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An Investigation on Advances in Metal Extraction from Electronic Wastes by Supercritical Water and Carbon Dioxide

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ABSTRACT

Today, the application of supercritical fluid extraction (SFE) has been the focus of many researchers in various industries due to suitable operating conditions, environmental friendliness (no use of organic solvents) and high efficiency. In this process, a solvent is used for separation in supercritical conditions. Pharmaceutical, oil extraction, and oil and gas industries have conducted extensive research in this field. Electronic and electric devices are constantly being upgraded and updated due to the rapid advancement of science and technology, which creates a number of issues with handling electric and electronic waste (e-waste). The most significant issue is that it is challenging to safely dispose of halogen flame retardants and refractory polymers in e-waste. Supercritical fluid (SCF) techniques provide significant environmental benefits over previous disposal methods like pyrolysis and acid leaching since they pose no dangers for air or water contamination. This study discusses and provides a summary of the basic concepts and appropriate factors of supercritical fluid extraction (SFE). SCF methods were claimed to have recovered precious metals, base metals, and other inorganic minerals from e-waste with a recovery efficiency of further 93%. This study reviews the recent advances in supercritical water (SCW) and supercritical carbon dioxide ($SCCO_2$) extraction technologies for metal recovery from e-wastes. On the other hand, hybrid technologies are significantly improving in this field which could be considered for future studies.

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INTRODUCTION

Energy and electrical waste (e-waste) generally describes discarded, obsolete and broken electrical tools or devices. Electronic wastes are special wastes which have main parts include capacitors, transistors, ICs, electronic circuits, cathode and anode parts, and etc., and electrical wastes are special wastes which work with electricity and has sensitive electronic parts such as capacitors, transistors, ICs, and the like (Nandy et al., 2021). Electronic waste is one of the most important wastes of the 21st century, and an effective method must be found to manage this hazardous waste (Khan, 2016). In the last two decades, the production and consumption of electrical and electronic equipment (EEE) has widely increased. Along with this growth, obsolete and old products of the industry are increasing day by day. Nowadays, most governments are thinking of recycling these products in solid form. The continuous emergence of new technologies during the last 20 years makes old electrical and electronic equipment obsolete. The useful life of these equipment is shortening due to scientific development, more attractive designs and recovery and competitive issues (Hamari & Lehdonvirta, 2010). On the other hand, a huge flow of these equipment enters developing countries in the second stage. The separation

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of heavy metals from electronic components and their release in various environments (air, water and soil) will cause many environmental and health problems (Khan et al., 2021).

In the Middle East, the fastest consumer market for electronic waste is related to Iran. The per capita production of these wastes is globaly 6.1 kg/person per year (Arya & Kumar, 2020). In Iran, over 53 million mobile phones are active, and every year 3 to 4 million of them are out of order, and in general, the weight of out-of-order electronic devices is 4 million tons per year (Amiri et al., 2020). In 2022, P. Thakur et al., reported a 53.6 million tons of e-waste production over the globe (Thakur & Kumar, 2022). In this type of waste, elements such as lead, strontium, mercury, cadmium, nickel, arsenic, copper, zinc, barium, beryllium, all kinds of plastics, aluminum, gold, silver, platinum, dangerous chemicals from consumables, diphenyl poly-brominated ethers and rarely persistent organic pollutants or ozone-depleting halogenated compounds such as polychlorobiphenyls are found, which have harmful effects on the environment and human health (Khajouei et al., 2018; Beula & Sureshkumar, 2021; Nfor et al., 2022; Yaashikaa et al., 2022; Khajouei et al., 2023).

In order to the recycling of metals in electronic waste, traditional methods of pyrometallurgy and hydrometallurgy have not been developed due to high energy consumption, high cost and the production of a lot of environmentally destructive substances (Chu et al., 2022; Yang et al., 2022). Biometallurgical methods include biological, environmentally friendly, effectiveness, and in many cases, they have been evaluated as very capable in extracting metals, but they require a long reaction time and are very slow (Priya et al., 2021; Yaashikaa et al., 2022).

The use of supercritical fluids, due to the need for high pressure and temperature, has a high operating and initial start-up cost (because both the material and the thickness and the type of equipment will be special), the product produced by this method is more expensive compared to other traditional methods but it could be solved by optimization of the process (Chen et al., 2021). The most regular fluids used in this method are water and carbon dioxide, the use of water fluid is much more expensive than carbon dioxide (due to the high temperature and critical pressure of water compared to carbon dioxide) (Banchero, 2020). Nowadays, due to the fact that the product produced in this method is solvent-free and also the expansion of high pressure technologies in the last few years, the tendency to use this method is increasing day by day.

In order to compare different methods for treatment of e-waste and extraction of precious materials from it, bellow points could be noticeable:

- Systematic physical methods: High feasibility, ease of use, low environmental impact, and high yield of metal and non-metal material separation, unable to recycle rare metals with low concentration and dispersion in an efficient manner (Islam et al., 2020).
- Pyrometallurgy: advanced technology, straightforward operation, and recovering calorific value, huge amounts of smoke, dioxin, and other dangerous gases are produced, and various metals are merged together (Krishnan et al., 2021).
- Hydrometallurgy: Low equipment costs, simple operations, excellent recovery rates for scarce precious metals, and good target component selection, arduous procedure, significant chemical reagent use, and considerable effluent contamination (Khaliq et al., 2014).
- Biometallurgy: significant environmental friendliness, great selectivity of scarce valuable metals, complex operation, large time commitment (Bhat et al., 2012).
- SCF technology process: High effectiveness and productivity of oxidation or extraction, significantly higher rate of recovering of scarce metals, and environmentally friendly nature, higher operational needs and a small processing capability (Chakraborty et al., 2022).

The main aim of this review is to summarize recent advances in SCW and $SCCO_2$ extraction technologies for e-waste recovery with a look at hybrid technologies for this purpose.

SUPERCRITICAL FLUID (SCF)

Supercritical fluid is generally referred to as a fluid whose temperature and pressure conditions are above its critical temperature and pressure (Brazhkin et al., 2012). Basically, the characteristics that increase the importance and attractiveness of supercritical fluids in a special way are: pseudo-gas permeability, selectivity, continuously adjustable solubility and the possibility of complete removal at the end of the process (Wang et al., 2021; Dorieh et al., 2022). The combination of quasi-gas-quasi-liquid properties in supercritical fluids can be particularly useful and effective in many fields (Lin et al., 2015; Ma et al., 2022).

