



# Supercritical fluid technology - an eco-friendly approach for resource recovery from e-waste and plastic waste: A review

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## ABSTRACT

Rapid technological development and modernization lead to increased production and consumption of electronic equipment as well as plastic commodities. This results in a large generation of electronic waste (e-waste) and plastic waste. The most severe problem associated with these wastes is their informal recycling and improper disposal, threatening the environment and human health. On the other hand, these wastes also provide an opportunity for the recovery of metals and the generation of valuable products. Supercritical fluid technology provides an attractive alternative for the sustainable management of e-waste and plastic waste, as well as resource recovery from these wastes. Therefore, the present review focuses on the applications and potential of supercritical fluid technology for resource recovery from these wastes. The review article presents a comprehensive discussion about the generation and composition of e-waste and plastic waste, the principle of supercritical fluid technology, and the application of various supercritical solvents such as CO<sub>2</sub>, water, and alcohol for resource recovery from e-waste and plastic waste. Finally, the economic aspect of supercritical fluid technology is also critically reviewed, and a comparative economic analysis with existing technologies is presented. Based on critical analysis, it is observed that supercritical fluid technology provides a viable route for the treatment of both e-waste and plastic waste. However, the pilot-scale studies are not reported, and there is a need to further explore the supercritical fluid technology to make a strong case for industrial application.

## 1. Introduction

The use of electrical and electronic equipment (EEE) and plastic has become a crucial part of the modern lifestyle. The most commonly used EEEs are mobile phones, laptops, large and small household appliances, etc. Similarly, plastics are used in our daily lives in bottles, plastic bags, cables, pipes, etc. The rapid technological advances in the EEE industry resulted in the decreased life span of EEE and ultimately generated electronic waste (e-waste) [1–3]. Since the last two decades, e-waste and plastic waste disposal have become a matter of concern due to the large generation [4,5]. As per the Global E-waste Monitor report 2020, 53.6 million metric tons (MMT) of e-waste was produced globally in 2019, excluding PV panels [6,7]. The annual growth of e-waste generation is around 4 %, and it is predicted that the e-waste generation will be around 74 MMT by 2030 [6,8]. In 2019, only 9.3 MMT, i.e., 17.4 % of the total e-waste produced, was recycled by the formal sector [6]. The fate of around 82.6 % of e-waste is not known, and it was landfilled or treated by the informal sector. Fig. 1 shows the e-waste generated by

various continents along with the e-waste recycled in 2019. Fig. 1(a) illustrates that Asia generated the highest e-waste i.e. 24.9 MMT in 2019 [6]. Asia is followed by America and Europe, which have generated around 13.2 and 12 MMT of e-waste in 2019, respectively. However, in terms of recycling, Europe has collected and recycled approximately 42.5 % of the e-waste produced in 2019, which is the highest compared to other continents, as shown in Fig. 1(b) [6].

Similar to e-waste, the generation of plastic waste is also increasing. The growth of plastic production in the last 65 years has considerably increased than any other manufactured material [9]. Plastics have applications in various fields, such as households, electronics, architecture, etc. The unique and outstanding properties of polymers, such as chemical, thermal and moisture resistance, durability, strength, etc., make them best suited for various applications [10]. Presently around 60 % of plastic waste generated is landfilled or accumulated on land and water bodies [9]. If the current plastic usage trend remains consistent, by 2050, around 12,000 MMT of plastic will be incinerated, 12,000 MMT will be landfilled or accumulated in the environment, and 9,000 MMT

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will be recycled [9]. Plastic is highly non-biodegradable and can persist in the environment for thousands of years [11,12]. Fig. 2 shows the worldwide average plastic consumption [13]. The increase in the production of plastics causes an enormous generation of plastic waste, which has created severe environmental problems.

### 1.1. Composition of e-waste and plastic waste

E-waste is a heterogeneous mixture that mainly consists of organic materials, metals, and ceramics [14]. Plastics such as polycyclic aromatic hydrocarbons (PAHs), polybrominated dibenzodioxins, persistent organic pollutants such as brominated flame retardants (BFRs), glass fibers such as epoxy resins, and paper are included in organic materials. The share of metals in e-waste is around 60 wt%, and that of plastic is around 15 wt% [15]. E-waste contains base metals (Cu, Fe, Al, Sn), rare metals (Ta, Ga), precious metals (Au, Ag, Pt, Pd), and other hazardous metals (Cr, Pb, Be, Hg, Cd, Zn, Ni). Silica, alumina, alkaline earth oxides, barium titanate, etc., are involved in the ceramic framework of e-waste [14]. The statistical view of the average composition of different materials present in e-waste is depicted in Fig. 3 [16]. The composition of plastic waste in municipal solid waste (MSW) varies according to the area, and the distribution of plastic waste in MSW is far from uniform. However, the average composition of plastic waste in MSW roughly corresponds to the data shown in Fig. 4 [17]. Low-density polyethylene (23 wt%) and high-density polyethylene (19 wt%) are the major plastics present in MSW [17].

### 1.2. Need for recycling

#### 1.2.1. Environmental protection and resource recovery

Improper disposal of e-waste and plastic waste, such as landfilling, unregulated pile-up, and incineration of these wastes, can affect the three pillars of the environment, i.e., air, water, and soil which eventually harm the entire eco-system. It can threaten the global environment at large, as shown in Fig. 5 [18]. Table S1 depicts the toxic materials existing in e-waste and their hazardous effect on human health [19]. The improper disposal of these waste in landfills or dumping sites can result in the leaching of heavy metals or hazardous chemicals into the soil, which eventually causes damage to the fertility of the soil and contaminates the groundwater [4,5]. Landfilling is an undesirable option for handling e-waste due to the poor biodegradability of plastic content and pollution of groundwater due to BFRs and heavy metals accumulation in the soil. In the aspects of water pollution, e-waste or plastic waste constituents can contaminate both the groundwater and surface water through diffusion and leaching, which causes disturbance

in the oxidative stress margin of aquatic life or even death of some plants and aquatic life. Improper management and recycling of plastic and e-waste release dust particles or toxins into the environment, which results in air contamination and causes air pollution. Burning these wastes generates toxic gases, which can cause chronic diseases and cancer [20,21]. Air pollution can extend up to a thousand miles away from recycling sites if not regulated and performed carefully.

Considering the above factors, such as composition, the toxicity of these wastes, food chain contamination, and occupational exposure to hazardous substances in recycling areas, it can be concluded that these wastes can disturb biodiversity and harm entire ecosystems if not recycled properly.

E-waste and plastic waste management are also crucial from the aspect of resource conservation. Table 1 depicts the total energy which can be saved compared to virgin mining if metals are recovered from e-waste [19]. Apart from environmental benefits, the motive behind recycling and recovery is the economic benefits associated with recycling. Moreover, the recovery of metals from e-waste is also beneficial due to higher concentration of metals in e-waste with respect to ores [1,4,24]. Limited metal resources on earth drive researchers to develop eco-friendly technologies to recycle and reuse valuable metals from e-waste [25]. In the case of plastic, the concern of discarding plastic waste can be treated as a source to replace the diminishing fossil fuels with the aspiration of encouraging sustainable development. The higher heating value of the plastic waste makes it a potential feedstock for the generation of fuel, and this can be a better option for plastic waste management than landfilling [10]. Attempts have been made to recycle and reuse plastic waste to lessen the hazardous impact on the environment and human health. Due to the decreasing fossil fuel, the economic growth at the present rate is non-durable. Plastic waste has the potential to overcome this lag in economic activity. It is both a challenge and an opportunity to convert waste plastic into wealth using an efficient and sustainable process.

#### 1.2.2. SCF technology for environmental protection and resource recovery

Based on the above discussion, it is imperative to develop sustainable technologies for e-waste and plastic waste recycling from the aspect of environmental protection and resource recovery. It would be worth transforming the waste into wealth by retrieving its metal content, converting plastic waste into valuable products, and preserving the resources. In this regard, supercritical fluid technology (SCF) has the potential to resolve these issues. Due to its specific properties, it has been observed as an attractive and feasible technique for treating e-waste and plastic waste [15]. The utilisation of greener solvents, such as supercritical water (SCW), supercritical CO<sub>2</sub>(scCO<sub>2</sub>), etc., makes it more

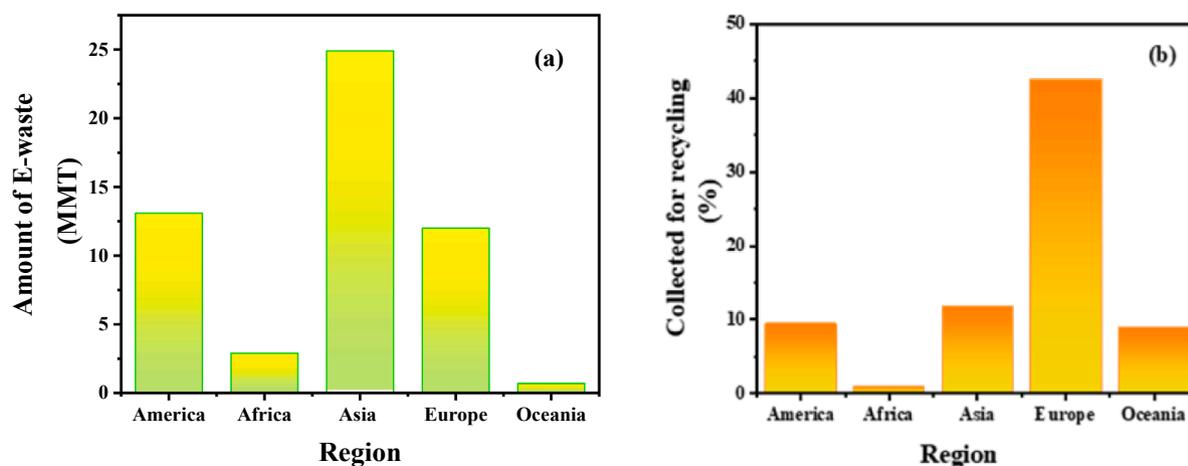


Fig. 1. Global scenario of e-waste in the year 2019 (a) E-waste produced (b) E-waste collected for recycling (%).

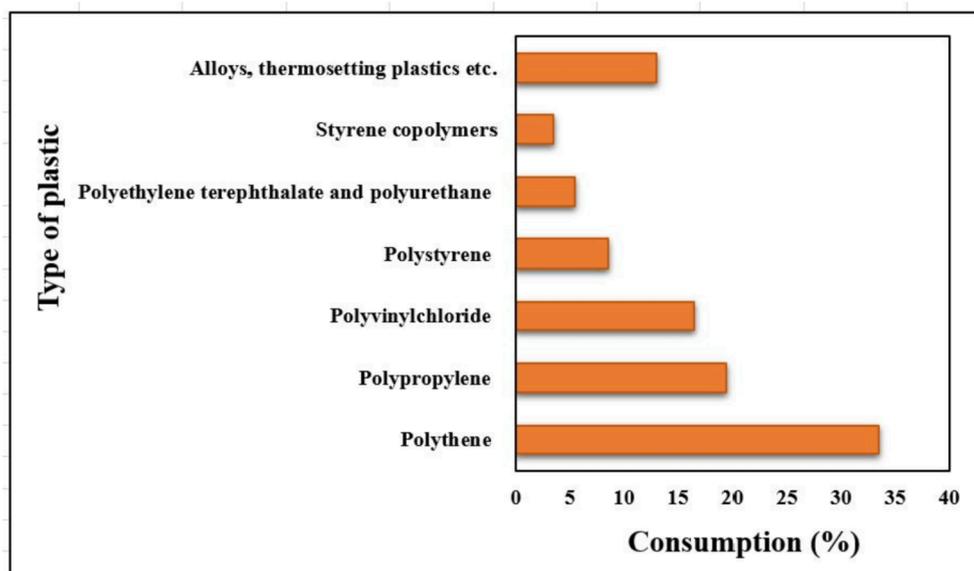


Fig. 2. Worldwide consumption of respective plastics.

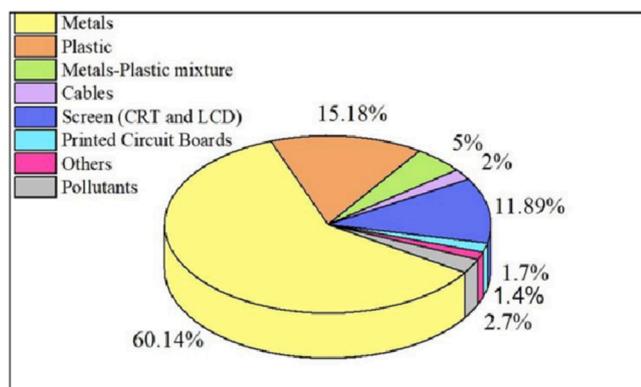


Fig. 3. Average composition of materials in e-waste (%).

