spontaneous and sustainable gold recovery from acidic solutions can be achieved without utilizing extensive metal-extracting reagents and external energy input. Research is also being conducted to increase the selectivity of the processes of recovering precious metals from waste. Tuncuk [44] developed a method to recover Au(III), Ag(I) and Cu(II) ions from WEEE based on the use of several oxidizing agents (i.e., 2% iodine, 3% hydrogen peroxide) and a two-stage leaching process that enabled efficient and selective recovery of gold (99.98%) and silver (96.90%) ions. New methods for recovering metals from e-waste are typically designed to be eco-friendly, which often requires hybrid designs or combinations of several different methods [45].

When recovering gold(III) ions from a solution obtained from e-waste leaching, the selection of the correct extractant and the conditions under which the extraction process is carried out are vital [46]. The used extractant must enable the selective transport of Au(III) ions from an aqueous mixture of metals into a hydrophobic (organic) phase through the coordination or supramolecular chemical recognition under properly chosen conditions [47]. Choosing the right extractant is not a simple process and the use of different compounds may lead to significantly different results even in cases where their structures are similar. For example, Doidge et al. [48] researched the recovery of gold ions from waste smartphones and the use of simple primary, secondary, and tertiary amides as reagents that selectively transport gold from aqueous to organic phases in solvent extraction experiments. They reported that while the strength of the extraction of gold from single-metal solutions is ordered 3° > 2° > 1° (where 3°, 2° and 1° stands for tertiary, secondary and primary amides, respectively), the 3° and 2° amides are ineffective at transporting gold from mixed-metal solutions due to the formation of a third phase [49]. New chemical compounds that can be used as gold extractants are constantly being sought. Oshima et al. [50] compared the extraction behaviour of Au(III) from acidic chloride media using cyclopentyl methyl ether (CPME) with processes conducted using such commercial extractants as dibutyl carbitol (DBC) and

methyl isobutyl ketone (MIBK). They reported that while the extractability of CPME was relatively low compared to DBC and MIBK, CPME was advantageous in the stripping and reductive recovery of the extracted Au(III). Alzate et al. [51] used optimized quantities of ammonium persulfate to extract non-leaching gold from WEEE and reported that $[(\text{NH}_4)_2\text{S}_2\text{O}_8]$ in aqueous oxygen enriched media could be used to extract superficial gold with an efficiency exceeding 98%. Campos et al. [52] developed a two-stage method for recovering gold from HCl solutions (such solutions can be obtained during the leaching of e-waste). The first step involved liquid/liquid extraction using toluene and hexane, whereas the second step included gold recovery with the use of tetra-alkyl phosphonium chloride (Cyphos IL-101) upon immobilization in a biopolymer composite matrix. Their results indicate that the resins could be re-used for at least 4 cycles and are specially adapted for the recovery of gold from low metal concentrations.

Using polymer inclusion membranes is a common solution for recovering gold(III) ions from e-waste. The primary factor that defines PIM capabilities is the carrier and the membrane's separation properties depend on its selection. One well-known commercial carrier is the Kelex 100. Due to its chelating ability, Kelex 100 enables the recovery of not only Cd(II), Pb(II), and Ni(II) ions [53,54] but also gold(III) ions. De San Miguel et al. synthesized a polymer inclusion membrane containing 32% w/w of Kelex 100, serving as the carrier, as well as 18% w/w of CTA (matrix) and 50% w/w of NPOE (plasticizer) for the transport of gold(III) ions from concentrated chloride solutions. The researchers have demonstrated the membrane's ability to recover approximately 85% of the gold, as well as the possibility of using it for fifteen 6 h cycles without the loss of its transport capability. Studies on the recovery of gold ions from concentrated hydrochloric acid media with the use of Kelex 100 in hybrid membranes containing hydroxyl-terminated poly(dimethylsiloxane) with (3-aminopropyl) triethoxysilane were also conducted. The results have shown that this method makes it possible to recover 70% of the gold [56]. Phosphonium ionic liquids are of great interest to researchers as well. Apart from their use in traditional extraction processes (recovery of d-electron metal ions and lanthanides [57–59]), compounds such as Cyphos IL-101, 102, 104, etc. are often used in polymer inclusion membranes for the recovery of transition metal ions, for example, V(V), Zn(II), Cd(II), Co(II), and Ni(II) [26,60–62]. Phosphonium ionic liquids have also been used to separate gold ions from e-waste. Bonggotgetsakul et al. [63] used PIM containing 30% w/w of Cyphos IL-104 as a carrier, and 70% w/w of PDVF-HFP as a matrix for the recovery of gold(III) ions from a solution obtained by leaching electronic scrap with aqua regia. Based on performed experiments, they determined the stoichiometric formula of the Au(III)/Cyphos IL-104 complex, $([\text{P}]^+[\text{AuCl}_4]^- \text{H}^+[\text{PO}_2]^-)$, where $[\text{P}]^+$ is the phosphonium cation and $[\text{PO}_2]^-$ is the phosphonium anion, and also concluded that the proposed PIM-based technology is an attractive alternative to conventional solvent extraction methods since it eliminates the use of diluents which are often toxic, volatile and flammable. Polymer inclusion membranes with different concentrations of Cyphos IL-104 as the membrane