Carbon dioxide is more useful than other fluids in the supercritical extraction process. Being cheap, non-polluting and having simple critical parameters in industrial devices are the most important factors for the using of carbon dioxide as supercritical fluid more than other fluids. In the experiments that are carried out to obtain fine polymeric particles, supercritical carbon dioxide due to its liquid-like density that can be adjusted by pressure and gas-like penetration and completely low viscosity (1/10 of water), mostly is selected as a supercritical fluid (Shi et al., 2023).

The use of supercritical fluids to separate mixtures is one of the processes that its economic aspects added to the motivation for research on this field. Being needless to design a distillation process during the extraction of components is one of the aspects that leads to a reduction in energy consumption compared to liquid-liquid extraction operating methods (Al-Otoom et al., 2014). In addition to the economic aspects of using supercritical fluid in industries, this fluid phase is widely used in various industries due to its very suitable properties. When the temperature and pressure of a single-component biphasic mixture rise on the evaporation curve, the material reaches a state where there is no longer a liquid-vapor two-phase equilibrium, which is called the critical point. If the temperature and pressure rise above this point, the material will reach a state called the supercritical range, and the fluid in this range is called the supercritical fluid.

Table 1 illustrates the temperature and pressure of commonly used solvents at critical point. The properties of supercritical fluid near the critical point are completely dependent on temperature and pressure. Near this point, the density of the material changes with a slight increase in pressure, so that the isothermal compressibility for the supercritical fluid near the critical point has large values (Di Maio et al., 2021). The ability of a solvent to extract the components of a liquid mixture depends on factors such as the difference between the density of the solvent and the liquid mixture, the coefficient of fluid penetration in the mixture, the permeability of the components extracted in the solvent, the viscosity of the solvent and the surface tension between the two phases. When a fluid reaches a supercritical state, its physical

Table 1. Temperature and pressure of different fluids in critical point (Jessop & Leitner, 2008)

Solvent	T_c (K)	P_c (MPa)	Solvent	T_c (K)	P_c (MPa)
Acetone	508.1	4.70	Hexafluoroethane	293.0	3.06
Ammonia	405.6	11.3	Methane	190.4	4.60
Carbon dioxide	304.1	7.38	Methanol	512.6	8.09
Cyclohexane	553.5	4.07	n-haxane	507.5	3.01
Diethyl ether	466.7	3.64	Propane	369.8	4.25
Difluoromethane	351.6	5.83	Propylene	364.9	4.60
Difluoroethane	386.7	4.50	Sulfur hexafluoride	318.7	3.76
Dimethyl ether	400.0	5.24	Tetrafluoromethane	227.6	3.74
Ethane	305.3	4.87	Toluene	591.8	41.1
Ethylene	282.4	5.04	Trifluoromethane	299.3	4.86
Ethyne	308.3	6.14	Water	647.3	22.1

Table 2. Comparison of properties of supercritical fluids with gases and liquids at ambient temperature (Marcus, 2012)

State	Density (ρ/kgm^{-3})	Viscosity (η/mPa)	Diffusivity (10^6D/m^{2}s^{-1})
Gases, ambient	0.6-2	0.01-0.03	10-40
SCF, at T_c, P_c	200-500	0.01-0.03	~0.1
SCF, at T_c, $4P_c$	400-900	0.03-0.09	~0.02
Liquids, ambient	500-1600	0.2-3	0.0002-0.002

properties alternate between the physical properties of the liquid and the gas. Supercritical fluid is very close to liquids in terms of density, and this characteristic has made it exhibit very good solubility. Above the critical point, with increasing pressure and decreasing temperature, the density of the supercritical fluid increases, which can be used to increase or decrease the solubility of the supercritical fluid (Zhang et al., 2019b; Kim et al., 2021).

The viscosity of the supercritical fluid is close to the viscosity of the gases and allows it to flow as easily as the gases. In addition, the molecular diffusion coefficient of supercritical fluid is close to the molecular diffusion coefficient of gases, which causes that due to its quasi-liquid density, the separation rate in the supercritical state is multiplied (Pishnamazi et al., 2020; Di Maio et al., 2021). The penetration rate of supercritical fluid is 10 times faster than that of liquids. For example, the permeability of supercritical carbon dioxide varies between 3-10 cm^2/s to 4-10 cm^2/s, while the permeability of liquids is about 5-10 cm^2/s. High mass transfer rate is a significant and important property, so that the separation speed is limited by the mass transfer rate (Zhang et al., 2019a). Table 2 compares the properties of supercritical fluids with gases and liquids at ambient temperature. The surface tension of the supercritical fluid is very low like that of gases, so that due to the increase in the contact surface between the phases in the separation processes, the mass transfer rate increases. The quasi-liquid density and gas-like viscosity of the supercritical fluid, its good penetration coefficient and its low surface tension have caused the solubility of the supercritical fluid to be much higher than that of ordinary fluids (Esmaeilzadeh et al., 2009; McHugh & Krukonis, 2013; Chen et al., 2022).

SCF EXTRACTION

One of the successes of the 20th century is the evaluation of systems that can directly transform information and knowledge into technology and commercial products with high efficiency. The use of supercritical fluid to extract natural products is one of these systems that has played an important role in the development of the industry. Extraction with supercritical fluid is one of the new processes that has the ability to produce products without any effect of the presence of solvents and contaminated or synthetic materials. By using this technology, it is possible to extract heat-sensitive materials and compounds that are easily oxidized (Zhou et al., 2021).

This method is based on the gas/liquid-like characteristics of supercritical fluids. When approaching the critical point, the solubility of the fluid changes with changing temperature and pressure, and due to the changeability of the fluid solubility, the extraction is also adjustable (Oliveira et al., 2011). The extraction process using supercritical fluid and has salient features compared to other extraction processes, which can be listed as follows:

- By adjusting the temperature and pressure, the selectivity of the supercritical fluid can be controlled.
- Supercritical fluid is easier to transport due to its low viscosity.
- Reducing the operating pressure reduces the solubility in the supercritical fluid and the adsorbed material can be recovered with high quality and the solvent returned to the system

(Chen et al., 2022).

The procedure of supercritical extraction process is investigated and reported in the literature. In SCCO2 extration, the CO2 gas is the main component and it is convenient to use some ligands (such as Kelex 100 and Cyanex) and cosolvent (such as methanol and ethanol) in order to enhance the process (Temelli & Güçlü-Üstündağ, 2005; Fayaz et al., 2022b; Li et al., 2022; Moradi et al., 2022; Jung & Zhang, 2023).

Figure 1 illustrates the schematic of the device used to separate a target material by supercritical carbon dioxide. In the procedure, first, crushed feed is placed in the extraction part. The CO_2 contained in the cylinder is cooled with a condenser, and with the help of a high pressure pump, it reaches the operating pressure and after passing a heating unit, finally transfers to the extraction device, which is jacketed, so that it is always kept at the operating temperature. The fluid coming out of the extraction device expands to ambient pressure while passing through gas-liquid separation unit.