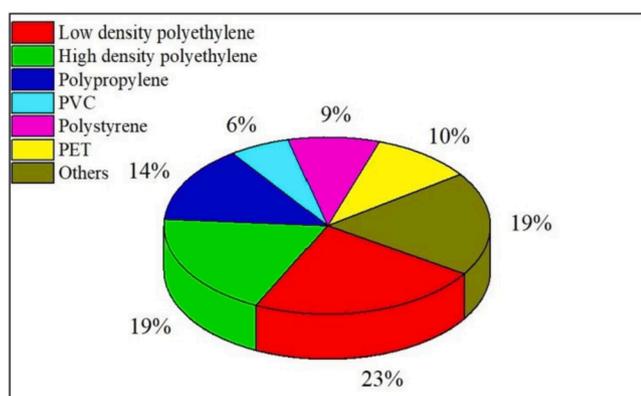


Fig. 4. Typical composition of plastic waste within MSW (%).

reliable and promising. Compared to other technologies like pyrolysis, acid treatment, etc., the SCF technologies provide rapid kinetics, high reaction rate, and efficient yield, making them superior [26,27]. An environmental and economic analysis of our previous research suggests that SCF technology has the potential to produce Br-free liquid products and enriched metal components without the utilization of any toxic chemicals. Even > 90 % of the solvent used can be recycled using a

rotary evaporator after the SCF treatment, which turned the SCF process towards a close-looped and minimal discharge technology [26]. Moreover, no emissions of NO<sub>x</sub>, soots, dioxins, etc., are observed during SCF operation in the degradation process of organic materials [28]. Hence, it can be inferred that SCF technology sustainably promotes effective resource recovery and significantly reduces the environmental pollution caused by e-waste. In this context, the present review article discusses the principle of SCF and the application of supercritical fluids such as water, CO<sub>2</sub>, and different alcohols (ethanol, methanol, etc.) for recycling e-waste and plastic waste. Furthermore, the review article presents a comparative and economic analysis of different technologies employed for waste treatment and SCF technology. A future perspective provided in the review article highlights the gaps and actions needed for the industrial application of SCF technology. This review article will guide the way forward for resource recovery and waste management using SCF technology.

## 2. Methodology

The present article analyses the application of SCF technology for the treatment of e-waste and plastic waste. The literature search was planned and organized in three parts. Initially, the research articles, reports, web pages, and book chapters were searched to know the current statistics of e-waste and plastic waste generation, recycling, and their environmental impact. The literature search was carried out using various search engines such as Scopus, Web of Science, Google Scholar, etc. Various keywords such as “supercritical fluid technology”, “electronic waste”, “plastic waste”, “detoxification”, “metal recovery”, and their combinations were employed for the literature search. The articles were analyzed initially by carefully reading the abstract and were selected according to the scope of this review. After selecting research articles, a thorough analysis was performed to categorize them based on plastic and e-waste recycling.

Moreover, the articles were again subcategorized based on different solvents, i.e., supercritical water, supercritical CO<sub>2</sub>, supercritical alcohols, and other solvents for both plastic waste and e-waste treatment. At last, the economic aspect with challenges, existing problems, and proposed solutions of supercritical technologies for both plastic waste and electronic waste were discussed. Mostly, the last 15 years of research articles were used for citation. Older papers were used to provide justification, support discussion, and provide examples if necessary.



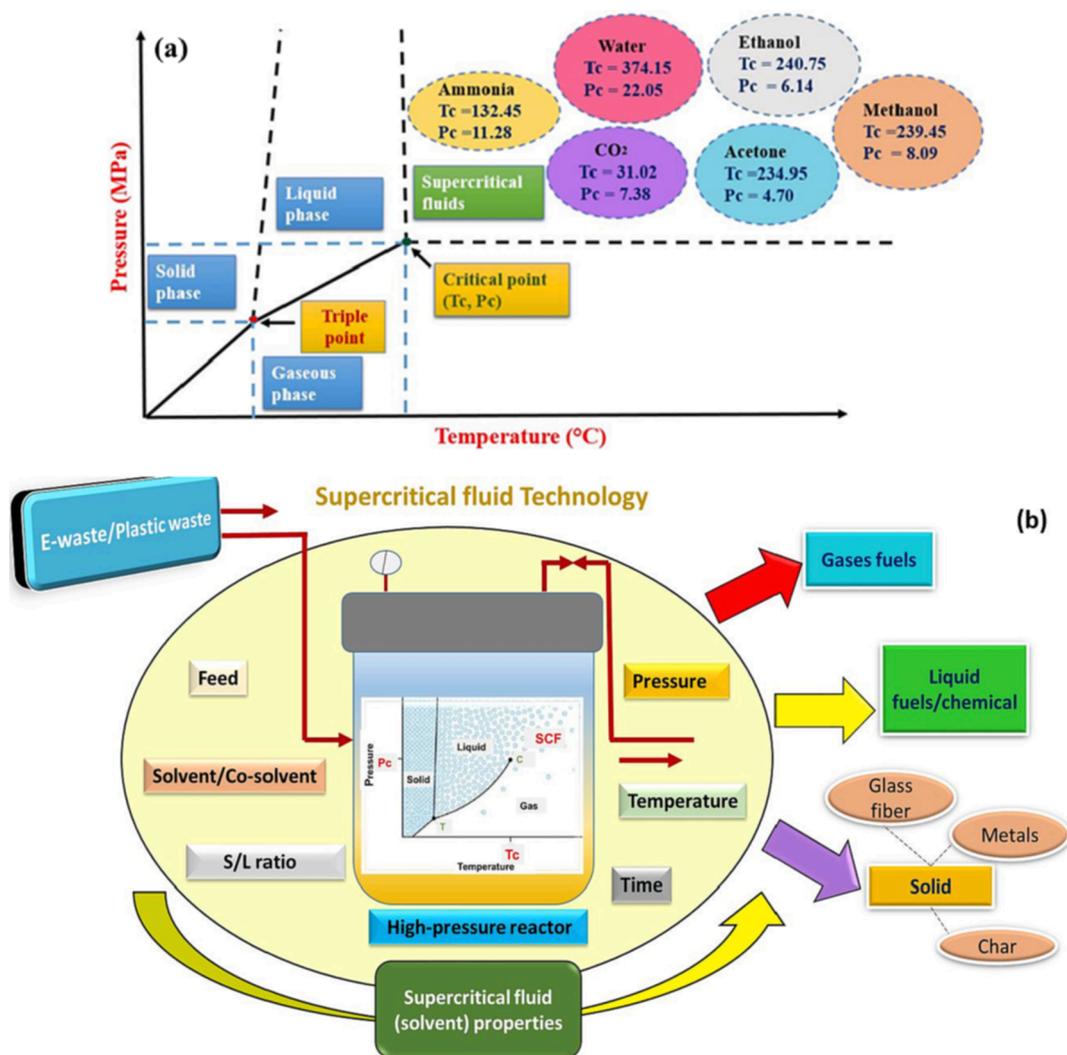


Fig. 6. (a) Schematic phase diagram for selected supercritical fluids along with their supercritical condition (b) Typical route of the SCF technology in the treatment of e-waste and plastic waste.

Table 2

Viscosities, densities, and diffusion coefficient of gases, liquid, and SCFs.

Fluid	Density ( $\text{g cm}^{-3}$ )	Viscosity (Pa. s)	Diffusion coefficient ( $\text{cm}^2 \text{s}^{-1}$ )
Gas	$(0.6-2) \cdot 10^{-3}$	$(1-3) \cdot 10^{-5}$	0.1-1.0
SCFs	0.2-0.9	$(1-3) \cdot 10^{-4}$	$(0.1-5) \cdot 10^{-4}$
Liquid	0.6-1.6	$(0.2-3) \cdot 10^{-3}$	$(0.2-3) \cdot 10^{-5}$

substances sensitive to heat [52]. Additionally, its compact and linear molecular structure leads to a speedy diffusivity, effective mass transfer, and lower viscosity, allowing rapid diffusion in waste material [53,54]. Furthermore,  $\text{scCO}_2$  has high solvation power than many SCFs, which can be attributed to the relatively higher density of  $\text{scCO}_2$  [49,55,56]. The  $\text{scCO}_2$  will neither contaminate the extract nor the remaining extracted solid [49]. The  $\text{scCO}_2$  can be used along with other solvents as well [57]. Furthermore, the  $\text{scCO}_2$  can also be recycled and reused [57,58].

#### 4.1.1. Supercritical $\text{CO}_2$ for resource recovery from e-waste

The role of  $\text{scCO}_2$  in e-waste processing is possibly the swelling of the organic constituents of e-waste. The swelling of polymers causes morphological changes, which may increase the transport phenomenon and kinetics of the leaching [37,38]. These properties have helped in the extraction of metals from e-waste. Various works have been reported on

the recovery of metals from e-waste using SCF technology. Bertuol et al. examined the effectiveness of  $\text{scCO}_2$  in the presence of  $\text{H}_2\text{O}_2$  and  $\text{H}_2\text{SO}_4$  as co-solvents for cobalt recovery from discarded lithium-ion batteries [59]. Authors reported 95 wt% extractions of cobalt within 5 min. The process is more efficient in  $\text{scCO}_2$  than in atmospheric pressure because the reaction time at atmospheric pressure was 60 min compared to 5 min in  $\text{scCO}_2$ . Similarly,  $\text{H}_2\text{O}_2$  used is 8 % (v/v) in the case of atmospheric pressure compared to 4 % (v/v) in  $\text{scCO}_2$ . Electrowinning results in the recovery of Co from leached solutions with a purity of 99.5 wt%. Similarly, Calgaro et al. examined the utilization of  $\text{scCO}_2$  with  $\text{H}_2\text{SO}_4$  and  $\text{H}_2\text{O}_2$  as a co-solvent for copper recovery [60]. It is reported that 90 wt% of the copper was recovered in 20 min with 2.5 M  $\text{H}_2\text{SO}_4$  and 20 % of  $\text{H}_2\text{O}_2$ . Also, it is reported that the SCF extraction was nine times more efficient and rapid than the atmospheric extraction. The  $\text{CO}_2$  does not take part in the reaction, and it just helps in removing phase boundaries by assisting in the mass transfer [60].

Recently, Hsu et al. and Peng and Park investigated the recovery of metals using  $\text{scCO}_2$  and suggested that  $\text{scCO}_2$  influences the morphology of waste PCB by delamination, formation of pores, and cracks [61,62]. Hsu et al. found that  $\text{scCO}_2$  physically changes the organic polymers (plastic), which enhances the metal leaching [27]. The morphological changes were discovered stable and permanent. Hsu et al. also explained the effect of  $\text{scCO}_2$  and reported the physical differences in the PCB sample after 6 h of the reaction in the  $\text{scCO}_2$  environment at solid to

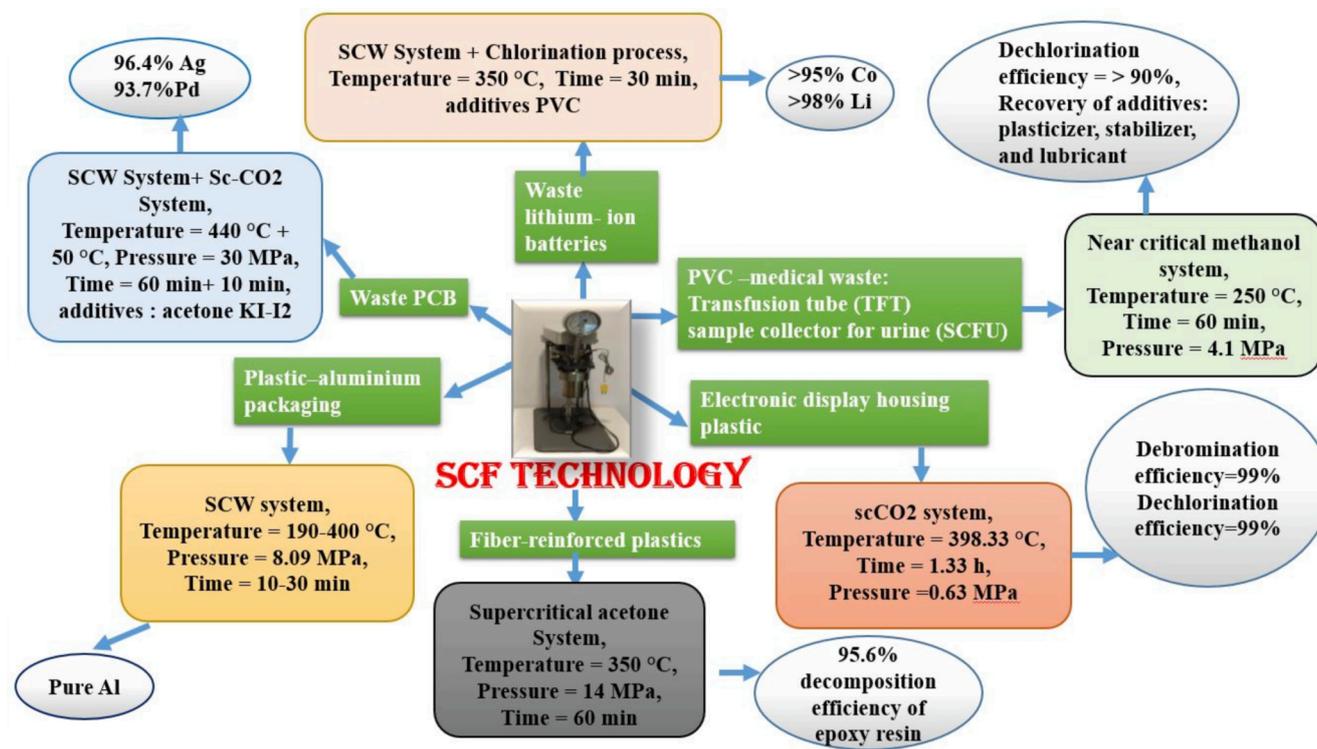


Fig. 7. SCF technology for e-waste and plastic waste treatment [38–43].

liquidi (S:L) ratio of 1:40, reaction temperature of 120 °C, and a reaction pressure of 150 bar. It is found that, due to the free volume expansion of the polymer, the surface of the polycarbonate swelled and became translucent and roughened. The authors reported that the pre-treatment of the PCB with  $scCO_2$  and aq.  $H_2SO_4$  at 120 °C temperature and 148 atm pressure for 30 min followed by leaching of the treated PCB using 2 M  $H_2SO_4$  and 0.2 M  $H_2O_2$ , resulted in the 82 % extraction of Cu within 4 h. Advanced examination of the kinetics of leaching and transport phenomena in the  $scCO_2$  environment is still required to completely investigate the mechanisms in which the  $scCO_2$  solvent upgrade the efficiency of metal leaching. Peng and Park investigated the physical and chemical changes in the polymer-metal matrix of WPCBs by the treatment of two-stage  $scCO_2$  solvent system [62]. The first stage treatment is performed at 120 °C temperature, 250 rpm, and 150 bar pressure by keeping 10 ml of 1 M  $H_2SO_4$  and WPCB in a  $scCO_2$  system, placed in a high-pressure reactor, which creates foaming, fracturing, and formation of micron-sized pores in the protective layer of the WPCBs. The second stage of treatment is performed at room temperature with 2 M  $H_2SO_4$  in the presence of an oxidiser (0.2 M of hydrogen peroxide) which results in the further delamination of the outer polymer coating of WPCBs. The treatment allows selective extraction of metals such as Ni and Cu, which makes further separation process (e.g., electrowinning) simpler. Additionally,  $scCO_2$  treatment leads to reduced cost of mechanical strength (grinding) of WCBs and also avoids the use of toxic reagents such as cyanide and aqua-regia. Further, the authors reported that Au could be recovered in solid rather than in dissolved ions which consequently reduces the separation and recovery steps and makes the process more sustainable. In another study, Hsu et al. reported that the pre-treatment of WPCBs at 120 °C temperature, time of 30 min, and 148 atm pressure and leaching with 2 M  $H_2SO_4$  and 0.2 M  $H_2O_2$  at ambient condition for 3 h creates pores inside the surface and on the surface [61]. These formed pores weaken the tough and flexible structure of WPCBs, make penetration of solvent easier, and open up the channels, which result in rapid leaching of metals. The changes found in the morphologies of polymers are completely physical.