extractant/carrier, were also examined for their ability to extract Au(III) from hydrochloric acid solutions. It has been reported that a PIM consisting of 25% w/w of Cyphos IL-104, 5% w/w of 1-dodecanol, and 70% w/w of PVC exhibited the best performance in terms of extraction rate and the amount of Au(III) extracted (recovery of 95% Au). Nonetheless, because of the loss of 1-dodecanol from the PIM to the aqueous solutions, the membrane was suitable only for a single efficient recovery of Au(III) from e-waste hydrochloric acid solutions [64]. Another carrier used in PIMs applied in the recovery of gold ions from e-waste is dm-3N-[N,N-di(2-ethylhexyl)aminocarbonylmethyl]glycine (D₂EHAG). Kubota et al. applied the extracting organic solution and the PIM containing the synthesized D₂EHAG extractant for the selective recovery of gold from synthetic (model) and real leachates obtained from e-waste (leaching with aqua regia). They reported, that PIMs with D₂EHAG exhibited high selectivity for the gold(III) ions (recovery of 96% of the gold) over the other metal ions present in much higher concentrations in the leachates [65]. Bonggotgetsakul et al. [28] used a polymer inclusion membrane containing 20% w/w of the Aliquat 336 commercial carrier and 2-nitrophenyl acetyl ether, 1-hexanol, 1-acetanol, 1-decanol, 1-dodecanol, or 1-tetradecanol as a modifier to recover gold ions from hydrochloric acid solutions. Their results show that gold recovery depends on the type of modifiers used and that the Au(III) extraction rate increases significantly when sonification is employed, as a result of the physical effects generated during acoustic cavitation, which eliminate the aqueous stagnant diffusion layer at the membrane/solution interface. Recently, Campo-Cobo et al. [66] applied salen-type ligands with electron-accepting substituents on the aromatic ring as extractant agents in polymer inclusion membranes for the extraction of different metal ions (Cu²⁺, Pb²⁺, Ca²⁺, Al³⁺, Co²⁺, Au³⁺, Fe²⁺, Fe³⁺, Ni²⁺, Mn²⁺, Zn²⁺, Au³⁺) from aqueous solutions. They reported that the examined Salen derivatives had higher selectivity to gold than to other metals and enabled the extraction of acceptable percentages of gold. The experiments were conducted in model solutions with low pH that was controlled using HNO₃, nonetheless, applying this method in the recovery of gold from real solutions obtained by e-waste leaching requires further research.

4. Recovery of silver(I) ions

Several methods have been developed for the recovery of silver(I) from waste electrical and electronic equipment, such as electrochemical processes [67] or adsorption processes [13]. However, many of these solutions have drawbacks and limitations that lead researchers to seek more effective methods. For instance, the application of cyclic voltammetry technique for gold and silver electrodeposition from synthetic solutions on graphite and copper electrode allows for removal of 97% of silver, but the metal deposits have not enough adherences on the electrode material [67]. Another solution is leaching of e-waste or a combination of leaching methods and other techniques, such as emulsion liquid membranes (ELM). In the latter case, as reported by Laki et al. [68], leaching first transfers silver from catalyst particles to an aqueous solution, and

then an ELM extracts silver ions from the leaching solution in a single separation step. The use of leaching methods for the recovery of silver from WEEE, depending on the leaching reagent used and the source of the e-waste, allows the recovery of about 50%–99% of silver(I) ions [11].