Golzary et al. (Golzary & Abdoli, 2020), were performed a research in order to improve the supercritical carbon dioxide extraction method's capacity to restore copper from printed circuit boards (PCBs) waste. To achieve this goal, supercritical water was used as a pre-treatment technique on PCB refuse to raise the copper content in the original solid feed. Methanol, ethanol, and propanol solvents, as well as the polyfunctional agents cyanide 301, 302, and tri-butyl phosphate, were employed to improve copper's solubility in Carbon dioxide. Figure 2 shows the procedure of this research.

ELECTRONIC WASTES (E-WASTE)

Among the most widely used circuit boards, we can refer to printed circuit boards (PCB). Mobile phones are the most important electronic waste due to their rich metal content and large volume of production and due to their short half-life (about 4 years) (Arshadi & Mousavi, 2015). According to reports, more than 70% of the value of mobile phones is related to the precious

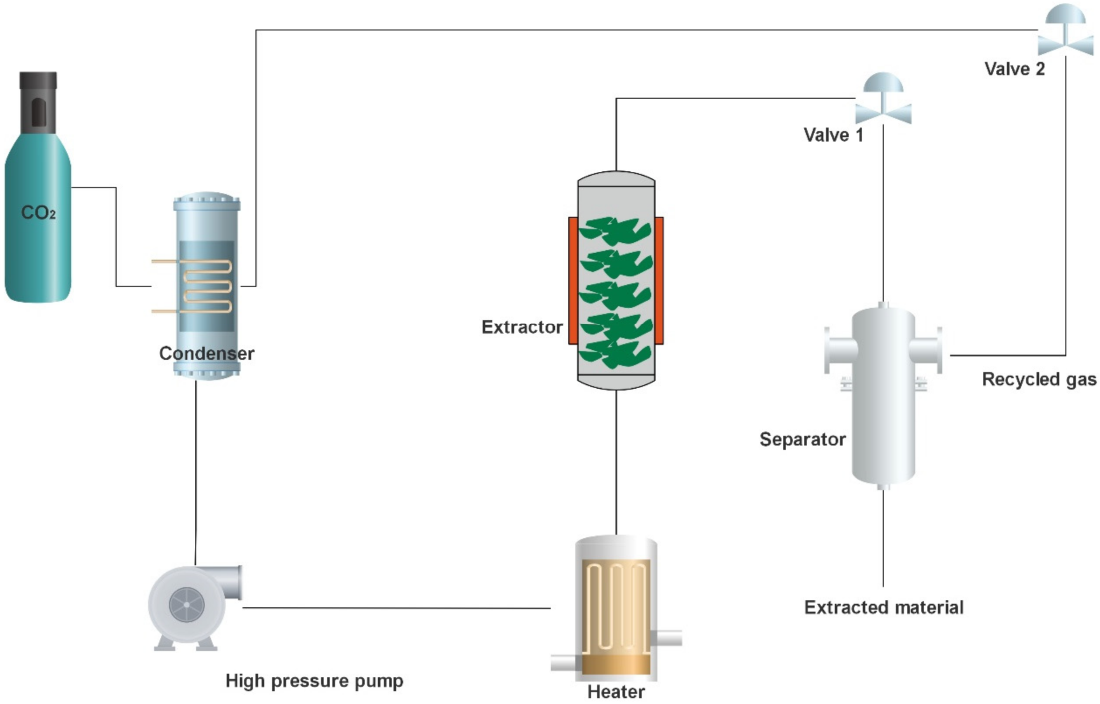


Fig. 1. Schematic for a typical supercritical extraction process

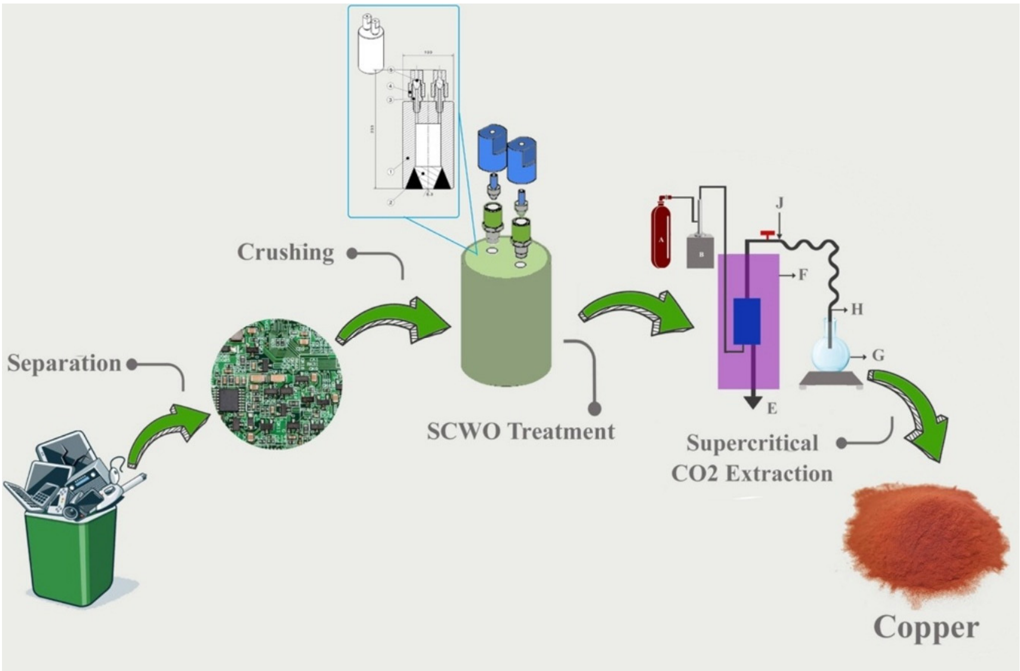


Fig. 2. Enhancement of copper extraction in SFE process (Golzary & Abdoli, 2020)

metals used in its range (Cui & Zhang, 2008). Printed circuit boards are a part of electronic waste, the main carrier of precious and dangerous metals (Willner & Fornalczyk, 2013). Printed circuit boards only make up about 6% of the total weight of electronic waste, but they are the most important carriers of valuable materials (Behnamfard et al., 2013). Printed circuit boards contain large amounts of dangerous substances such as lead, tin and cadmium along with valuable and precious metals such as silver, gold and copper. Computers are produced several times faster than other electronic waste. Considering that the half-life of each computer is between 2 and 5 years, according to estimates, about 17 million computers are retired every year (Askari et al., 2014). The amount of gold found in one ton of computer is more than the amount of gold found in 17 tons of ore (Yamane et al., 2011). On average, one ton of mobile phone without battery contains 130 kg of copper, 3.5 kg of silver, 340 grams of gold and 140 grams of palladium (Annamalai & Gurumurthy, 2021). Considering that 166 million mobile computers are being sold in annually, it is possible to understand what a huge amount of these metals are needed for their production (Woidasky & Cetinkaya, 2021).