Furthermore,  $scCO_2$  has also been used for the conversion of e-waste

plastic to valuable materials along with the dehalogenation of e-waste plastic. Zhang and Zhang have utilized  $scCO_2$  for dechlorination and debromination of electronic display housing plastic [38]. Computers, mobile phones, television, etc., contain halogenated plastic, including BFRs and PVC [63]. Acrylonitrile-butadiene-styrene (ABS) and high-impact polystyrene (HIPS) are other significant polymers present in e-waste. The presence of various halogenated compounds in e-waste plastic requires safe handling to prevent harmful effects on human health and the environment. The decomposition mechanism of the electronic housing display suggests that the ABS decomposes into aromatic hydrocarbon radicals such as benzene, ethylbenzene, and minor molecular gases [38]. The probable degradation mechanism of electronic housing display by  $scCO_2$  is depicted in Fig. 8 [38]. The decomposition of PVC generates HCl by the zipper mechanism, whereas the polyenes degrade to form the small molecules via chain scission along with the production of benzene rings [43]. The primary decomposition pathway for tetrabromobisphenol A is the scission initiated due to cleavage between two phenyl groups, producing 2,6-dibromo-4-(1-methyl ethyl) phenol. This dibromo product further debrominates to form 2, 6-bis (1, 1-dimethyl-ethyl)-4-ethyl-phenol, which could be further oxidized to produce benzenbutanoic acid and benzenpropionic acid [64,65]. These aromatic hydrocarbon molecules diffuse in the  $scCO_2$  system. The aromatic hydrocarbon molecules attain a homogeneous combination in the  $scCO_2$  system as they have unique liquid solvents and gas properties. The assemblage of the decomposition products leads to the development of clusters composed of benzene derivative compounds with an increase in exposure time and temperature. Carbon materials can be formed by degrading and converting aromatic hydrocarbon at 550 °C. For example, the combination of ethylbenzene and cumene radicals might give 1,1'-(1,3-propanediyl) bis benzene and 1,1'-(methyl-1,3-propanediyl)bis benzene. The consumption of aromatic hydrocarbon radicals of benzene, toluene, and ethylbenzene leads to a higher concentration of 1,1'-(1-methyl-1,3-propanediyl)bis benzene and 1,1'-(1,3-propanediyl)bis at 375 °C. The highest dechlorination and debromination efficiencies were reported as 99.12 % and 99.51 %, respectively [38].

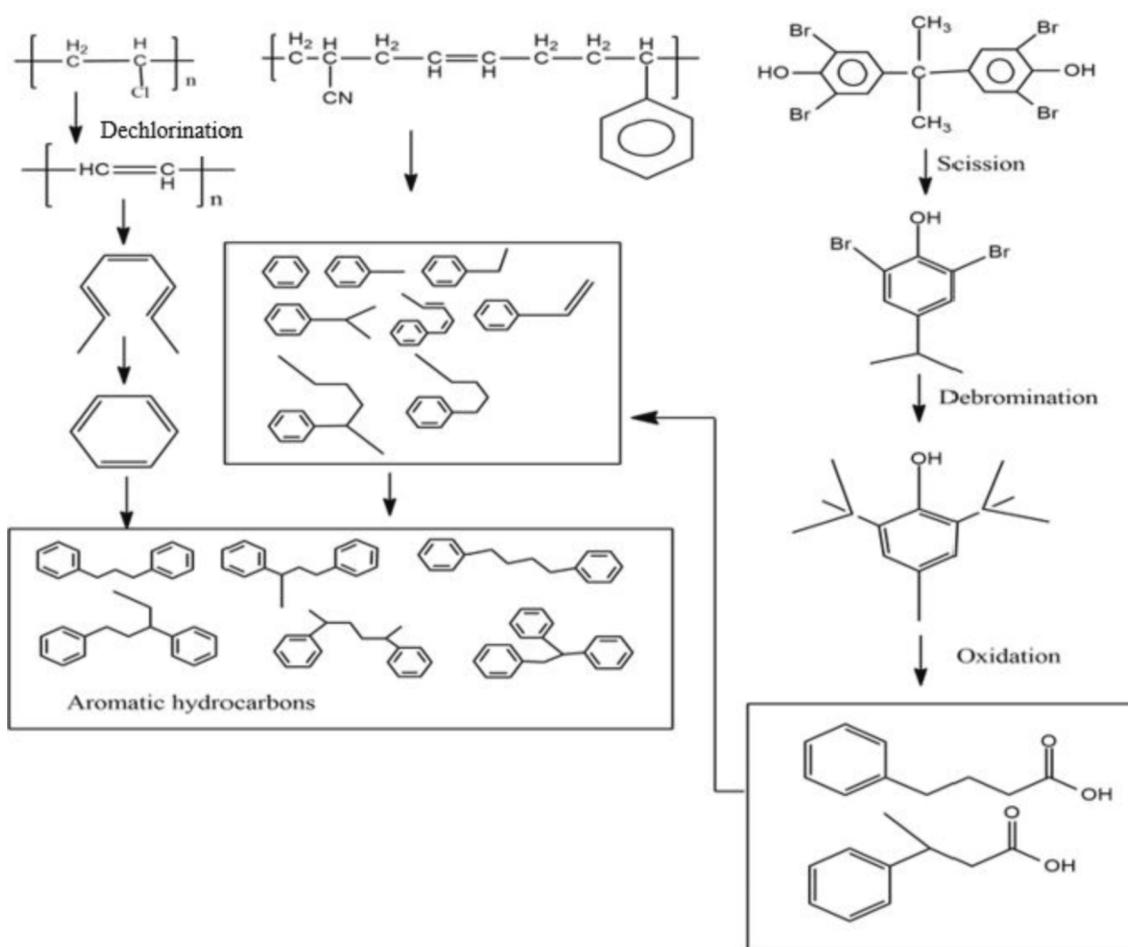


Fig. 8. Dehalogenation of BFRs and PVC in the electronic display housing plastic “Adapted with permission from [38], copyright (2022) Elsevier”.

Based on the above investigations, it can be summed up that  $scCO_2$  is found to be a promising solvent to recover metals or polymers from waste electronics by tuning it with appropriate co-solvent such as  $H_2SO_4$ , ethanol etc. It can also help to significantly improve the reaction kinetics as compared to ambient conditions and reagent consumption (co-solvent or oxidant amount) without compromising the reaction yield. It has been observed that the  $scCO_2$  atmosphere creates permanent morphological changes and induces pores and cracks, which eventually leads to the weakening of organic components. This phenomenon enhances the transport of the solvent by opening up the channels. Additionally, this also results in a major reduction in the mechanical process (grinding, crushing, and physical separation) of the feed material.

Table 3 presents a summary of research work carried out using  $scCO_2$  for resource recovery from e-waste. Nonetheless, further investigation of  $scCO_2$  in the extraction of metals, transport phenomenon, and leaching kinetics is still required to fully understand the fundamental mechanism of the  $scCO_2$  solvent system.

#### 4.1.2. Resource recovery from plastic waste using $scCO_2$

The streams of plastic waste are a heterogeneous mixture composed of a broad array of polymers, chemical additives, and contaminants which pose a significant challenge for waste plastic recycling [71]. Therefore, the threat of the availability of unwanted materials that can gather in the plastic cycle and migrate into the environment poses a considerable challenge to the closed-loop recycling of plastics [72]. Solvent extraction is an efficient approach for treating hazardous soluble compounds present in solid waste. The SCFs have excellent solvability and high penetration power compared to any other solvents, due to which it is particularly suitable for the solid-liquid extraction process.

Peng et al. investigated the application of  $scCO_2$  for the recovery of PBDEs from HIPS plastic [73]. The supercritical process at 65 °C temperature, 20 MPa pressure, 2:1 vol ratio of  $scCO_2$  to HIPS solution, and centrifugation of solution after the supercritical process at 10,000 r/min and 30 °C results in 97 % recovery of PBDEs. Moreover, the concentration of PBDE residue in HIPS was reduced to lower than 0.1 % (dry).

Compared to the conventional method for extracting polymeric matrices,  $scCO_2$  has been effectively applied. Said et al. examined and compared the experimental and theoretical model for the extraction of impurities from post-consumer polypropylene using  $scCO_2$  [74]. The theoretical model considers the overall effect of two parameters in the  $scCO_2$  extraction, i.e., diffusion through matrix and solubility in  $scCO_2$ , to reveal kinetics limitations and analysis of the extraction process. This process helps to examine extraction and revealed appropriate kinetics limitations affecting the extraction process. Variations in pressure (100–300 bar) and temperature (50–90 °C) conditions were explored and analyzed by fitting the model to the experimental data. As a result, details about diffusion coefficients and activation energies’ effect on diffusion and solubilities were acquired. The modeling result showed that the  $scCO_2$  pressure is essential and affects the diffusion coefficient. The diffusion coefficient of contaminants through swollen polypropylene has a magnitude of  $10^{-11} - 10^{-10}$ . An inverse correlation was reported between the diffusion coefficient and molecular weight. Good consistency was reported between experimental and theoretical data, revealing that contaminants from polypropylene could be reduced by monitoring solubility and diffusion.

In another study,  $scCO_2$  is used for the extraction of carbon by the degradation of organic waste. Zhao et al. have investigated the use of SCW combined with  $scCO_2$  for the gasification of polystyrene (PS) [75].

**Table 3**  
Research work on e-waste and plastic waste treatment by using scCO<sub>2</sub>.

E-waste	Reaction condition	Degradation content	Major product	References
PCB	170 °C, 7.5 MPa, Ethanol as a co-solvent	Brominated epoxy resin	69.53 wt% of phenolic and other compounds	[37]
PCB	35 °C, 20 min, 7.5 MPa, S/L ratio-1:20, 20 % (v/v) H <sub>2</sub> O <sub>2</sub> and 2.5 M H <sub>2</sub> SO <sub>4</sub>	–	90 % Cu	[60]
Cell phones	90 °C, 30 min, 10 MPa, 1 M Malic acid and Citric acid, 5 % (v/v) H <sub>2</sub> O <sub>2</sub> , S/L ratio – 1:20	Liquid crystal displays screens	94.6 % In	[66]
PCBs	Supercritical CO <sub>2</sub> extraction, co-solvent: Cyanax 302 and acetone, 51 °C, 40 min, 21.7 MPa.	Ag extraction from PCB	98.75 % of Ag	[67]
PCB	1st step: scCO <sub>2</sub> condition: 120 °C, 148 atm., 30 min, 2 M H <sub>2</sub> SO <sub>4</sub> 2nd step: 2 M H <sub>2</sub> SO <sub>4</sub> , 0.2 M H <sub>2</sub> O <sub>2</sub> at ambient temperature and pressure, time – 3 h	–	80 % Cu 58 % Ni 34 % Al ~10 % Fe	[61]
Lithium- ion batteries	75 °C, 5 min, 75 bar, 4 % (v/v) H <sub>2</sub> O <sub>2</sub> , 2 M H <sub>2</sub> SO <sub>4</sub> , S/L ratio – 1:20	–	95 % Co	[59]
PCB	60 °C, 200 bar, 30 min, 1 ml/min CO <sub>2</sub>	–	97 % Cu	[68]
PCB	1st step: scCO <sub>2</sub> condition: 1 M H <sub>2</sub> SO <sub>4</sub> , 120 °C, 150 bar, 20 min. 2nd step: at ambient temperature and pressure, 2 M H <sub>2</sub> SO <sub>4</sub> , 0.2 M H <sub>2</sub> O <sub>2</sub> in 2 h	–	1st step: 87.3 % Ni, 90 % Cu. 2nd step: 97.4 % Ni, 99.99 % Cu and 96.6 % Au.	[62]
Electronic display housing plastic	398 °C, 90 min, 0.63 MPa	BFRs and PVC	99.51 % debromination and 99.12 % dechlorination	[38]
Oil adhesive high-density polyethylene (HDPE)	300 bar, 80 °C, 19 h	Decontamination of (HDPE) oil containers	Reduction of polycyclic aromatic hydrocarbons contamination	[49]
Brominated flame retardants from polymers	2 h, 60 °C, 25 MPa, 1000 rpm	Brominated flame retardants		[69]
Polypropylene	45 °C, 100 bar, 24 h, PP: Manganese oxide aerogel or nanoparticle ratio 20:1.	Polypropylene	Acetone, acetic acid and formic acid	[70]

The gasification of PS degradation was investigated in different conditions, such as a combined SCW/scCO<sub>2</sub>, and scCO<sub>2</sub> environment. The authors reported that under SCW conditions at 700 °C, the presence of CO<sub>2</sub> enhances the amount of carbon conversion to 46.7 % in 20 min. In the case of CO<sub>2</sub> condition, the highest amount of CO<sub>2</sub> consumed was 12.5 % by PS degradation. At 400 °C, in scCO<sub>2</sub> conditions or in SCW/scCO<sub>2</sub> environment the composition of the liquid product of PS degradation was the same. The composition of the liquid product included ethylbenzene, alpha-methylstyrene, styrene, and 2-phenylnaphthalene at 400 °C. At 600 °C, it majorly consists of naphthalene, biphenyl, anthracene, and fluorene. The authors investigated the formation of carbon microspheres and reported that the increase in temperature and time results in the reduced diameter of carbon microspheres. For instance, the average radius of the carbon microspheres found at 700 °C for 20 min under the supercritical water/CO<sub>2</sub> mixed environment is much smaller as compared to that of carbon microspheres obtained at 600 °C for 20 min under the CO<sub>2</sub> environment. Because the formation of carbon conversion efficiency was increased over 20 min of time, and the carbon microspheres continued to get smaller. Hence, the authors

revealed that it is possible to create the desired carbon microspheres with a specific radius at a micro-nano level, which can be used in various applications such as capacitors templates, adsorption for pollutants, and in drug delivery field.