Often, recovery processes are conducted using solvent extraction and membrane separation. Much of the research involves extraction processes carried out on model solutions, the results of which can be used in the future to recover silver from real WEEE after leaching. For instance, Shimojo and Goto [69] performed solvent extraction and stripping of silver ions in room-temperature ionic liquids (RTILs) containing calixarenes and reported that pyridinocalix-[4]arene showed a high extraction ability and selectivity for silver ions (90% silver recovery). Their results highlight the great potential of calixarenes as extractants in RTILs systems. Due to the high efficiency and selectivity of the process, silver ion recovery studies are increasingly being conducted using polymer inclusion membranes. For example, Nowik-Zajac et al. [70] conducted Ag(I) recovery studies from aqueous nitrate source phase using polymer inclusion membranes, which contained derivatives of calixpyrroles with methyl (KP1) and carboxyl (KP2) groups, as ion carriers, o-nitrophenyl pentyl ether (o-NPPE) as a plasticizer and cellulose triacetate (CTA) as support. Their results indicate that the competitive transport of Ag(I) from the aqueous nitrate source phase through KP1 and KP2 is an effective separation method for Ag(I) ions and allows the recovery of 98.8% of silver ions from solution. Recently polymer inclusion membranes with different concentrations of a calixpyrrole ester derivative as the membrane carriers have been studied to determine their ability to transport Ag(I) from aqueous nitrate solutions. Authors reported that the effectiveness of the transport process across the membranes depends on the concentrations of ion carriers and metal ions, the pH of the source aqueous phase, and stripping agents [71]. Radzimska-Lenarcik et al. [72] applied polymer inclusion membranes (PIMs), which consisted of cellulose triacetate as a polymeric support, o-nitrophenyl pentyl ether as a plasticizer, and either 1-hexylimidazole (1) or 1-hexyl-2-methylimidazole (2) as an ion carrier for the separation of silver ions from model solutions and after battery leaching (silver-oxide battery). They found that Ag(I) is more easily transported through PIMs doped with (1) in the case of the model solution. In the case of the leaching solution after 24 h transport, the recovery coefficient of Ag(I) for PIM doped with (1) was 86%, and for PIM doped with (2) 47%. Examples of the use of PIMs with different carriers for the recovery of silver ions from model solutions show the high efficiency of these methods, which also exhibit high selectivity towards Ag(I) ions. In order to apply these solutions to the recovery of silver ions from real solutions obtained from WEEE leaching, further research is needed to determine the exact conditions conducive to the recovery processes or the type and amount of carriers and plasticizers.

5. Recovery of palladium(III) ions

Many different solutions have been used to date for the recovery of palladium(II) ions. Popular methods

include hydrometallurgical processes based on applications of various types of ionic liquid (IL) extractants, as understanding their extraction mechanisms and influencing factors is advantageous for further development in various industries, for example, wastewater treatment [73].