The PCB is a fiber with a thickness of about 1.6 mm, made of compressed cardboard (phenolic fiber) which is brown and cream colored or made of glass wool fibers (fiberglass), on the other side of this fibers have a copper layer in the form of a thin sheet with a thickness of 0.25 mm, which is covered with the help of glue and pressure (Preddy et al., 2019). The printed circuit board includes a set of electric circuits that are designed and produced in the form of one layer, two layers or even multiple layers and are used in all electronic products, even the simplest ones. The global standard for the production of printed circuit boards is based on the Underwriters' Laboratories (UL) and Institute of Printed Circuits (IPC) standards. While the board connections only contain copper metal and no other metal is used in its structure, it is called a printed wiring board (PWB). The printed circuit board can be one-sided (one layer of copper), two-sided (two layers of copper) or even multi-layered (Kishore et al., 2021). A wide range of epoxy, ceramic, copper and aluminum materials are used in this type of boards. D.S. Achilias et al., introduced a solvent-based method to recycle polymers from electric and electronic wastes. The study subjected three types of polymers (polycarbonate (PC), poly(acrylonitrile-butadiene-styrene)

(PABS), and polystyrene (PS)). The dissolution/reprecipitation method was employed in a part of study and the results revealed that more than 90% recovery could be achieved in optimum conditions. Maximum recovery for PC from compact disc, PS from radio board and PABS from TV set was achieved in dichloromethane/methanol as solvent/nonsolvent at 50, 100, and 100 °C with 98%, 98%, and 96% recovery respectively in 30 min. Although using toxic solvents could lead to some environmental issues, short process time, mild temperature and ease of design and process could lead to an economic implementation which could be modified by environmental safety protocols (Achilias et al., 2009; Khajouei et al., 2021; Kol et al., 2022).

In PCB, the thickness of copper (micrometers or millimeters) can be used to set the amount used, but for ease of work, the weight of copper used on the surface (ounces per square foot) is used for measurement (Ritchey & Edge, 1999; Chen et al., 2022). Silver is considered one of the basic elements in electronic products and is used in a wide range of components and applications. The wide use of this element is due to its very good thermal and electrical conductivity. Silver is used in wires and printed circuit boards, cables, switches, relays, lead-free solders and electromagnetic motors (Suponik et al., 2021).

There are precious and toxic metals in electronic waste, so the way of managing this waste is somewhat different from traditional waste management. Currently, electronic waste is buried in incorrect ways in the world, which risks leaking harmful substances into underground water and soil (Askari et al., 2014); furthermore, by returning metals used in electronic waste, it can help preserve natural mines. Therefore, the recycling of electronic waste is not only important from the aspect of reducing environmental pollution, but also as a source of metal extraction, and it is possible to supply about 40 million tons of materials in this way (Hagelüken & Refining, 2008). The rapid growth and development of technology indicates the growth of demand for electrical and electronic equipment, for which printed circuit boards are critical components; Because these devices become obsolete after short periods. PCB is composed of ceramics, polymer and metals, especially copper with the highest percentage. To recover the metals in this waste, the traditional methods of pyrometallurgy and hydrometallurgy have not been developed due to high energy consumption, high cost and the production of a lot of environmentally destructive substances. Bio-hydrometallurgical methods include biological, environmentally friendly, effective and new, and in many cases, they have been evaluated as very capable in extracting metals.

Today, due to the progress made in various industries and the increasing demand for the purchase of raw materials with a high percentage of purity and with higher quality, extensive research has been carried out in the field of suitable methods of producing materials used in these industries and the optimization of these methods in order to increase efficiency. In some separation processes, it is not possible to use the usual distillation and extraction methods, which are mainly in terms of economic issues, because the physical and chemical properties of the materials as well as the required conditions are such that the necessary facilities for the use and application of the methods does not provide the usual satisfactory and high quality. The most important of these cases include high boiling point, closeness to the boiling point of the target material (formation of azeotrope; mixtures with "azeotrope" boiling point), sensitivity of materials to high temperature, and supply and recovery of solvent. As a result, most research and academic centers have paid attention to using processes with less energy consumption.

In the process of extraction with supercritical fluid, unlike liquid-liquid extraction, solvent recovery is done by sudden expansion (Figure 3), and there is no need for distillation to recover the solvent which reduces energy consumption. Another reason for expanding the use of supercritical fluids is the need for raw materials with high purity and selectivity in industries. For example, complete solvent recovery is very necessary in industries to prevent chemical solvent pollution, while in common methods such as distillation and liquid-liquid extraction, complete solvent recovery is not possible. Therefore, by replacing CO_2 gas as a solvent in