In a recent study, Elmanovich et al. investigated the recycling of polyethylene in an oxygen-rich scCO<sub>2</sub> environment [76]. Recycling of polyethylene was carried out by thermal oxidation in an oxygen-enriched scCO<sub>2</sub> condition and in a pure oxygen atmosphere under a pressure of 215 and 14 bar, respectively at 140 °C. Thermogravimetric analysis revealed that the amount of volatile fraction increased from 10 mass% to 40 mass% upon increasing the oxygen to polymer ratio from 1:1 to 1:3. The authors reported that acetic acid, formic acid, propionic acid, butyric acid, and valeric acid were the major components of the volatile fraction. Furthermore, the authors reported that the lower oxygen content in scCO<sub>2</sub> is more favourable for the decomposition of polyethylene to oligomers. Hence, the authors concluded that the scCO<sub>2</sub> environment with lower oxygen quantity is more useful for performing the thermal oxidation of polyethylene. Few other studies on the application of scCO<sub>2</sub> for the processing of waste plastic are shown in Table 3.

The utilization of  $\text{scCO}_2$  has mainly been explored as a green alternative to conventional solvents for industrial applications in numerous fields such as separation processes, cleaning, synthesis, particle forming, particle modification of polymers, and organic reactions [77]. The use of SCFs like  $\text{CO}_2$  in industrial applications has great benefit to develop a closed-loop process that will replace the conventional toxic solvents that are more hazardous to the environment [78]. However, future study is needed to explore the design process for the treatment of polymer residue, solvent recycling, and detailed cost analysis that will eventually incorporate the overall waste management and makes the process more sustainable. Fig. 9 summarises the application of  $\text{scCO}_2$  for the sustainable management of e-waste and plastic waste along with the generation of valuable products and metal recovery.

Overall, it has been found that for plastic waste treatment, the mechanism of  $\text{scCO}_2$  extraction has been controlled by diffusion coefficient and solubility. Both these parameters are considerably dependent on the organic material matrix and contamination. The parameters such as temperature, pressure, and reaction time are essential parameters in the  $\text{scCO}_2$  operation for plastic treatment. Pressure has a positive effect on the polymer dissolution by causing a swelling effect and increasing mobility, eventually decreasing the activation energy for diffusion. Increasing residence time and reaction temperature in accordance with the experimental condition have also caused a considerable increase in the yield of the degradation or recycling of organics in the  $\text{scCO}_2$  atmosphere.

#### 4.2. Resource recovery using water as a solvent in SCF technology

Water is an ideal green solvent for SCF technology due to its non-toxicity and non-flammability [79]. The critical temperature and pressure of water are  $374^\circ\text{C}$  and  $22.1\text{ MPa}$ , respectively. At critical conditions, the characteristics of liquid and vapor change dramatically. The probable reason for this is that the H-bonding weakens at the critical point at high temperatures [29]. Supercritical water (SCW) has a high mass transfer coefficient, high solubility, and low viscosity as compared to ambient water [80–82]. At the critical point, the dissociation constant

$K_w$  for water is three times greater than that of ambient water [82]. Its indistinguishable phase boundaries increase the mass transfer rate and improve the kinetics of the reaction. Some of the reactions which involve SCW are gasification, liquefaction, hydrolysis, and oxidation [83–88]. SCW can be widely used in different fields to treat toxic waste such as plastic, biomass, dyes, sewage sludge, etc. [89–91].

Supercritical water gasification (SCWG) is another novel technology for the treatment of organic waste. The unique properties and environmental friendly nature of SCW make it more reliable and attractive for efficient waste treatment [92]. High diffusivity, high solubility, low viscosity, and low dielectric constant are gasification's prime properties which provide a uniform reaction medium and high reaction rate for the efficient degradation of organic compounds and gases [92–94]. Therefore, a uniform reaction environment can be created that accelerates the reaction by reducing the phase between the reaction [93]. Ciuffi et al. define the benefits of SCWG over incineration by depicting its environmental friendly nature and generating valuable products [95]. Incineration produces toxic chemicals such as dioxins, furans, HCl,  $\text{NO}_x$ ,  $\text{SO}_2$ , and HF, whereas, in gasification, a lower level of pollutants is created, as said over [95]. Another reason for SCWG treatment of waste is preferable to incineration because, unlike incineration, SCWG of waste produces syngas. Several studies have been performed to treat e-waste and plastic waste using subcritical/SCWG; some of the studies have been discussed in the subsequent sections.

##### 4.2.1. SCW for resource recovery from e-waste

SCW is a promising technique to recover resources from e-waste by decomposing the hazardous organics material into valuable liquid products and recovering metal simultaneously. The two major and most common SCW approaches for the treatment of e-waste are (i) SCW oxidation (SCWO) performed in the presence of oxygen and (ii) SCW depolymerization (SCWD) performed under a reduced atmosphere [96]. Depending upon the temperature, pressure, and time SCW can act as a reactant, product, and catalyst as it can provide abundant  $\text{OH}^-$  and  $\text{H}^+$  species due to its unique properties [82].

Li and Xu investigated the SCW treatment to degrade the brominated

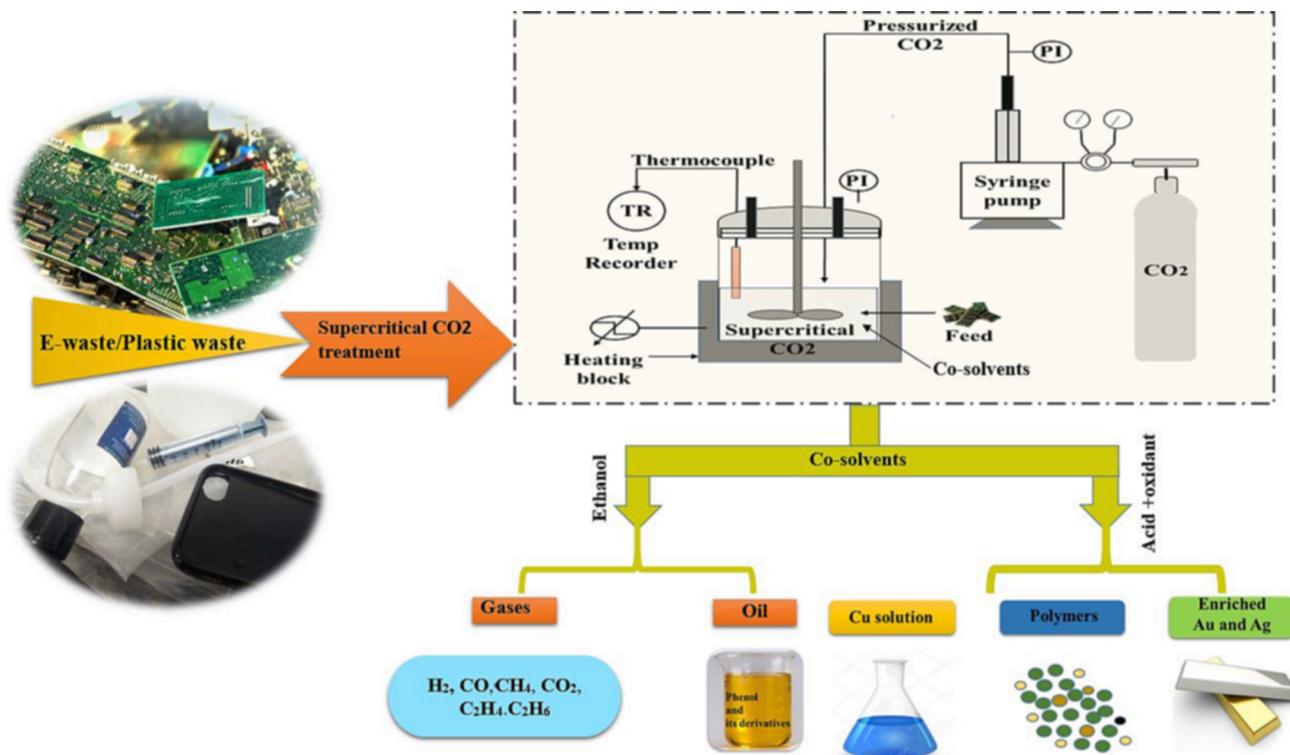


Fig. 9. Resource recovery from e-waste and plastic waste using supercritical  $\text{CO}_2$  treatment [37,38,62].

epoxy resins (BERs) and recover metals from waste random access memory (WRAM) [82]. The mechanism for organic decomposition is probably the free radical mechanism, as shown in Fig. 10. The main decomposition products were phenol, 2-methylphenol, 4-(1-methyl-ethyl)-phenol, 4-methylphenol, 4-ethylphenol, 4-ethylphenol, 4-phenylphenol, and HBr. Moreover, the authors reported around 90 % degradation of the BERs. As per the bond-energy theory, free radicals are formed when the supplied energy is more than the molecule dissociation energy, leading to bond breakage [82]. The initial decomposition method is concerned with producing some monomers starting from the degradation of a comparatively weaker bond such as  $C_6H_5O-CH_2$ . Subsequently, the higher energy bonds such as  $C-C$ ,  $C_6H_5-O$ , and  $C_6H_5-Br$  would be broken with an increase in the energy input. Afterward, the free radicals can form new compounds by conjugation and catalysis. The metal (Cu, Al, Au) recovery rate was 99.80 % under optimized conditions, as mentioned in Table 4.

Soler et al. also investigated the decomposition of BFRs in a batch reactor using subcritical water conditions [97]. The influence of temperature on debromination was examined by varying temperature from 225 to 275 °C at S/L ratio of 1:5 and process time of 180 min. It is reported that debromination efficiency was increased with reaction temperature (225 °C to 275 °C) from 18.5 to 63.6 %. Mostly bromine was retained in the water. The application of SCW oxidation was also examined to recover tantalum (Ta) from the waste tantalum capacitor,

which usually exists in small appliances [98]. However, the removal of Ta is comparatively tricky due to the tight bonding of mold resins. Niu et al. studied the two environmentally friendly approaches of SCW, i.e., SCWD and SCWO [98]. The results obtained by SCW treatment indicate that the mold resin packing was easily degraded by SCW treatment. However, the SCWO showed more decomposition efficiency than SCWD. After SCWD treatment, the color of the mold resin was changed to black, and some cracks were formed, indicating that the mold resin was damaged and a significant segment of the organics was carbonated. The mold resin was destroyed after SCWO treatment, and the  $SiO_2$  package became powders. The Ta electrode, Ni-Fe terminals, and  $SiO_2$  powder could quickly recover. Additionally, the Ta electrode, along with Ni-Fe terminals was separated. This showed the superior organic degradation efficiency of SCWO treatment. After SCWD treatment, Ta powder could be effectively recovered by mechanical processing, whereas after SCWO treatment Ta electrode could be easily recovered with a purity of 93.18 %. The optimized process conditions for both processes are mentioned in Table 4.