Cieszyńska and Wiśniewski [74,75] carried out extraction processes of palladium(II) from chloride solutions using two ionic phosphonic liquids, Cyphos IL-101 and Cyphos IL-104 (in the presence of toluene), as extractants and showed that extraction proceeds at the interface according to the anion-exchange mechanism. The results indicate that when these compounds are used, the extraction efficiency depends both on the hydrochloric acid concentration (the increase in HCl concentration has a negative influence on the extraction) and, to a lesser extent, on the temperature (increase in temperature causes a slight decrease in the palladium(II) extraction). Depending on the extraction conditions, from 54% to 97% of palladium (II) was recovered when Cyphos IL-101 was used and from 52% to 96% of Pd(II) when Cyphos IL-104 was used. The obtained results indicate that both Cyphos IL-101 and Cyphos IL-104 are very efficient and fast extractants [74,75]. Regel-Rosocka et al. [76] carried out solvent extraction and membrane separation of palladium(II) ions from aqueous chloride solutions using three phosphonium ionic liquids: Cyphos IL-101, Cyphos IL-104 and Cyphos IL-102, which was used for the first time as a metal ion carrier. They reported that Cyphos IL-102 also efficiently extracts palladium(II) ions both with liquid-liquid extraction and PIMs. However, similarly to results obtained previously for the application of Cyphos IL-101 and Cyphos IL-104 [74,75], increasing HCl content in the feed aqueous phase caused a decrease in extraction efficiency when Cyphos IL-102 was employed. Palladium (II) recovery using Cyphos IL-102 ranged from 84% to 90%, depending on process conditions. In addition, the results obtained with extraction using the discussed ionic liquids were significantly better than those obtained with polymer inclusion membranes. Fajar et al. [77] performed separation of Pt(IV), Pd(II), and Rh(III) by sequential transport through polymer inclusion membranes containing another ionic liquid, trioctyl(dodecyl) phosphonium chloride ($P_{8812}Cl$), as a metal carrier. Their results indicate that $P_{8812}Cl$ ionic liquid is an effective carrier in PIM for the recovery of Pd(II) ions, and that generated PIM exhibited a stable performance with multiple uses (after the seventh cycle, the recovery of palladium(II) was 96%). Additionally, recovery of palladium(II) ions from hydrochloric acid solutions can be performed by application of polymer inclusion membranes containing Cyanex 471X (triisobutylphosphine sulphide) as the ion carrier. Pošpiech [78] showed that the course of the process depends on Cyanex 471X concentration in the membrane and hydrochloric acid concentration in the source phase and potassium thiocyanate (KSCN) concentration in the receiving phase. Mixed ionic liquids such as $[C_8\text{bet}][Br][C_4\text{mim}][NTf_2]$ were also successfully used as a new extraction system to extract Pd(II) from multimetal-ion solutions. The obtained results indicated the existence of three different types of Pd(II) complexes and high palladium recovery (over 91%) after three cycles of experiments [79]. Recently Bourgeois et al. [80] have described a simple process for the recovery of palladium from printed

circuit boards waste. The solution obtained by leaching waste PCBs with nitric acid was processed through solvent extraction (with a solvent based on *N,N'*-dimethyl-*N,N'*-dibutyltetradecylmalonamide (BDMA), as an extractant). They reported that the extraction yield was 99%, volume of effluents generated during the solvent extraction sequence was small and no specific metal chelator was required for palladium stripping. Jha et al. [81] stated that "PIMs are one of the most promising techniques" because they are environmentally friendly (reduction of the amount of solvents), allow for selective separation of metal ions, simultaneous extraction and re-extraction, and show long-term stability. Table 1 shows structures of selected chemical compounds used for palladium recovery in classical extraction and polymer inclusion membrane processes.

The results of the use of extraction processes and polymer inclusion membranes presented in this section indicate that these methods can be useful in the recovery of palladium found in WEEE, such as PCBs, which is important both because of the limited access to natural resources (ores) and the need to reduce the amount of e-waste generated [82].

6. Summary

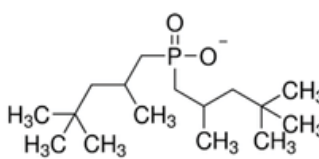
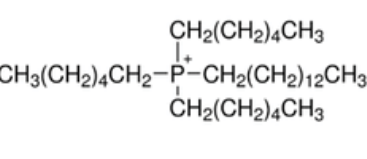
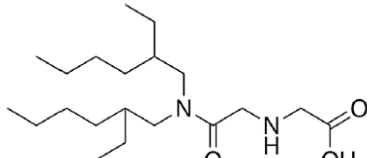
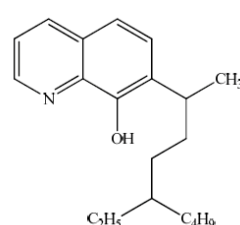
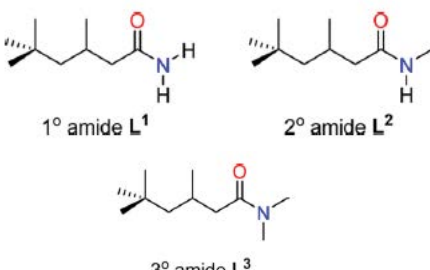
Due to the increasing development of civilization, more and more waste electrical and electronic equipment is produced, which contain among others materials made of gold, silver or palladium. Research conducted to date has proven that separation methods such as solvent extraction, and polymer inclusion membranes are effective when recovering valuable precious metals from waste. Currently, research is primarily focused on finding an effective extractant/carrier (Table 1) that will enable high recovery of precious metals from e-waste.