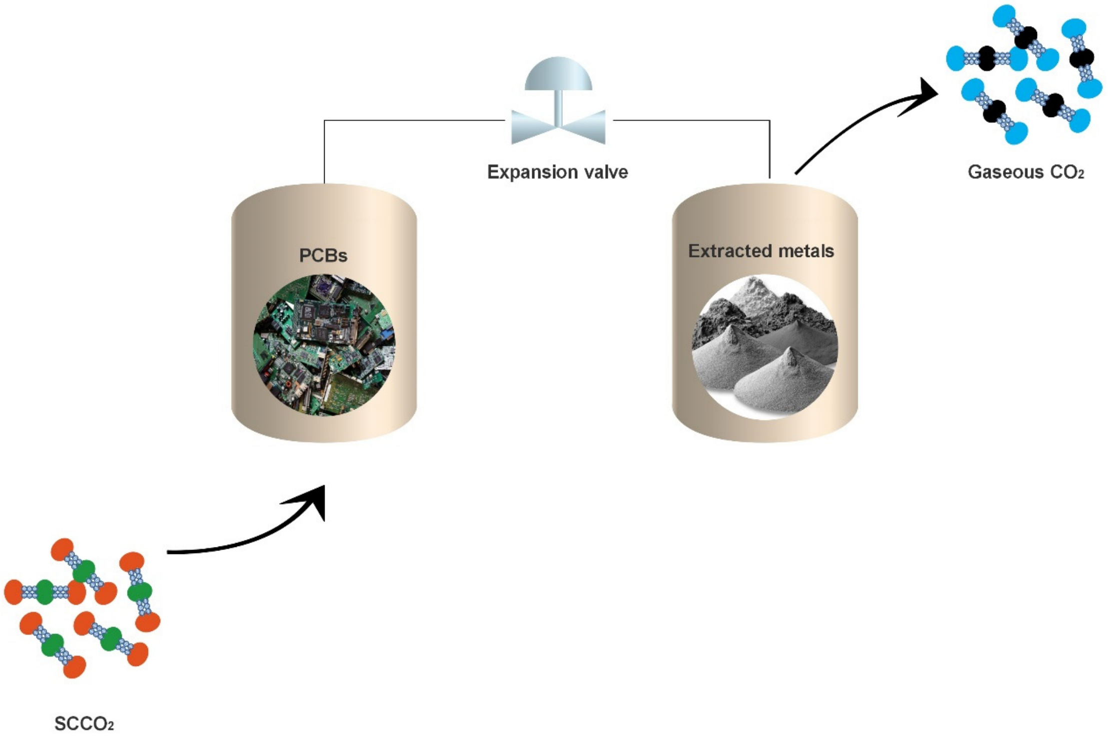


Fig. 3. Supercritical CO_2 extraction for PCBs (expansion unit)

supercritical processes, this problem has been solved.

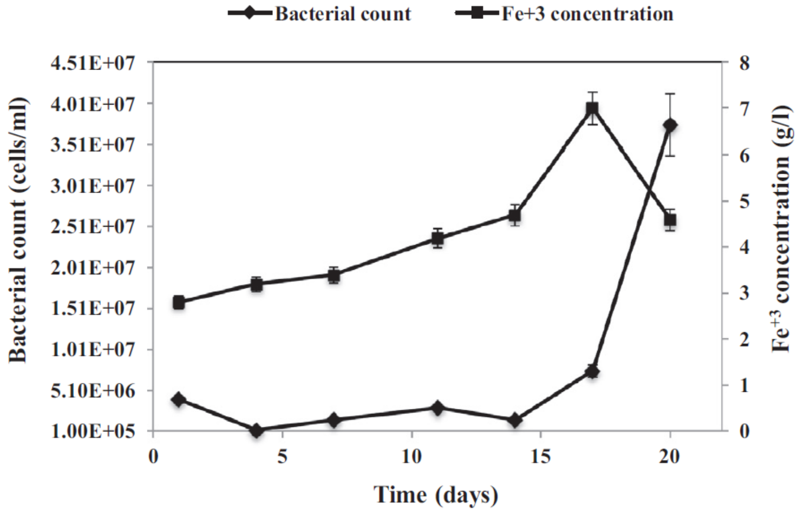
In the mentioned and similar cases that arise from economic considerations and practical and component limitations, a method for performing separation should be used or the possibility of using it should be checked that the method can meet most of the required conditions and the use of supercritical fluids is one of the most considerable options in this case. The obtained results show that the use of supercritical extraction method in extracting valuable metals from printed circuit waste has a very high yield and its superiority over traditional and conventional methods is more satisfactory from a commercial point of view.

LITERATURE REVIEW ON METAL EXTRACTION FROM E-WASTE

Traditional technologies

Hydrometallurgical removal has worked successfully to recover metals from electronic waste. Solvent extraction is the most important separation method from aqueous solutions. Several leaching agents such as nitric acid (Lee et al., 2004; Rajahalme et al., 2022), hydrochloric acid (Islam et al., 2020) and sulfuric acid (Rao et al., 2021) have been used for leaching electronic and electrical waste. But it should be noted that although sulfuric acid is very cheap, it has little selectivity (Lu & Dreisinger, 2013). In addition, in this type of solution, lead is precipitated as lead sulfate, and practically the possibility of recovering lead is lost. If hydrochloric acid solution is used, it is possible to form $PbCl_2$ deposits. Also, some copper is lost as $CuCl_{n-2}$ and has a negative effect on copper recovery. In nitric acid solution, many impurities such as silver will remain (Veglio et al., 2003). Fluoroboric acid has been used as a selective leaching agent to dissolve tin and lead in electronic waste (Spyrellis, 2009); the fluoroborate form of elements is more than their sulfate, sulfide, and chloride forms, and the possibility of better control of impurities increases with this acidic environment. Copper recovery from solvent extraction method has been studied with several types of extractors such as LIX860, LIX6022, LIX84, LIX64 and LIX984N (RW & IM, 2003). LIX984N has provided better results for extracting copper from chloride solution.

Arshadhi and Mousavi (Arshadi & Mousavi, 2014) used the pure culture of the acidophilic bacterium Acidothiobacillus ferrooxidans to recover copper and nickel from computer printed circuit boards. The main goal of this research was to provide solutions to increase the extraction of copper and nickel simultaneously by using software optimization methods on the parameters of pH, mass density, particle size and initial concentration of metal ions. The self-improvement stage started with an initial concentration of 1 g/l and reached a final concentration of 26 g/l after 80 days. In this research, using "Design Expert" software, the optimal values of initial pH, ferric ion concentration, mass density and particle size have been obtained respectively 3, 8.4 g/liter, 20 g/liter and 95 microns for simultaneous maximum extraction of copper and nickel. The authors indicated that: As shown in Figure 4(a), the concentration of Fe^{3+} ions increased



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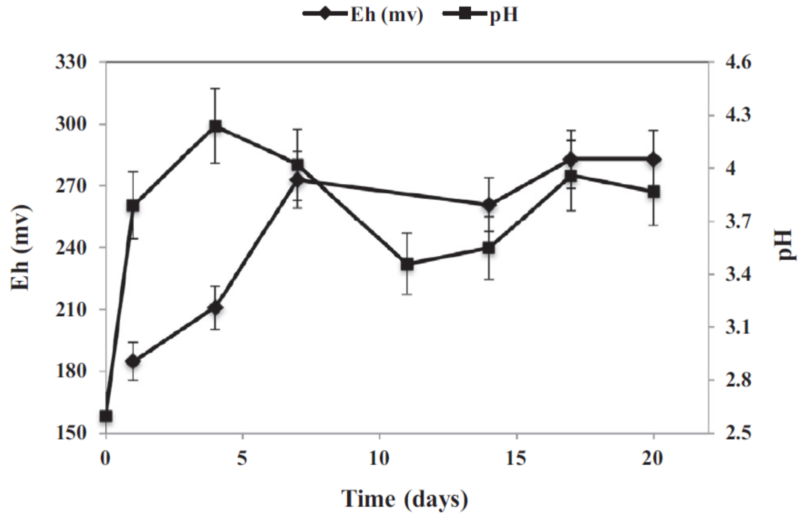

b

Fig. 4. Growth characteristics of A. ferrooxidans versus time at optimum conditions: (a) variation of bacterial count and Fe^{3+} concentrations; (b) variation of pH and Eh (redox potential) (Arshadi & Mousavi, 2014)

during 17 days, which is the result of increased bacterial activity and slow bioleaching. In 20 days, however, the concentration of Fe^{3+} decreased from 7 to 4.6, which indicates the highest amount of bioleaching in this area. Figure 4(b) shows that with the dissolution of PCB in alkaline solution and bacterial activity, the acid produced in the biological process has caused a drop in pH.