In another study, Xiu et al. investigated the efficiency of SCWO and SCWD pre-treatment coupled with acid leaching to recover metal from waste PCBs [96]. At supercritical conditions, the organics and water form a single homogenous phase, reducing the potential mass transfer limitations [82]. The hazardous organics matters present in waste PCBs can be decomposed by SCWO treatment, and bromine atoms present in

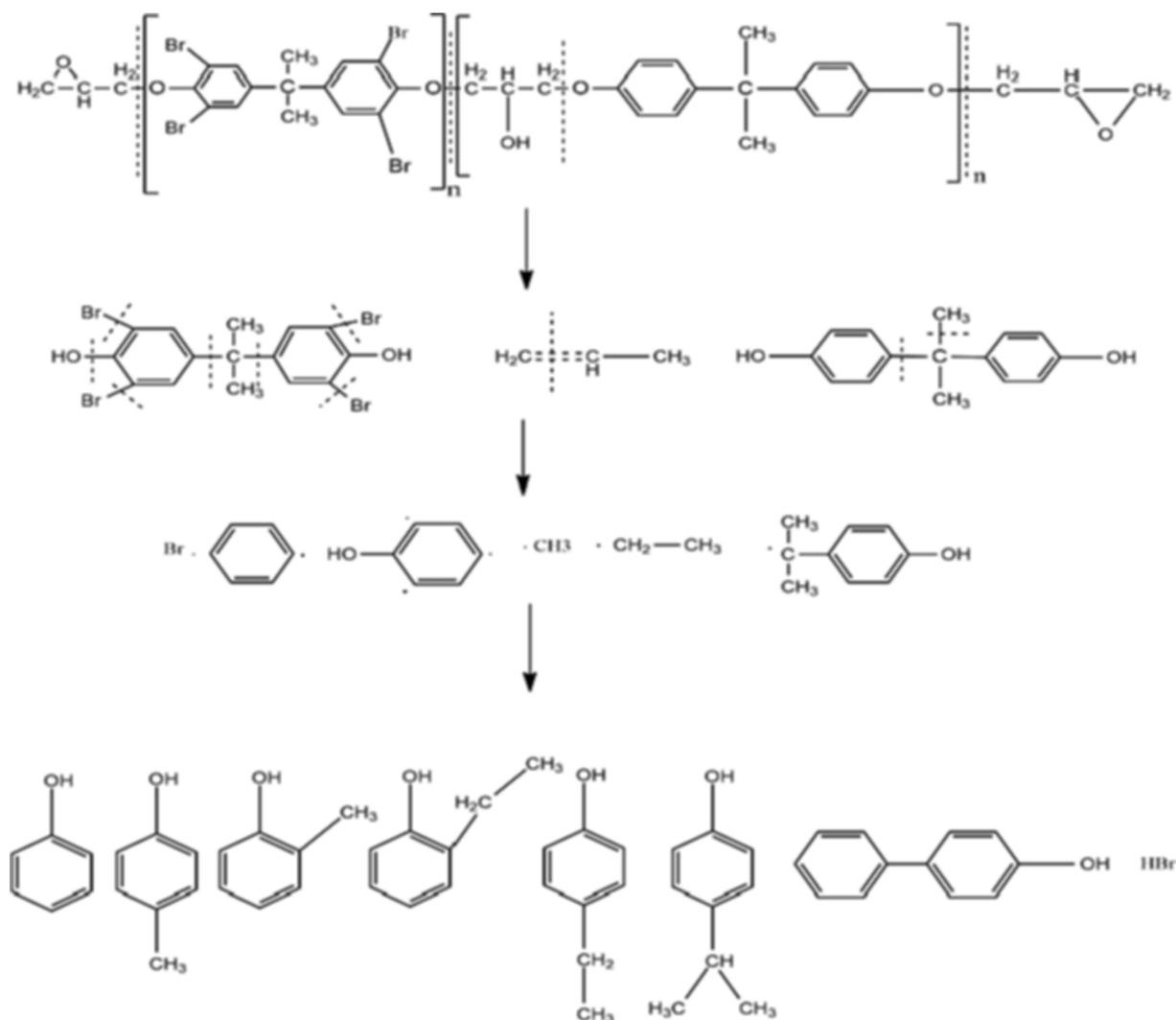


Fig. 10. Possible degradation and debromination mechanism of BER in SCW condition “Adapted with permission from [82], copyright (2022) American Chemical Society”.

**Table 4**

Summary of research work done on e-waste and plastic waste treatment using sub-critical and SCW.

E-waste	Reaction condition	Degradation content	Major product	References
LCD	400 °C, 23 MPa, 5 min	Polarizing film	99.77 % organics were eliminated, 78.23 % acetic acid.	[102]
Plastic shell	420 °C, 22.5 MPa, 60 min	BFR	95.7 % debromination efficiency, 60 % oil production (phenol and its derivatives)	[101]
RAM	495 °C, 33 MPa, 305 min	Brominated epoxy resin	Phenol and its derivatives, metal (Cu, Au)	[82]
Tantalum capacitor	(SCWO) 400 °C, 25 MPa, 90 min	Mold resin	Phenol and its derivatives, metal(Ta)	[98]
Tantalum capacitor	(SWD) 425 °C, 25 MPa, 120 min	Mold resin	Phenolic compounds, metal(Ta)	[98]
Integrated circuits (ICs)	500 °C, 23 MPa, 90 min	BER	95.51 % conversion rate, phenol and its derivatives	[103]
PCB	519 °C, 25 MPa, 10 min	BER	Phenol and its derivatives, Cu	[100]
Polycarbonate/ acrylonitrile–butadienestyrene blends (PC/ABS)	500 °C, 90 min, 23 MPa, 100 % excess oxygen	Debromination of PC/ABS	Maximum weight loss rate and debromination rate of PC/ABS were 78.57 % and 99.62 %, respectively.	[104]
Poly(ethylene terephthalate) (PET)	Autoclave reactor, 300 °C, 30 min	Depolymerisation of PET	90 % terephthalic acid, 60 % ethylene glycol	[105]
Nylon-6	300 °C, 85 min, 3 % phosphotungstic heteropoly acid as catalyst	Conversion of Nylon-6	Monomers and 77.96 % ε-caprolactam.	[106]
High impact polystyrene resin	SCWO: 500C, 23 MPa, 60 min, 200 % excess oxygen	Decomposition of HIPS and debromination of decabromodiphenyl ethane (DBDPE)	HIPS 99.12 %, debromination rate of decabromodiphenyl ethane (DBDPE) 99.38, antimony trioxide	[107]
Polystyrene	420 °C, 320 bar,	Degradation of Polystyrene	Toluene, ethyl benzene, styrene monomers, triphenyl benzene, and others	[108]
Fiber-reinforced plastic	275 °C, 60 bar, <40 min	Decomposition of fiber-reinforced plastic	Propylene glycol and phthalic acid and their secondary product.	[109]
Carbon-fiber reinforced plastic	405 °C, 280 bar, 120 min	Degradation of carbon-fiber- reinforced plastic	99.5 % epoxy resin removed, carbon fiber.	[110]
Polycarbonate	300 °C, 60 min, additives flame retardant decabromodiphenyl ether, and di- <i>n</i> - octyl phthalate	Depolymerisation of polycarbonate	Bisphenol A and phenol	[111]

BFR can be easily removed. On the other hand, the recovery of the valuable polymeric materials can be made simultaneously using SCWD treatment because the SCW is an outstanding hydrolysis agent [96]. Furthermore, SCW treatment can efficiently help in the enrichment of metals present in e-waste. The leaching was performed with diluted HCl (1 mol/L) at 60 °C for 80 min and at a stirring rate of 300 rpm. Both of these methods showed a significant result in the recovery of metals. The recovery of Pb and Cu was increased by increasing the temperature of the pre-treatment process. The recovery of Cu and Pb at a temperature of 420 °C was 99.8 % and 80 %, respectively, in the case of the SCWO process, and 90 % for Sn, Cr, Zn, Cd, and Mn in the case of SCWD pre-treatment process at a temperature of 440 °C.

Wang et al. investigated the SCWG to treat discarded circuit boards [99]. The optimized process conditions were reported as 700 °C temperature, 10 min reaction time, 5 % feedstock concentration, and 23 MPa pressure in the presence of FeOOH as additives. The presence of additives assists in efficient debromination and reduces the formation of pollutants such as dioxins and other volatile pollutants. There were different products formed at different temperatures. At 400 °C, H<sub>2</sub>, CH<sub>4</sub>,

and HBr gas molecules formed in smaller amounts. With further increase in the temperature, phenol and its derivatives were produced at 500 °C. With the increase in temperature to 700 °C and reaction time to 20 min, the conversion rate of H<sub>2</sub> increased. Bromine atoms are found in all phases. The temperature was an essential parameter for the bromine transfer from the solid to the liquid phase. Prolonged time resulted in the formation of inorganic bromine from organic bromine [99].

Various other research groups have also investigated the application of SCW for e-waste treatment, where the feasibility and effect of SCW have been explored [100,101]. The outcomes depict that SCW is an excellent eco-friendly solvent for resource recovery and organic detoxification. Table 4 depicts the summary of research work performed using water as a subcritical and supercritical solvent to treat e-waste and plastic waste.

Overall, it has been noticed that e-waste can be treated in two common ways in the SCW technique, i.e., SCWO and SCWD. Due to its unique properties, as discussed before, the polymer dissolution capacity increases in the SCW operation, eventually leads to water diffusion in the molten phase. In SCWO, the combined effect of oxidation and

hydrolysis causes the decomposition of organics. Also, the small organic molecules oxidized to form small organics in SCWO. Whereas in SCWD, hydrolysis is the main reaction for the decomposition of organics. Organics of high molecular weight depolymerize to form smaller-weight compounds. SCW technique can efficiently dehalogenate the compounds from e-waste, it can also efficiently produce the desired yield of fuels, and can effectively recover the metallic fraction of e-waste. Moreover, it has been observed that in e-waste treatment, the temperature and reaction time have more impact on the SCW treatment than reaction pressure in most studies.

#### 4.2.2. SCW for resource recovery from plastic waste

SCW is one of the most environmentally friendly and effective processes for converting waste plastic into a wide range of valuable products [87,112]. Zhang et al. showed that the temperature and residence time significantly affect the products [113]. The 79 % yield of oil products was reported at 530 °C, and the yield of gaseous products varied from 3 % to 22 % with an increase in temperature from 500 °C to 550 °C. Alkenes, alkadienes, and alkanes were the primary liquid-oil products in the continuous SCW reactor. Su et al. studied the decomposition of polyethylene (PE) into valuable oils in SCW conditions [114]. The authors reported that the SCW process is quick and efficient, leading to 90 wt% oil yield under the optimal condition of 460 °C temperature, 1 min of residence time, and water/PE ratio of 6. The water/PE ratio varies the constituents of oil products and influences the yield of products.

Supercritical water partial oxidation (SCWPO) technology is another technique that utilizes the properties of SCW in the presence of an oxygen-deficient environment to oxidize the polymer partially [115]. Using SCWPO, the emerging waste polystyrene can be efficiently depolymerized into valuable products such as oligomers, monomers, and other valuable hydrocarbons in a shorter residence time [116]. Onwudili & Williams investigated the decomposition of BFR plastics (Br-ABS and Br-HIPS) in SCW at 450 °C temperature and 31 MPa pressure [117]. The impacts of alkaline additives such as NaOH and Ca(OH)<sub>2</sub> were also studied. It was reported that the oil obtained from the supercritical treatment of both plastics has a similar composition and mainly consists of toluene, ethylbenzene, cumene, acetophenone, naphthalenes, diphenylpropane, phenyl-naphthalenes, diphenylcyclopropane, and a variety of multi-benzene compounds. Ethylbenzene was the major component of the oils obtained from both plastics. Furthermore, around 99 wt% of the bromine was eliminated and transferred into the aqueous phase from the waste plastics. In the gaseous phase, CO<sub>2</sub> was the main compound of Br-ABS. Also, both plastics gave rise to hydrocarbon gases and hydrogen of which alkane gases were significant compounds. NaOH additives make the oil product free from bromine and antimony content. It has been observed that NaOH as an additive is also more effective than Ca(OH)<sub>2</sub> in the elimination of organobromine or in suppressing their formation in oil. In both cases, the use of alkali additives leads to a decrease in gas production which marginally increases the solid residue. At low concentrations and in the absence of NaOH, Br-HIPS produces more char than Br-ABS. The addition of additives does not significantly affect the yield and composition of oil products. Additives were majorly assisting in the ionic reactions and the inorganic species released from the plastic materials during their decomposition. Additionally, alkaline additives, have majorly enhanced the removal of bromine atoms from the liquid product. Some other studies have explored the effect of different additives such as KOH, Na<sub>2</sub>CO<sub>3</sub>, etc., to understand the degradation of organics in SCF technology [101,118,119].

Recently, Chen et al. investigated the liquefaction of polypropylene (PP) into valuable oil using SCW [87]. The authors employed model PP and varied the temperature in the range of 380–500 °C at a pressure of 23 MPa while the residence time varied from 0.5 to 6 h. The authors reported 91 wt% yield of oil under the optimum conditions of 425 °C temperature, 23 MPa pressure, and 2–4 h of reaction time. A similar

yield was reported at 450 °C temperature but at a lesser reaction time of 30–60 min. Increasing reaction temperature (>450 °C) and reaction time (>4h) form more gaseous products. The liquid products were composed of olefins, cyclic, paraffin, and aromatics. The oil product has a similar boiling point range as naphtha, i.e. (25–200 °C) and 48–49 MJ/kg of heating values.

The authors have proposed a possible reaction mechanism as depicted in Fig. 11 [87]. The possible reaction pathway of converting PP by SCW liquefaction includes depolymerization in the first step depicted by (a) in Fig. 11. The other reactions, such as cracking, hydrogenation/saturation, cyclization, aromatization, gasification, and dehydrogenation are denoted by (b), (c), (d), (e), (f), and (g), respectively. Box marked with green color depicts oil products and box marked with red color depicts gas products. The width of arrows illustrates the relative quantity of products formed. Ethane, propane, propene, butane, and pentane were reported as the major components of the gaseous product. The liquid product mainly comprises of saturated aliphatics (C<sub>6</sub> – C<sub>11</sub> paraffins), unsaturated aliphatics (C<sub>6</sub> – C<sub>13</sub> olefins), cyclic (C<sub>6</sub> – C<sub>15</sub>), aromatics (C<sub>8</sub> – C<sub>9</sub>), and C<sub>5</sub>-C<sub>10</sub>. The proposed reaction pathways depict the major intermediates, and they may not depict all reactions occurring under SCW treatment. Firstly, at 425 °C, the PP quickly depolymerizes into oligomers in less than 30 min. Additionally, with the increase in the time from 30 min to 240 min, unsaturated aliphatics transferred into cyclic compounds. Simultaneously, some unsaturated aliphatics (olefins) may become saturated aliphatics (paraffins) and aromatics. In addition, the authors reported that with SCW, the conversion of PP into valuable products is net-energy positive, emits lower greenhouse gaseous than conventional processes, and has potentially higher energy efficiency. The oil produced from PP is beneficial and can be used as gasoline blend stocks or feedstocks for various other compounds.