In summary, finding efficient extractors/carriers can significantly contribute to environmental quality by reducing landfill as well as the extraction of natural resources.

7. Conclusions

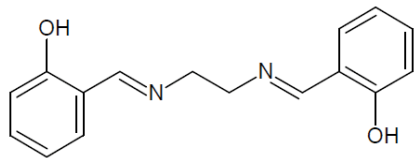
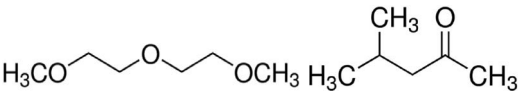
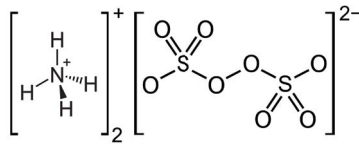
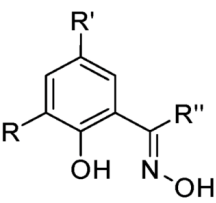
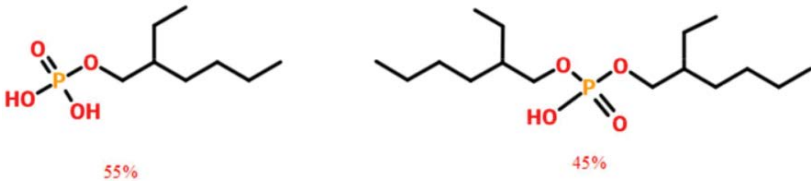
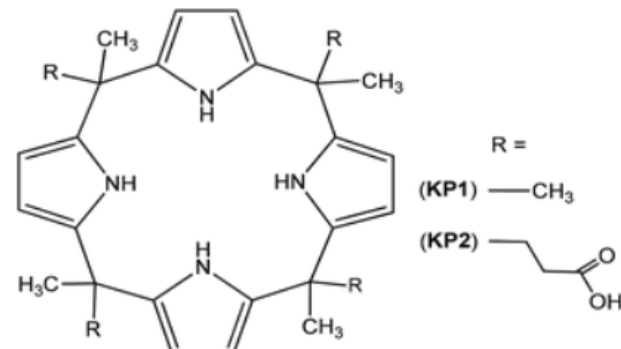
Waste electrical and electronic equipment is an important secondary source of precious metals such as gold, silver and palladium, among others, but the recovery of metals from WEEE using solvent extraction (SE) and polymer inclusion membrane (PIM) methods is a rather complex process influenced by many factors. Furthermore, before applying SE or PIMs, the WEEE leaching must first be carried out, for example, using acids or acid mixtures, resulting in acidic solutions. Therefore, the extractants used in the solvent extraction process must allow selective transport of recovered metal ions from acidic aqueous solutions containing a mixture of various metals into an organic phase. It should be emphasized that even relatively small differences in the structure of chemical compounds can significantly affect their metal ion chelating properties. Due to the variety of factors influencing the SE process, usually, the properties of a new extractant are evaluated using a less complex model solution, for example, containing only one/several metal ions, and only after determining the appropriate process conditions (e.g., pH, temperature,

Table 1
Review of extractants/ion carriers used in separation processes (SE, PIMs) for metals ions recovery