Also, a similar research was done on mobile phone printed circuit boards. In this research, all the test conditions were similar to the previous test and the only difference is in the type of prototype. The computer sample has an alkaline property due to the presence of more plastics, while the mobile phone sample has a neutral property. This seemingly small difference has produced different results (Cui & Zhang, 2008). In the computer sample, the results prove that in order to achieve large amounts of copper recovery, high values of mass density and pH are effective, while in the mobile phone sample, lower values of mass density and pH are effective, because the alkaline property of the computer sample means that in higher acidity, the possibility of sample dissolution and the common contact surface of bacteria increases. High pH and mass density mean that the industrial process becomes more economical (Arshadi & Mousavi, 2015).

In 2013, Willner used the pure culture of the acidophilic bacterium Acidothiobacillus ferrooxidans to recover copper from printed circuit boards. The purpose of this article was to investigate some physical and chemical factors on the bioleaching rate. The results of the study indicated that the pH range of 1 to 2 can improve copper recovery and iron oxidation by bacteria decreases rapidly at a pH higher than 2.5. Also, temperature is an important factor in the growth of bacteria and the most activity of this bacteria is in the temperature range between 28 and 37 degrees Celsius. Also, it was observed that at higher concentrations of the inoculating liquid, the leaching rate of copper accelerates. This study shows that during 19 days using 20, 50 and 90% of the inoculating liquid, more than 90% of copper can be recovered, and this indicates that biological extraction is more effective than chemical extraction (Willner, 2013).

In China, in 2014, the environmental effects of heavy metals released from electronic waste were investigated by Song and his colleagues. In this study, the heavy metals chromium, cadmium, copper and lead were examined. This study and similar studies show that in order to preserve the environment and prevent its pollution, it is necessary to control the release of heavy metals from printed circuits (Song & Li, 2014).

In 2014, Bizzo et al. investigated the recycling of heavy metals from used computer printed circuit boards. The boards had approximately 26% metal, most of which are copper, lead, aluminum, iron, tin, cadmium and nickel. Comparisons showed that the concentration of metals such as gold and silver decreased over time (Bizzo et al., 2014).

In 2013, Amfo-Otu et al., investigated the amount of lead, chromium, nickel, cadmium and zinc in the soil of electronic waste recycling sites in Nigeria using atomic absorption spectroscopy. The concentration of heavy metals in all investigated sites was higher than the control site. The results of this study showed that in the places of storage or disposal or recycling of electronic and electrical waste, this waste plays a role in the release of heavy metals in soil and water samples (Amfo-Otu et al., 2013).

Behnam Fard et al. (2013) extracted 85% of the copper in computer PCBs using 100 mL of 2M sulfuric acid and 25 mL of 35% hydrogen peroxide during a typical 3-hour leaching process (Behnamfard et al., 2013).

SCW and $SCCO_2$ technologies

In a research in 2013, Fu-Rong Xiu et al. used metal recycling from printed circuit board waste using supercritical water as pre-treatment along with acid leaching process, and super-critical oxidation and de-polymerization were investigated as pre-treatment. Using the supercritical water oxidation method as a pre-treatment at 420 degrees Celsius, 99.8% of copper and 80% of lead were extracted (Xiu et al., 2013). In this work, an high yield and compatible pretreatment was proposed to recycle metals from printed circuit board waste. The experimental results showed that the supercritical oxidation preprocessing technology is a promising method for the decomposition of organic materials and the enrichment of metals in the processing of printed circuit board residues. System pressure in supercritical conditions affects the reactivity of organic materials. The supercritical fluid used in the SCW process, which is water along with an auxiliary component, has enough power to destroy solid particles and create proper porosity in solid particles due to its high pressure. As a result of this effect and the creation of this porous structure, the penetration path for gases and liquids is easily provided and the polymerization and oxidation reactions are carried out at a high speed. In addition, the created porosity facilitates the leaching process to remove heavy metals that takes place in the next steps (Xiu et al., 2013; POURESMAEEL et al., 2016).

Leaching of Au, Ag and Pd from mobile phone printed circuit boards after supercritical water pretreatment was investigated in another study by Xiu et al. in 2015 (Xiu et al., 2015). In the process used in this research, the mobile printed circuit board was pre-treated using supercritical water, and in the next step, the leaching process was performed using Assad's hydrochloric acid to recover the copper metal on it. The experimental results showed that the temperature, time and pressure of SCWO (supercritical water oxidation) processing had a significant effect on the extraction of Au, Ag and Pd from the residues of printed circuit boards processed in (SCWO + HCl). The process developed in this study is believed to be environmentally friendly for the recovery of Au, Ag and Pd from mobile phone printed circuit board wastes by SCWO pretreatment combined with iodide leaching process. The iodide-iodide leaching process is performed without using cyanide and is usually implemented in the recovery of precious metals after supercritical and hydrochloric acid pretreatment.

This group continued to investigate the recycling of materials from printed circuit boards by supercritical methanol. The main focus of this study was on investigating the characteristics of solid products and oil obtained from waste electrical and electronic boards using supercritical methanol. Operating conditions were temperature range from 300-420 ^{o}C, processing time between 30 and 120 minutes and solid to liquid ratio (1:10-1:30 g/ml) to determine the products and mechanism of de-polymerization of printed circuit board residues in supercritical methanol. The results of the analyzes showed that these oils mostly contain phenol and its methyl products, which increase with increasing reaction temperature. Liquids also contain a notable number of phosphate compounds such as terphenyl phosphate, which decrease significantly with increasing reaction temperature. The main solid products obtained from this process included iron, copper, tin, zinc, lead, as well as silver and gold in lower concentrations (Xiu & Zhang, 2010).

In 2013, Sanyal et al (Sanyal et al., 2013) investigated the optimization of printed circuit board recycling using the supercritical carbon dioxide process and showed that the supercritical carbon dioxide process created a suitable environment that successfully separates the layers of the printed circuit boards. This study mainly focused on the reuse of materials in supercritical conditions with low temperature and pressure and comparing the data with previous studies. Techniques for detecting material properties such as dynamic mechanical analysis, differential scanning calorimetry, and Fourier transform infrared spectrometer were used to investigate the separation of different material layers. Based on the results of this study, it can be said that the base of printed circuit boards can be easily separated into its constituent materials, i.e. glass fibers, copper and polymers. The interesting result of this study was the stability of the physical and chemical properties (glass transition temperature, transverse density and FTIR spectrum) of the materials before and after the supercritical extraction process.