Huang et al. investigated the sub-critical water gasification study for the depolymerization of reagent-grade and industrial-grade polycarbonate (PC) in the presence of plastic additives [120]. The major difference between industrial-grade and reagent grade is that industrial-grade PC have lower purity and a broad range of molecular weight distribution containing high content of low molecules. The additives i.e. flame retardant (decabromodiphenyl ether, DBDPO) and a plasticizer (di-*n*-octyl phthalate, DnOP) were chosen for the depolymerization of PC. Bisphenol A (BPA) and phenol were the main products of PC depolymerization in the presence or absence of the additives. In the absence of additives, the yield of BPA for reagent-grade PC was found as 40.13 % at 299.85 °C for 30 min of reaction, whereas for industrial-grade PC, the yield of BPA was observed as 30.9 %. For reagent-grade PC, the addition of additives for DBDPO yields only 35 % of BPA, while the addition of DnOP results in only 19.8 % of BPA. In the presence of both the additives, the yield of phenol increases while the yield of BPA is reduced. The yield for phenol was higher for reagent-grade and industrial-grade PC at 299.85 °C in 1 h of reaction time [120]. Furthermore, the authors reported the activation energies for PC depolymerisation with DBDPO additives as 116.48 kJ/mol, whereas the activation energy for DnOP additives as 147.17 kJ/mol. The activation energy for PC depolymerisation in the absence of any additives was 125.68 kJ/mol. Hence the authors concluded that the addition of DBDPO results in the reduced activation energy for depolymerization of PC in sub-critical water.

Based on the above discussion, it can be concluded that SCW technology is an emerging and promising sustainable technology with several applications and implementations for the treatment of plastics. The full technical potential of SCW technology for plastic treatment has yet to be discovered, and it can be an attractive option for the treatment of both e-waste and plastic waste. Fig. 12 summarises the application of the SCW process for the recovery of resources from e-waste and plastic waste. Overall, it can be stated that the SCW is effective for the degradation or decomposition of polymers into their monomers for plastic waste treatment. Temperature, S/L ratio, and time are the important factors found in SCW operations for the treatment of plastic waste.

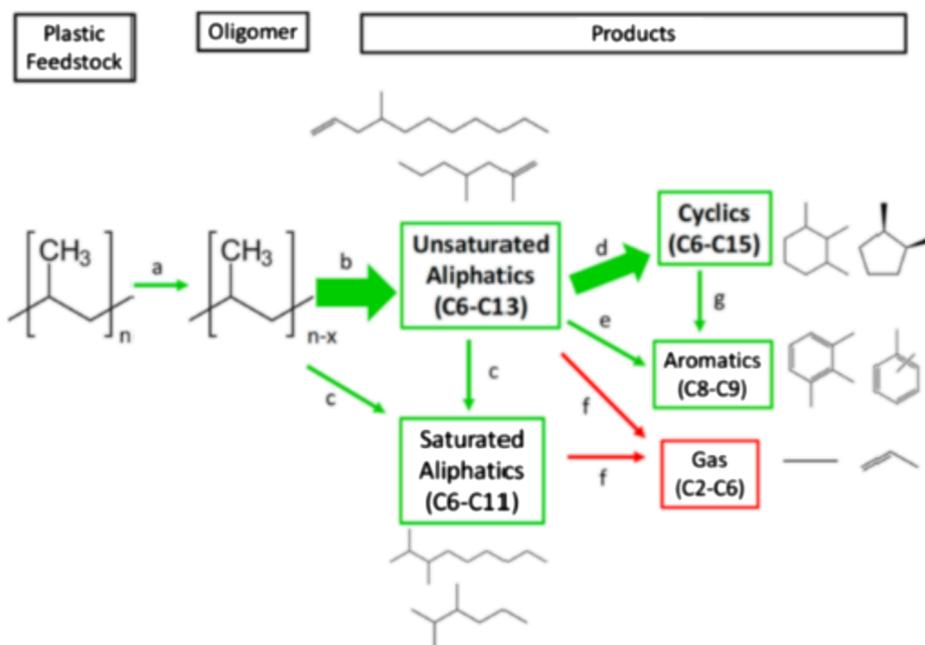


Fig. 11. Possible reaction pathway of converting PP via SCW liquefaction “Reprinted with permission from [87], copyright (2022) American Chemical Society”.

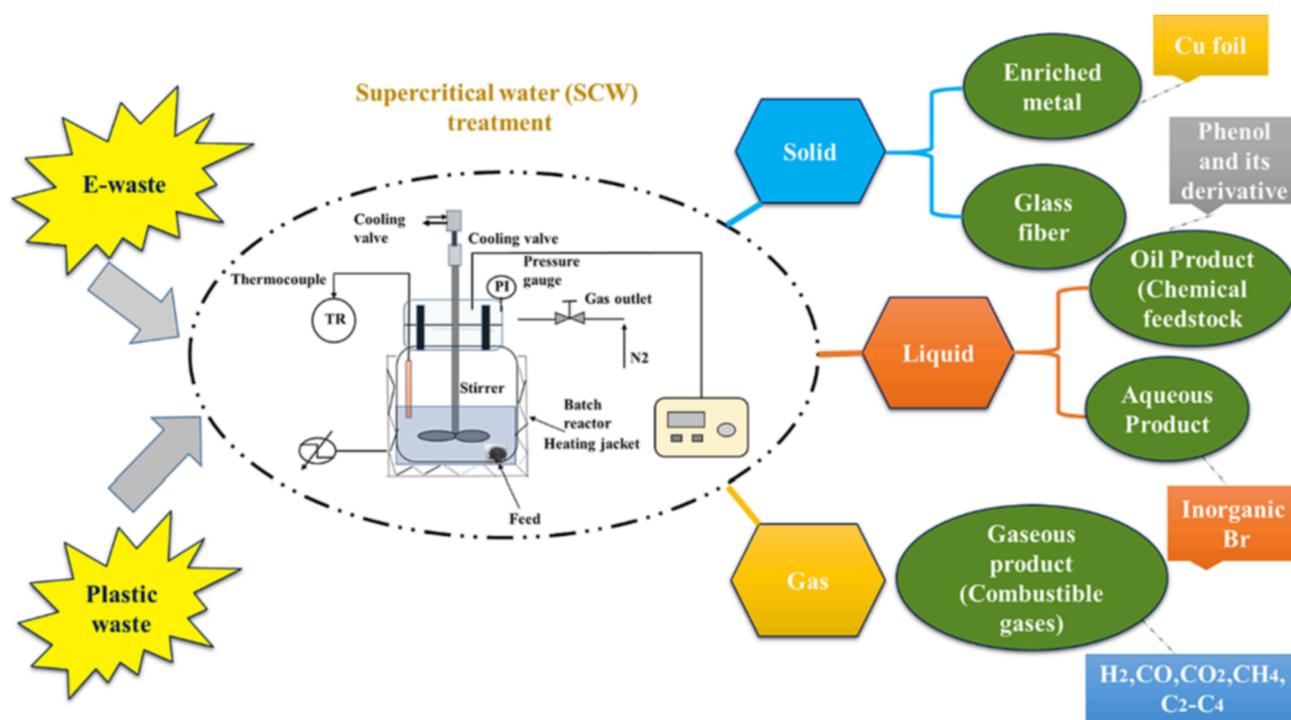


Fig. 12. Resource recovery from e-waste and plastic waste using subcritical/supercritical water treatment [44,82,121,122].

Selectivity and composition of (liquid or gas) product may also be influenced by these parameters along with the catalyst or additives used in the reaction. Pressure has less impact on the SCW operation in plastic waste treatment. Moreover, with or without the addition of any catalyst or additives, SCW is found to be more efficient in producing gas or liquid yield as compared to conventional processes, such as pyrolysis, etc., at the same process variables.

#### 4.3. Application of alcohol and other solvents in SCF technology for resource recovery from e-waste and plastic waste

Apart from SCW and  $\text{scCO}_2$ , several other solvents have been used in SCF technology, such as ethanol, methanol, acetone, etc. [101,123]. Solvents such as methanol, ethanol, etc., have a lower critical temperature, and pressure than water, e.g., methanol has  $T_c = 239^\circ\text{C}$  and  $P_c = 8.09\text{ MPa}$ . The use of methanol over water has some advantages, such as 1) lower critical temperature and critical pressure will be helpful to choose a wide variety of options for reactor's materials of construction,

2) the boiling point of methanol is less than that of water, and this will make the separation of products easier [124].

#### 4.3.1. Resource recovery from e-waste using supercritical alcohol and other solvents

Xiu and Zhang investigated the application of supercritical methanol (SCM) to recycle waste PCB [125]. It is reported that polymeric materials were converted into liquid products, and the metals were enriched in the solid product. The liquid product mostly contains phenol, phosphate fire retardant additives such as triphenyl phosphate, and the methylated derivatives, which increase with an increase in reaction temperature up to 400 °C. The operating conditions of SCM treatment were 300 to 420 °C temperature, reaction time of 30–120 min, and a solid–liquid ratio of 1:10–1:30 (g/mL). A purity of 55 % phenol was reported under 380 °C temperature, the solid–liquid ratio of 1:20, and 30 min of residence time. The solid product concentrates up to 62 % under the optimum conditions and majorly consists of copper, followed by iron, tin, lead, zinc, and a smaller amount of valuable metals such as silver and gold.

Xiu et al. investigated the application of SCM for the recovery of ultrafine copper from waste PCBs [126]. Methanol can be easily recycled by a simple distillation process and can be reused up to 5 cycles in the reaction process. Using nitric acid, waste PCBs were pre-treated twice. Sn and Pb were extracted after the first pre-treatment. After the second nitric acid pre-treatment, copper ions were introduced to the SCM treatment. The SCM treatment was carried out in the high-pressure reactor at 300 °C and 360 °C, and the time was varied from 5 to 15 min. Increasing the SCM temperature could produce smaller copper particles, whereas lower temperature agglomerates the copper particle; no particles were formed below 200 °C. The average diameter of a particle was 300 nm. The optimum condition of SCM was 300 °C temperature, 28 MPa pressure, and a residence time of 10 min. In the SCM process, the production of zero-valent copper can be assigned to reducing copper ions, which may be favorable to the nucleation and increase metallic nuclei [126]. This study showed that the SCM process is efficient, simple, and effective for the recovery of ultra-fine copper without using any surfactants or reducing agents. Due to the reducing property of SCM, copper ions in the leach liquor of waste PCBs had reduced to zero-valent copper. Some zero-valent copper could be oxidized to Cu<sub>2</sub>O due to the presence of Fe<sup>3+</sup> in the leach liquor. Ultrafine, uniform, monodispersed, and pure zero-valent copper could be obtained by removing Fe<sup>3+</sup> from the leach liquor. The presence of Fe<sup>3+</sup> in the leach liquor increases the growth of ultrafine copper in SCM. After separating ultrafine copper particles, a small number of other metals such as Zn, Fe, and Ni could be recovered by ion exchange, electrochemical or precipitation method from the leach liquor.

Recently, Preetam et al., have investigated the effect of subcritical to supercritical range (150 °C –300 °C) for the conversion of organics from WRAM and WPCB as well as the enrichment of metallic fraction [26]. The optimum conditions were reported as 300 °C, 3 h, and S/L ratio of 1:20 at autogenous pressure with atmospheric pressure in the beginning. Terephthalic acid and phenol are the prime compounds observed in the liquid product. High amount of Terephthalic acid was recovered at lower temperature whereas, phenol was found at an intermediate temperature and time. More than 90 % of the concentrated metals such as Cu, Ni, silver, zinc, and gold were recovered. The authors also compared the SCM process with pyrolysis and have observed that Br-free liquid product with better metal enrichment was recovered under the same condition. An economic estimation suggests that SCM has the potential

to efficiently enrich metals and treat the organics fraction by converting them into oil at a lower temperature and hence at a lower cost.

In another article, Xiu et al. discussed the critical water-alcohol medium as an effective method to recover valuable chemicals from waste PCBs [127]. The critical water system has disadvantages when utilized for degradation and debromination of waste PCBs such as the requirement of the high critical condition, low value-added oil products, and at a lower temperature, it gives midway debromination. To overcome these drawbacks, the critical water-alcohol combined process was investigated. Critical water–ethanol (CWE) and critical water-methanol (CWM) were employed for the waste PCB treatment. The temperature of the water-alcohol system plays a crucial role in the oil product constitution. The optimal debromination parameter of CWE/CWM was reported as 350 °C temperature, 60 min of reaction time, a solid-to-liquid ratio of 1:20 g/mL, and (H<sub>2</sub>O)/ (CH<sub>3</sub>OH) ratio of 2:1 (v/v). Br free oil was obtained above 300 °C during CWM/CWE process. At a temperature of 300 °C CWE/CWM, 4-(1-methylethyl)-phenol in high concentration was obtained. The temperature has a considerable impact on the oil phase composition. At 400 °C, in CWE condition, high content of 4-(1-methylethyl)-phenol and 4-isopropyl-phenetole were reported. At 450 °C, high content of 2-ethyl-phenol was reported. At 400 °C, in the CWM condition, no phenol and its derivative were formed, and anisole is observed in high content. At 450 °C, p-xylene and multi substituted alkylation phenol were reported. A summary of research work carried out using alcohol as solvents in SCF technology for the recycling of e-waste and plastic waste is depicted in Table 5.

On the whole, it can be concluded that the utilization of alcohol or other solvents in SCF technology is a feasible, eco-friendly, and promising option for the treatment of e-waste. Using alcohol or other solvents can significantly reduce the operation condition of the process, which leads to reduce the energy consumption and helps in promoting sustainability. The use of these solvents even provides efficient results of polymer degradation or recovery of metals without the use of any chemical reagent, and also the solvent used in the reaction can even be efficiently recycled after the treatment.

Based on the above discussion, it can be implied that SCF technology provides an attractive and efficient alternative for e-waste treatments along with the degradation of harmful organics into valuable and non-toxic products. In addition, the SCF technology also facilitates the recovery of metals from e-waste. Fig. 13 summarises the overall possible implementations of SCF technology in the treatment of e-waste [35].