Methods of recovery of metal ions	Carrier/extractant	Metal ions
Polymer inclusion membrane	Cyphos IL-101 $\text{H}_3\text{C}(\text{H}_2\text{C})_5-\overset{\text{+}}{\underset{\text{(CH}_2)_5\text{CH}_3}{\text{P}}}-\text{(CH}_2)_{13}\text{CH}_3 \quad \text{Cl}^-$	Au(III) Pd(II)
	Trihexyltetradecylphosphonium chloride Cyphos IL-102 $\text{H}_3\text{C}(\text{H}_2\text{C})_5-\overset{\text{+}}{\underset{\text{(CH}_2)_5\text{CH}_3}{\text{P}}}-\text{(CH}_2)_{13}\text{CH}_3 \quad \text{Br}^-$	
Polymer inclusion membrane	Trihexyltetradecylphosphonium bromide Cyphos IL-104 $\text{CH}_3(\text{CH}_2)_4\text{CH}_2-\overset{\text{+}}{\underset{\text{CH}_2(\text{CH}_2)_4\text{CH}_3}{\text{P}}}-\text{CH}_2(\text{CH}_2)_{12}\text{CH}_3$ 	Au(III) Pd(II)
	Trihexyltetradecylphosphonium bis(2,4,4-trimethylpentyl)phosphinate D ₂ EHAG 	
Polymer inclusion membrane		Au(III)
Polymer inclusion membrane	N-[N,N-di(2-ethylhexyl)aminocarbonylmethyl]glycine Kelex 100 	Au(III)
	7-(4-ethyl-1-methyloctyl)-8-hydroxyquinoline 1°-, 2°- and 3°- amides 	
Extraction		Au(III)

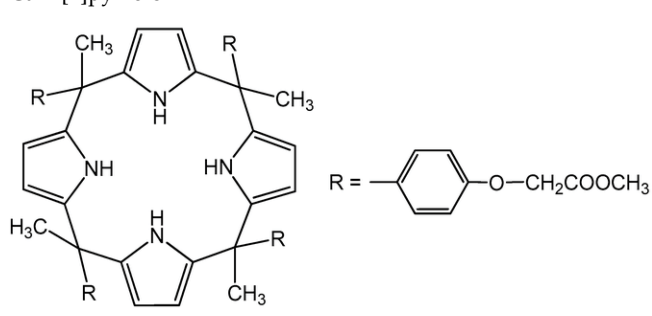
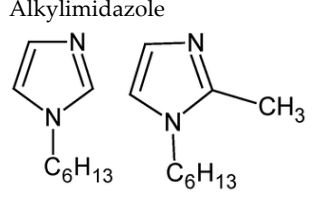
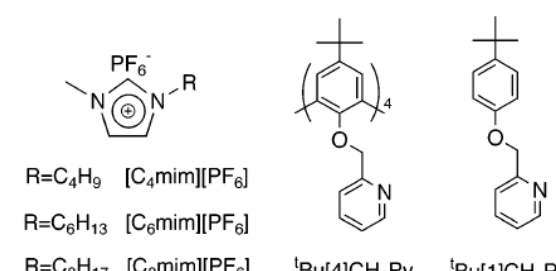
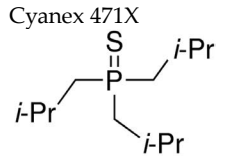
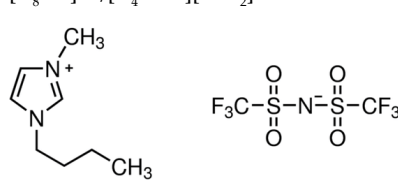
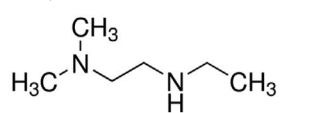
(Continued)

Table 1 Continued

Methods of recovery of metal ions	Carrier/extractant	Metal ions
Polymer inclusion membrane	Salen 	Au(III)
	N,N'-Bis(salicylidene)ethylenediamine DBC and MIBK	
Extraction	 Dibutyl carbitol and Methyl isobutyl ketone Ammonium persulfate (NH ₄) ₂ S ₂ O ₈	Au(III)
Extraction		Au(III)
Extraction	Phenolic oximes 	Au(III)
	MEHPA 	
Emulsion liquid membrane	2-Ethylhexyl phosphoric acid ester (a mixture of 55% mono and 45% diesters) Calixpyrroles 	Ag(I)
Polymer inclusion membrane		Ag(I)