There is a research published in the field of rapid extraction of copper from printed circuit boards using supercritical carbon dioxide is by Calgaro et al. in 2015 (Calgaro et al., 2015). Developing a new technique to recover copper metal from printed circuit board scraps was the main goal of this study. The old cell phone was to obtain faster reaction kinetics using supercritical carbon dioxide scrubbing and solvent assistance. Estimates in these tests showed that copper is about 10-15% of the total weight of the mobile phone and 25-45% of the weight of the PCB. The kinetics of leaching reaction was performed at atmospheric pressure with H_2O_2 and H_2SO_4 as solvents using supercritical CO_2 (Azizi et al., 2023). The results showed that 34.83% by weight of printed circuit board residues had copper. According to the results, solvent extraction in supercritical conditions has a speed 9 times higher than extraction in atmospheric pressure. Almost 90% in the structure of the printed circuit board was recovered after twenty minutes using supercritical extraction. These results show the efficiency of this process (Figure 5 illustrates the recovery of Cu during the time using different solid-liquid ratios in supercritical CO_2 extraction process). As a result, carbon dioxide as a solvent in supercritical conditions has shown a very high ability to separate printed circuit board components and can be considered as a suitable alternative. Also, this material is environmentally friendly and has the ability to be reused.

Wang and Khoo (2014) showed that the use of supercritical CO_2 causes a fast reaction through the disappearance of the boundary between phases and the acceleration of mass transfer. Therefore, the phenomenon of the disappearance of the boundary between phases in supercritical CO_2 accelerates the kinetics of copper extraction (Wang & Xu, 2014). extracted the metal ions in the liquid by placing suitable ligands in supercritical carbon dioxide. Complex donors are responsible for converting various metals into soluble compounds in supercritical CO_2 (Sunarso & Ismadji, 2009). For example, in a study using supercritical carbon dioxide and cyanex 302 ligand as co-solvent, copper metal was separated from wood waste containing copper chromate copper arsenate (Wang & Chiu, 2008).

In 1997, Neil Smart and his colleagues investigated the use of organic phosphorus agents in the presence of supercritical carbon dioxide and studied the complexing agents Kelex 100, Cyanex 272, 301 and 302 and D2EHTPA in extraction. For a wide range of heavy metals, high

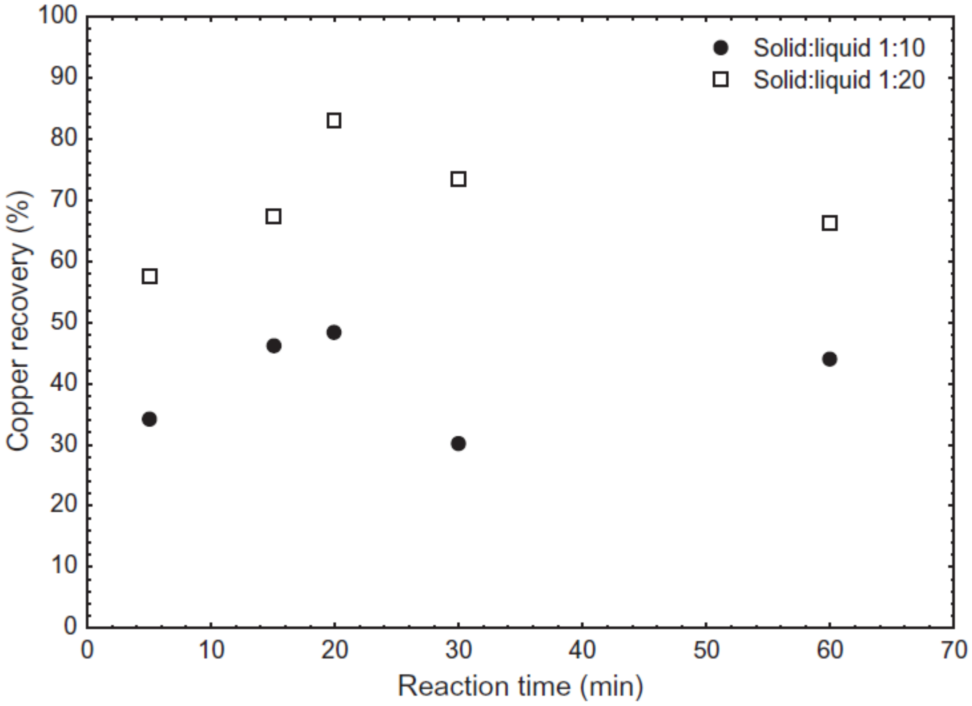

Fig. 5. Copper recovery from PCBs for supercritical extraction using different solid:liquid ratios, with H_2SO_4 (2 M) and 15% H_2O_2 (30%) co-solvents, at 35 °C and 7.5 MPa (Calgaro et al., 2015).

efficiency was observed using Cyanex 301 and 302 and D2EHTPA in samples with low pH and it was concluded that the extraction efficiency increases with increasing pressure. Kelex 100 is very selective for extracting copper metal using CO_2 supercritical fluid (Smart et al., 1997).

In 2001, Jicheng Liu et al presented a new strategy for the supercritical extraction of copper ions. For copper extraction, complexing agent was used along with supercritical fluid. The effects of pressure, temperature and total volume of carbon dioxide on extraction efficiency were investigated. Extraction efficiency with pure supercritical carbon dioxide was low (57.32%). Adding an appropriate amount of methanol (five percent by volume) to supercritical carbon dioxide increased copper extraction to 72%, and in the presence of nonionic surfactant, the yield increased to 90% (Liu et al., 2001).

In 2016, Kang Liu et al In a study, they examined palladium and silver extraction directly from waste circuit boards using supercritical fluid oxidation extraction. This study was to develop an environmentally friendly process for the direct recovery of palladium (Pd) and silver (Ag) from printed circuit board (PCB). The combination of supercritical carbon dioxide and supercritical water was used for extraction in this study. Extraction with supercritical water can significantly increase the efficiency of the process in the separation of lead and silver by eliminating non-metallic compounds. It was also proved that the extraction process with supercritical carbon dioxide, which includes various complex physicochemical steps, is an effective process in the selective recovery of precious metals from electronic and electrical waste (Liu et al., 2016).