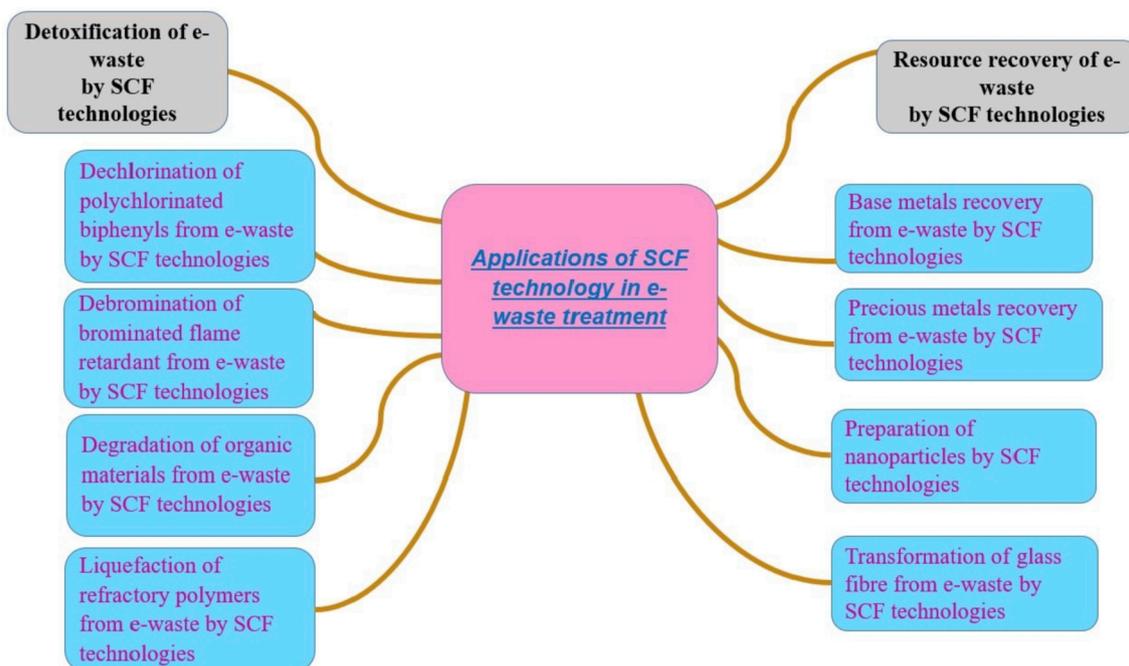
#### 4.3.2. Resource recovery from plastic waste using supercritical alcohol and other solvents

The degradation of different polymers can be performed easily and quickly by solvolysis in subcritical or SCFs [134]. For example, condensed polymers such as PET and nylon 6 can be simply depolymerized by solvolysis [135]. Solvolysis can be either hydrolysis for SCW, alcoholysis for supercritical alcoholic solvent such as methanolysis in SCM, or named as per the respective solvent [136]. In SCW or SCM, condensation polymers having linkages such as ester linkage, ether linkage, or acid amide linkage are simply degraded into their monomers by solvolysis [137]. Goto et al. have investigated the degradation of PET using SCM to recover the monomers [135]. The optimal condition was applied in a batch reactor at a temperature of 573 K at the pressure of 20 MPa under the reaction time of 2–120 min. Methanolysis using SCM decomposes PET to its monomers, i.e., dimethyl terephthalate (DMT), and ethylene glycol (EG). Significant reactions involved in the monomerization are as follows:

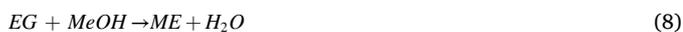
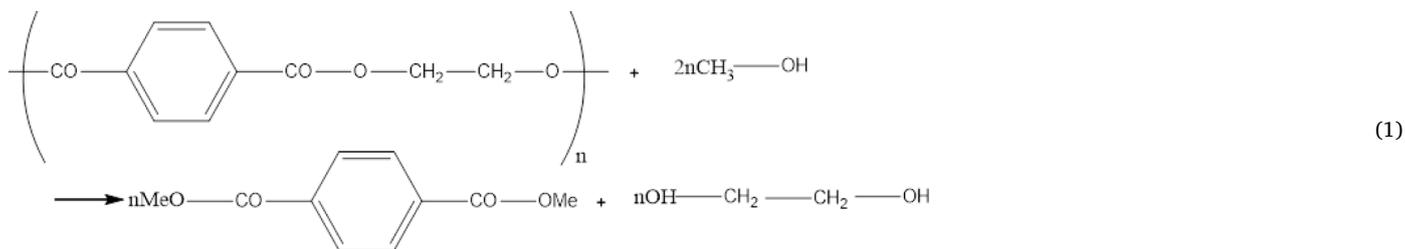
**Table 5**

Research work for e-waste and plastic waste treatment by using subcritical and supercritical alcohol and other solvent in SCF technology.

SCFs	E-waste	Reaction condition	Degradation content	Major product	References
Methanol	PCB	380 °C, 30 min, 8.03 MPa	BER	Phenolic compounds, 67 % conversion efficiency, 58 % purity of phenol, 41 % of HBr, metal concentration up to 62 %	[125]
Methanol	Plastic shell	420 °C, 60 min, 22.6 MPa	BFRs	Phenolic compounds	[101]
Isopropanol	Plastic shell	400 °C, 18 MPa, 60 min	BFRs	60 % oil product, 42 % mixture of aromatics, and 21 % oxygen- containing compounds (benzene derivatives and phenol)	[101]
Acetone	Plastic shell	400 °C, 11.6 MPa, 60 min	BFRs	Phenol and its derivatives	[101]
Ethanol	PCB	300 °C, 6.14 MPa, 180 min	BER	62.4 % phenol	[44]
Aqueous ammonia (Near-critical)	PCB	300 °C, 1:15 g/ml, 60 min, 4.1 mol/Litre ammonia concentration.	BER	99.9 % debromination efficiency, liquid product: pyrazine, pyridine, and other nitrogen-containing fine compounds. Metal: Cu foil, Pb = 82.7 %, Sn = 73.8 %, Zn = 92.5 %, and glass fibers.	[128]
Acetone	Epoxy resin	320 °C, 1 MPa, 20 min	Decomposition of epoxy resin	95.6 % decomposition efficiency, carbon fibers	[129]
Ethanol	Food package composed of Polyethylene, Aluminium, PET as multilayer film.	Delamination with acetone: 50 °C, 4 h Depolymerisation with supercritical ethanol: 255 °C, 11.65 MPa, 120 min	Delamination of multilayer film and depolymerisation of PET	80 % diethyl terephthalate	[130]
Methanol	Cross-linked polyethylene	360 °C, 15 MPa, 10 min	Decross-linking polyethylene	Thermoplastic polyethylene	[131]
Methanol	Polycarbonate	220 °C, 10 min	Depolymerisation of Polycarbonate	Monomers of bisphenol A and dimethyl carbonate	[132]
Methanol	Poly(ethylene terephthalate)	269.85–300 °C, 0.1–15 MPa, 30–60 min	Depolymerisation of PET	Ethylene glycol, 98 % dimethyl terephthalate, and complete depolymerisation of PET	[133]



**Fig. 13.** Utilities of SCF technology in e-waste treatment.



Here, MHET, DMT, EG, TAMME, ME, and DEG are methyl 2-hydroxyethyl terephthalate, dimethyl terephthalate, ethylene glycol, terephthalic acid monomethyl ester, 2-methoxyethanol, and diethylene glycol, respectively [137]. Fig. 14 shows the reaction for PET decomposition into oligomers and monomers. The two reaction pathways depict the decomposition of polymers into monomeric species. First is random decomposition, illustrated by binary scission of bonds at some location

along the chain. Second, by scission at the chain tip, the extreme is a particular reaction, which liberates monomeric species of the polymer.

Goto investigated the fundamental and commercialization aspect of waste plastics recycling by degradation reactions in subcritical and SCFs [138]. The authors reported that the degradation reactions proceed quickly and selectively in SCF technology compared to traditional processes. For plastics recycling, pilot-scale or commercial scale set-up utilizing subcritical and SCFs has already evolved. As we know, condensation polymerization plastics can easily be depolymerized to their monomers in SCW or alcohols, and selective decrosslinking reactions can also recycle cross-linked polymer in SCFs without severe degradation of the backbone chains.

Depolymerization of the resin composition can be applied to recycle fiber-reinforced plastics to recover fibers and monomers [42]. However, carbon-fiber-reinforced plastic (CFRP) can be hard to recycle due to the presence of polymers such as epoxy resin which is a type of thermosetting plastic. Hence, developing a more sustainable and efficient process for recycling CFRP is necessary. Okajima & Sako established an excellent recycling process for CFRP containing epoxy resin and explored its degradation by utilizing superheated and supercritical acetone [42]. Initially, the correlation between the pressure and degradation of epoxy resin in the CFRP was investigated at a temperature of 350 °C, decomposition efficiencies increased for the first 60 min then reduced with reaction time due to the carbonization of the

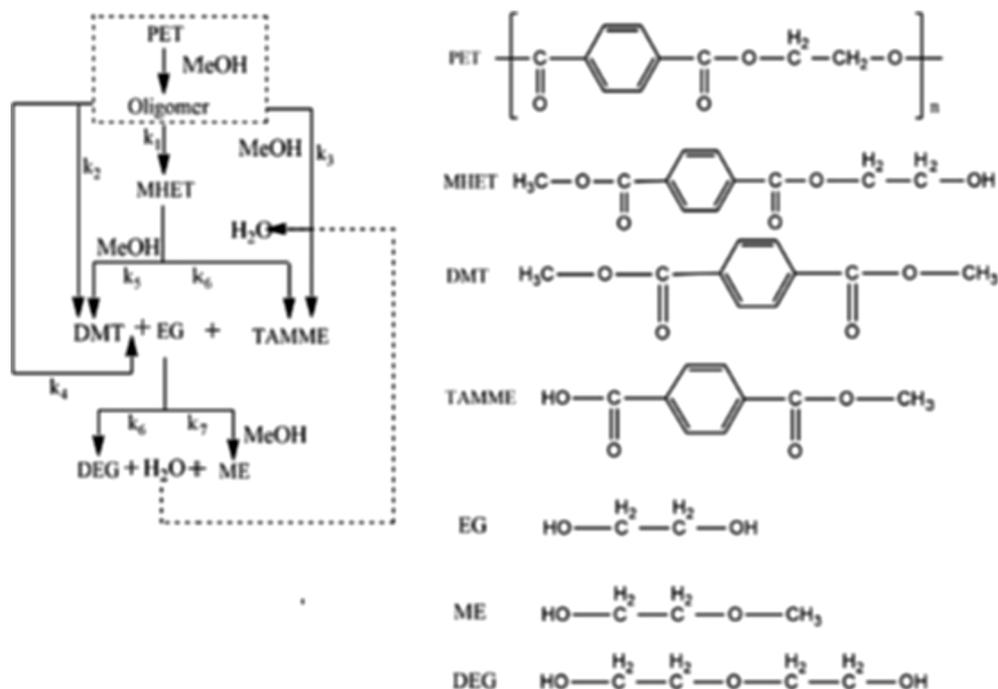


Fig. 14. Reaction pathways for decomposition of PET in methanol “Adapted with permission from [135], copyright (2022) American Institute of Chemical Engineers (AIChE)”.

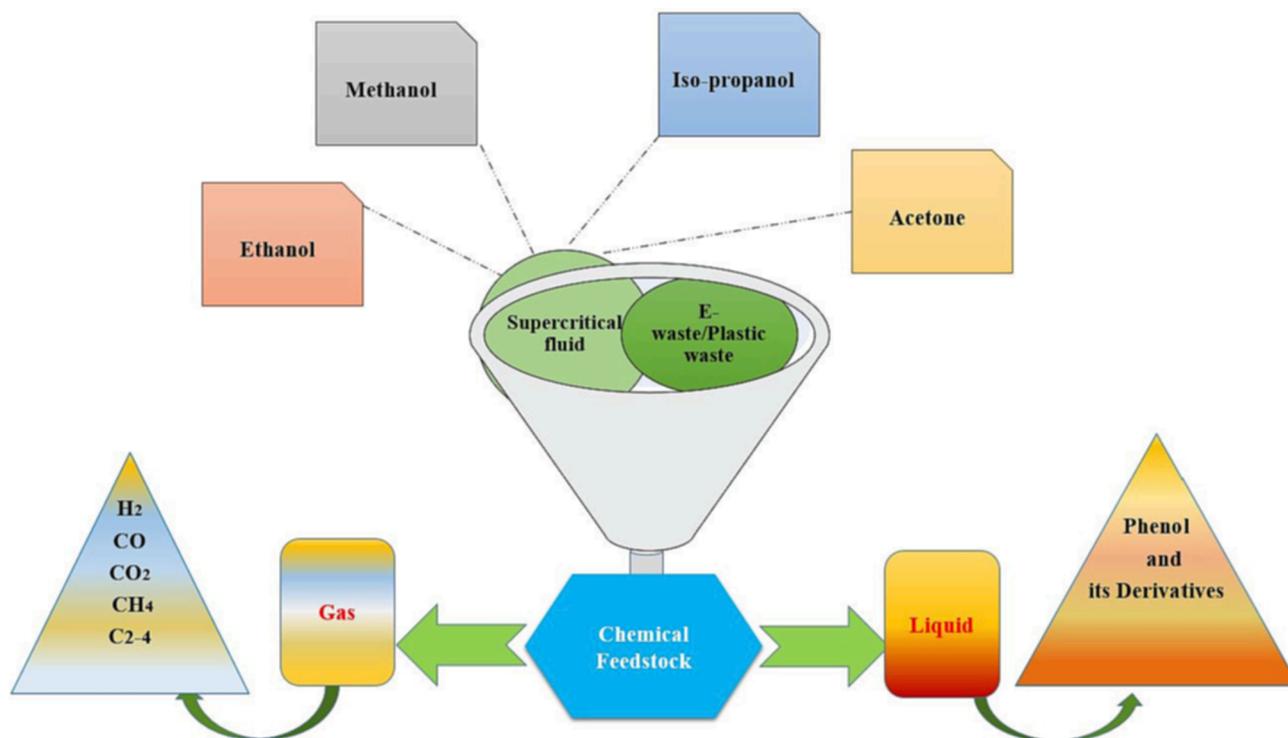


Fig. 15. Degradation of polymers of e-waste and plastic waste in supercritical alcohol system [44,101,119].

degradation products.