(Continued)

Table 1 Continued

Methods of recovery of metal ions	Carrier/extractant	Metal ions
Polymer inclusion membrane	Calix[4]pyrrole  $R = \text{—C}_6\text{H}_4\text{—O—CH}_2\text{COOCH}_3$	Ag(I)
Polymer inclusion membrane	Alkylimidazole  1-Hexylimidazole and 1-Hexyl-2-methylimidazole	Ag(I)
Extraction	Calix[4]arene-bearing pyridine  $R = \text{C}_4\text{H}_9$ [C ₄ mim][PF ₆] $R = \text{C}_6\text{H}_{13}$ [C ₆ mim][PF ₆] $R = \text{C}_8\text{H}_{17}$ [C ₈ mim][PF ₆] ^t Bu[4]CH ₂ Py ^t Bu[1]CH ₂ Py	Ag(I)
Polymer inclusion membrane	Ionic liquid trioctyl(dodecyl) phosphonium chloride (P ₈₈₈₁₂ Cl)	Pd(II)
Polymer inclusion membrane	Cyanex 471X 	Pd(II)
Extraction	Triisobutylphosphine sulphide [C ₈ bet]Br/[C ₄ mim][NTf ₂] 	Pd(II)
Extraction	1-Butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide BDMA  N,N'-Dimethyl,N,N'-dibutyltetradecylmalonamide	Pd(II)

extractant concentration and others) tests are conducted on real solutions. Choosing the right extractant is not easy, as its use should also allow for a high level of precious metal recovery (above 90%) and not pose a threat to the environment. Currently, commercial extractants such as Cyphos IL-101, Cyphos IL-102, or Cyphos IL-104, DBC, as well as non-commercial extractants such as amides, or cyclopentyl methyl ether, among others, are used in the SE processes of WEEE solutions.

The use of PIMs allows for a significant reduction in the amount of solvents used during the recovery of precious metals from WEEE compared to solvent extraction and is therefore important for environmental and economic reasons. Proper carrier selection in PIM is critical, as is extractant selection in SE. Appropriate polymer inclusion membranes should recover precious metals at high levels and show selectivity for the metals recovered. It is also important that they can be used repeatedly, with only a slight decrease in performance from cycle to cycle. This paper presents examples of the application of various PIMs for the recovery of Au, Ag and Pd from WEEE, both containing commercial carriers (e.g., Cyphos IL-101, Kelex 100), as well as new ones, not used for this purpose so far (e.g., salen and its derivatives). Currently, due to the high efficiency of the process of recovering precious metal ions from WEEE, the simplicity of performing experiments, the relatively low cost of reagents/solvents and the need to use only a small amount of them, PIMs are considered by many researchers as one of the most promising techniques that are likely to play an essential role in the recovery of precious metals on a larger scale.

Symbols

K_D	–	Partition coefficient
A_{org}	–	Ratio of activities of a solute concentration of the metal ions in the an organic phase
A_{aq}	–	Ratio of activities of a solute concentration of the metal ions in the an aqueous phase
$\%E_M$	–	Extraction percentage, %
D_M	–	Division ratio determined experimentally
V_{aq}	–	Volume of the water phase, dm^3
V_{org}	–	Volume of the organic phase, dm^3
M_{org}	–	Concentration of metal ions in an organic phase, mol/dm^3
M_{aq}	–	Concentration of metal ions in an aqueous phase, mol/dm^3
V_{org}	–	Volume of an organic phase, dm^3
V_{aq}	–	Volume of an aqueous phase, dm^3
J_0	–	Initial metal ions flux, $\text{mol}/\text{m}^2 \text{ s}$
P	–	Permeability coefficient
c_0	–	Initial metal ions concentration in the feed phase, mol/dm^3
A	–	Effective membrane area, m^2
V	–	Volume of feed phase, m^3
k	–	Reaction rate constant, s^{-1}
RF	–	Recovery factor, %
c	–	Metal ions concentration in the feed phase at given time, mol/dm^3
c_i	–	Initial metal ions concentration in the feed phase, mol/dm^3

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