Wai et al. in 1993 investigated metal complexes in supercritical solvent. Metal species in solid and liquid materials can be extracted by organic ligands with supercritical CO_2. The efficiency of metal extraction using the simultaneous complex method in SFE depends on a number of factors: (1) ligand stability and solubility, (2) metal chelate solubility, (3) water content and pH, (4) temperature and pressure, (5) chemical structure of metal species and (6) matrix. The results of this research showed that the selection of suitable functional groups in extraction with supercritical fluid along with metal complexing is vital (Wai et al., 1993).

Hsu et al. (Hsu et al., 2021), in a study, the improved extraction of copper metal from electronic waste was investigated and the induced morphological changes were studied using supercritical carbon dioxide. This process included pre-treatment using supercritical carbon dioxide and then using lichen acid using sulfuric acid at a process temperature of 120 degrees Celsius and 148 atmospheres, which lasted for 30 minutes. After that, the treated PCB was washed using a solvent containing 2 M sulfuric acid and 0.2 M hydrogen peroxide. According to the obtained results, 82% of copper was extracted in less than 4 hours during this process.

Fayyaz et al. (Fayaz et al., 2022a), have used supercritical water pretreatment to PCBs to enhance the silver concentration and organic matter removal. The experiments were conducted at the pressure of 250 bar, at 350–450 °C, and the extraction duration of 15–45 min. After pretreatment, the $SCCO_2$ process were conducted using previous stages' product as the feed. Temperature of 51°C, pressure of 217 bar and time of 40 minutes were considered as optimal test conditions. Silver metal was extracted from PCB waste with a yield of 98% without the use of auxiliary solvent and ligand, which was considered as the optimal point of the process.

By examining this process and the studies conducted in relation to the extraction of precious metals from the waste of printed circuit boards, it is shown that this method is a very new and promising and green method for extraction. The results of studies on the field of supercritical carbon dioxide and supercritical water extraction of metals from e-wastes are summarized in Table 3.

Table 3. Summary of the studies on $SCCO_2$ and SCW extraction of metals from e-wastes

Extraction method	Extracted materials	Feed	Results	Reference
Using supercritical water as a pre-treatment along with acid washing process	Metal recycling	Residues of printed circuit boards	The use of supercritical water oxidation as a pre-treatment at 420 degrees Celsius reached a recovery of 99.8% and 80% for lead. While for tin and chromium, they remained in the residue and in supercritical polymerization, over 90% of tin, zinc, chromium, cadmium and manganese metals could be recovered at 420 degrees Celsius.	(Xiu et al., 2013)
Pretreatment of supercritical water with supercritical carbon dioxide extraction	Au, Ag and Pd	Residues of printed circuit boards	The obtained results showed the significant effect of time, temperature and pressure of the supercritical water extraction process on the extraction of precious metals from PCBs waste. Environmental compatibility was proven in this process, which was carried out using iodide-iodide leaching.	(Xiu et al., 2015)
Supercritical methanol	A study on the characteristics of oil and solid products obtained from printed circuit board residues processed with supercritical methanol.	Residues of printed circuit boards	According to the results of this study, the oils contain derivatives of methylphenol and pure phenol, and their amounts increase with the increase of process temperature. Liquid products also contain a significant number of phosphate compounds such as terphenyl phosphate, which decrease significantly with increasing reaction temperature.	(Xiu et al., 2017)
Supercritical carbon dioxide	Recycling printed circuit boards	Residues of printed circuit boards	The results of the recycling process test showed that the base of printed circuit boards can be easily separated into copper foil, glass fibers and polymers.	(Sanyal et al., 2013)
Supercritical carbon dioxide	Cu	Residues of mobile phone printed circuit boards	The results showed that 34.83% by weight of the residues of printed circuit boards were copper. According to the results, it was found that the speed of extraction in supercritical conditions is 9 times the speed of extraction in atmospheric conditions.	(Calgaro et al., 2015)
Supercritical carbon dioxide	Metal ions in liquid samples	Residues of mobile phone printed circuit boards	The disappearance of phase boundaries in supercritical carbon dioxide increases the speed of copper metal extraction.	(Wang & Xu, 2014)
Supercritical carbon dioxide	Cu, Zn and Cd metals	Residues of printed circuit boards	Methanol was adjusted as a ten percent volume modifier and pH=10. The extraction of three heavy metals Cu, Zn and Cd from aqueous samples in the presence of dithizone as a chelating agent was performed by extraction by supercritical CO2 at 120 bar and 50 degrees Celsius by dynamic method.	(Behnamfard et al., 2013)
Supercritical liquid carbon dioxide	Cu	Residues of printed circuit boards	For a wide range of heavy metals, high efficiency was observed using Cyanex 301 and 302 and D2EHTPA in samples with low pH and it was concluded that the extraction efficiency increases with increasing pressure.	(Smart et al., 1997)
Supercritical carbon dioxide	Copper chromate arsenate extraction	wood waste	The results showed that the use of in-situ chelation/SFE technique and the extraction of precious metals with it from wood treated with CCA significantly reduces the risk of releasing toxic metals and metal ions into the environment.	(Wang & Chiu, 2008)
Supercritical carbon dioxide	Cu	Printed circuit boards	The simultaneous use of water and supercritical carbon dioxide was investigated in this study. The highest efficiency for extracting copper metal with high purity was determined at 97% efficiency and 98.7% purity.	(Liu et al., 2001)

CONCLUSION

In this review, an attempt was made to explain the supercritical extraction process. The best known supercritical fluids that is most used in various industries, is carbon dioxide and water. Supercritical extraction is a simple process that is of great interest due to the convenience of operating conditions, good efficiency and low risk for humans and the environment. Today, there is a lot of e-waste that has to be appropriately handled because it is an environmental challenge which cannot be neglected. The extraction of resources and environmental conservation are seldom ever balanced by conventional recovery techniques. According to our literature survey, SCF method may be an excellent choice for treating e-waste because of its special qualities. SCF technique, as opposed to conventional techniques like pyrometallurgy and hydrometallurgy, may effectively eliminate dangerous elements and extract precious items without causing additional contamination. From the perspective of hybrid technologies which combined conventional technologies with SCF method, it is evident that there are improvements in efficiency and yield of metal extraction from e-waste using these combined methods. Furthermore, it could be suggested to use catalysts in combination with $SCCO_2$ and SCW extraction technologies to fasten the process and reducing residence time of the reaction (Mirmousaei et al., 2019). The information provided here makes clear that SCF techniques have greatly advanced recently. The removal of dangerous components and the recycling of precious items have produced extremely excellent outcomes. Nevertheless, several operational and technological challenges prevented the commercial use of scaled-up SCF recovery for e-waste. Additional technological alternative study is required owing to excessive equipment expenditures and operation expenses.

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CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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