Qi et al. in their work utilized transfusion tube (TFT) and sample collector for urine (SCFU) as PVC-medical waste and subjected to near-critical methanol for dechlorination and recovery of additives, for example, plasticizer, stabilizer, and lubricants [40]. The optimum temperature for the reaction was found to be 250 °C, solid-liquid ratio: 1:10 in 60 min. The dechlorination reached up to 90 %. The dechlorination was found in order of TFT > pure PVC > SCFU. The additives such as dibutyl phthalate (DBP), dimethyl phthalate, and dioctyl phthalate could be efficiently recovered from these PVC-medical wastes. Fig. 15 depicts the degradation of polymers of e-waste and plastic waste in a supercritical alcohol system.

SCF technology has excellent potential as an environmentally friendly green process due to its unique properties as chemical reaction media. Therefore, for the treatment of waste plastic, the use of alcohol or other solvents in SCF technology can act as extraordinary reaction media for the degradation or depolymerization of polymers. Polymers can easily depolymerize or decompose into smaller components in SCF technology in alcoholysis in comparison to conventional processes. These solvents can even act as either excellent reactants or reaction mediums. Furthermore, rapid reaction rate and selectivity are also noticed in alcoholysis during SCF technology without introducing any catalysts or additives. This SCF technology system can potentially solve commercial challenges such as acidification, salt precipitation, etc., of SCF technology. Recycling technology for waste plastics is very desirable, and research has already been going on for the decomposition of plastics using SCFs, from fundamental research to practical applications. However, the main disadvantages of SCF technology are considered as high investment costs and commercialization. However, the solutions regarding the same are continuously evolving, such as recent developments in semi-continuous processing or multi-vessel extraction and many other proposals that attempt to overcome the drawbacks associated with it [56].

## 5. A comparative study, economic analysis, and future perspective

Different technologies have been employed to recover metals (pyrometallurgy, hydrometallurgy, or biohydrometallurgy) from e-waste or conversion of e-waste plastic or regular waste plastic into valuable products (pyrolysis, catalytic pyrolysis, gasification) [139–142]. None of the mentioned technology considers both the aspects of e-waste recycling, i.e., metal recovery and plastic conversion or efficient degradation. Also, there are environmental or technical drawbacks attached to each of the available methods, i.e., the pyrometallurgical process releases harmful gases, soots, dioxins, and hydrometallurgical methods that utilize toxic reagents and create secondary pollution. Bioleaching involves complex operations and has slower kinetics. Pyrolysis converts e-waste plastic into valuable products, and mainly produces liquid products (oil) containing halogenated compounds. Broadly, e-waste and plastic waste treatment processes are categorized as 1. Incineration, 2. Landfill, 3. Mechanical/physical recycling, and 4. Chemical recycling [63]. Table 6 compares common techniques utilized for the treatment of e-waste and plastic waste [2,143,144]. SCF technology provided an attractive choice in the chemical recycling process as an excellent chemical medium because of its unique physical and chemical properties. It was broadly used in numerous hydrothermal reactions such as gasification, depolymerization, synthesis hydrolysis, and hydrogenation [145]. SCF technology has an excellent ability for e-waste and plastic waste recycling due to its superior properties and ecologically benign methodology [35,119]. Subcritical and SCFs such as water, CO<sub>2</sub>, and alcohol are efficient reaction media for the depolymerization, degradation of plastics, and polymer decomposition [146].

At present, economic studies on SCF technology are scarce in the literature. Li and Xu reported that without involving any catalysts, the SCW technology was more effective and convenient than any conventional method [82]. The initial decomposition temperature of SCW treatment is less than that of pyro-metallurgy, which indicates the reduced cost of the process. Li and Xu evaluated a simple economic calculation of the laboratory research as illustrated in Table 7 [82]. As a

**Table 6**

Comparison of common technologies applied for e-waste and plastic waste treatment.

E-waste		
Technology	Advantages	Disadvantages
Physical Separation	Simple in operation, high separation rate of metals	Low content and dispersion, least selectivity.
Pyro-metallurgy	Simple operation. High recovery rate	Different metals fused, produce a large amount of SO <sub>x</sub> , NO <sub>x</sub> , soots, and other harmful gases.
Hydrometallurgy	Low equipment investment, simple in operation.	High consumption of chemical reagents, produces a lot of sewage pollution.
Bio-leaching	Environmental friendly, good selectivity.	Good selectivity for specific metals (Cu, Zn, Au), slow kinetics, low industrialization.
Pyrolysis	Efficient output generation	High-temperature requirement, low quality of liquid product
Electrochemical Technology	High selectivity, High recovery efficiency	Waste-water and residue pollution
Plasma Technology	Produce less dioxin and furans, treat high waste capacity, fast heating, high reactant, and transfer rates.	High electricity and energy requirements, require more space, economic consideration has become a barrier to treating waste. Contamination of products with carbon.
SCF Technology	Environmental friendly, high recovery rate, high efficiency	High initial investment
<b>Plastic Waste Technology</b>	<b>Advantages</b>	<b>Disadvantages</b>
Physical recycling	low recycling cost and simple operation	Garbage classification is required, labor cost is needed, requires policy support from the government.
Incineration	Simple in operation, mostly used.	Produce toxic gases and hazardous substances.
Pyrolysis	Feasible technology, efficient results	Requires a significant amount of heat energy, coke formation, regular maintenance of the equipment
Gasification	Formation of C <sub>3</sub> -C <sub>6</sub> , syngas, rich in H <sub>2</sub> and CH <sub>4</sub> , which can be utilised as a combustible gas.	Emission of harmful gases such as NO <sub>x</sub> . Requirement of high gas flow rate and temperature atmosphere, difficult separation, and lower calorific value of products. It contains impurities such as NH <sub>3</sub> , H <sub>2</sub> S, NO <sub>x</sub> , alkali metals, and significant amounts of tars, which need an additional purification step.
Biodegradation	Environmental friendly, efficient degradation of plastics.	Time-consuming, very selective in process, less industrialisation
SCF Technology	Environmental friendly, high recovery rate, high efficiency, solvent can be recycled, high quality of product.	Initial higher equipment requirement

result, it is reported that the profit of the SCW process was \$122.69/kg, which was more than that of pyro-metallurgy (\$55.52/kg) and a little less than that of hydrometallurgy (\$127.78/kg). Through this analysis, it can be observed that the SCW treatment has economic feasibility. Besides economic advantage, SCF technology is environmentally sound and acceptable. Additionally, as compared to other methods, it generates no secondary pollution.

Similarly, Liu and Zhang examined the economic feasibility and

**Table 7**

An economic evaluation of different processes "Adapted with permission from [82], copyright (2022) American Chemical Society".

Process	SCW process	Pyro-metallurgy	Hydrometallurgy
Apparatus cost (K\$)	100	600	100
Power (kW)	20	65	
Depreciation cost (\$/kg)	8.33	50	5.55
Service life (year)	10	10	15
Basic raw materials cost (\$/kg)	13	13	13
Electricity cost (\$/kg)	3.75	29.25	–
Chemical materials cost (\$/kg)	–	–	1.44
Profit (\$/kg)	122.69	55.52	127.78
Non-metals profit (\$/kg)	0.04	0.04	0.04
Metals profit (\$/kg)	147.73	147.73	147.73
Environmental effects	Rare	Soot, dioxins, and other harmful gases.	Waste-water

**Table 8**

Economic and environmental analysis of different processes [43].

Process	Pyrometallurgy	Hydrometallurgy	Co-treatment with SCW
Spent LIBs cost (Feed) (\$/kg)	10	10	10
Chemicals cost (\$/kg)	0	H <sub>2</sub> O <sub>2</sub> and acid	0
Metals recovery rate (%)	<95 %	<95 %	<95 %
Apparatus cost (K\$)	600	–	100
Power consumption (kW)	4	Not any.	2
Temperature (°C)	1000	60–80	350
External earnings	Not any	Not any	Derivative of benzene
Environmental impacts	Water-gas	Waste-water	None

environmental aspects using the subcritical water co-treatment process as shown in Table 8 [43]. Comparison has been made by process flows, effects of recovery, and operational energy consumption of these three processes. Hydrometallurgy involves the use of strong acids (such as HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, and HCl) or inorganic acids [147]. To convert Co<sup>3+</sup> to Co<sup>2+</sup>, the acceleration of leaching is needed for Co<sup>3+</sup> from LiCoO<sub>2</sub>, also the hydrometallurgy usually needs the addition of a reductant (e.g. H<sub>2</sub>O<sub>2</sub>). As compared to SCF technology, pyrometallurgy is energy-intensive as it employs higher temperature i.e. 700–1000 °C, which also increases the cost of the process. However, in subcritical technology, it takes place at a relatively low temperature and mostly it does not need any other reagents other than the waste feed. In this case, the feed is a waste PVC, and the temperature requires is only 350 °C.

Moreover, in SCF technology, the waste feed has the additional advantage of being converted to chemical feed or raw materials simultaneously. In environmental aspects, SCF technology does not lead to any harmful impact. At the same time, other technologies like pyrometallurgy produce toxic gases and thus cause secondary pollution to the environment, and hydrometallurgy also requires the use of toxic reagents like acids and other chemicals [148]. Liu and Zhang reported that the chlorine present in PVC bound with the metals to produce non-harmful inorganic chlorine after the reaction and did not generate any hazardous organic chlorine compounds [43]. Therefore, it has been found that subcritical co-treatment has a considerable benefit of technical, economic, and environmental as compared to that of the traditional recovery method. Thus, it has a broader development perspective for recovering metal from a waste feed.

SCF treatment is extensively applied on a small-scale or lab scale. Nevertheless, SCF technology for e-waste and plastic waste is less explored at the pilot scale. The characteristics of SCF technology require a high investment cost for implementation [35]. Furthermore, it has a few disadvantages, such as corrosion and salt precipitation, so industrial set-up and commercialization are still complex [149–151]. However, technical solutions are attached to every drawback of SCF technology [151]. For example, high temperature and density lead to an increase in corrosion, which can be resolved by (1) a cooling strategy to prevent its occurrence, (2) utilizing high resistance materials, (3) optimizing operating conditions by utilizing a base to preneutralize the flow of feed and adding quench water (4) eliminating all the corrosive aspects by pre-treating the feedstock [152]. In the same way, to deal with salt precipitation, measures that can be employed are: (1) improvements like reverse flow in a tubular reactor and tank reactor with brine pool can be done by upgrading and improving the reactor, (2) by applying technical modifications such as scraper rotating, high-velocity flow, mechanical brushing, cross-flow filtration, reactor flushing, density separation, etc. [153]. The energy recovery of the reactor effluent, such as the heat exchanger and circulation system, is an essential aspect of the economic feasibility of the process and can also help to save a good amount of energy and materials.

It can be concluded that SCF technology is considered as a practicable process that includes the environmental aspects and attained the removal of those hazardous wastes. Organic polymers in plastic waste or e-waste could be removed efficiently in a greener way. Furthermore, metals could also be recovered successfully by using a supercritical treatment. From an economical aspect, the design of SCF technology plants or a more extensive set-up had to upgrade by including an energy recovery system to gain the economic feasibility of the operation. In terms of the future of SCF technology, it is important to continue investigating and analysing the technical solutions to reduce the initial capital investment and operational cost to attain the further commercial development of this technology.

## 6. Conclusion

Environmental issues are a priority and cannot be ignored. E-waste and plastic waste are continuously increasing due to our changing lifestyle, convenience, and modernisation. Unregulated accumulation and improper disposal of e-waste and plastic waste lead to severe health impacts on humans and animals. Furthermore, these wastes can also cause environmental hazards as they contain toxic substances capable of leaching into the soil and water, thereby leading to environmental pollution. Hence, there is an urgent need to discover an eco-friendly route for the sound management and treatment of both e-waste as well as plastic waste. The recycling of e-waste and plastic waste is essential from the aspect of resource recovery and environmental protection. SCF technology is considered to be a feasible technique for the treatment of e-waste and plastic waste. The conventional method such as incineration, pyrolysis, and many other technologies used to recycle e-waste or the treatment of plastic waste lacks in maintaining the balance between efficient results for the recovery and environment-friendly approach. SCF technology can be a good alternative for treating these wastes because of its unique combination of properties. SCF can successfully degrade or detox hazardous compounds and recover valuable resources or chemicals simultaneously without generating any kind of pollution in the environment. SCF treatment showed promising results for both e-waste and plastic waste treatment. Hence, SCFs can be an efficient and sustainable substitute for treating e-waste and plastic waste. However, there are a few hurdles associated with the SCF treatment, such as high investment cost, corrosion, and salt precipitation, especially for industrial applications but for that, technical solutions are continuously developing and emerging with fruitful outcomes. Over the past few years, it has been observed that SCF technology is continuously evolving. Hence, it can be concluded that the application of SCF

technology has emerged to be novel, promising, and sustainable for the treatment of e-waste and plastic waste.

## CRediT authorship contribution statement

**Amrita Preetam:** Conceptualization, Methodology, Investigation, Writing – original draft, Writing – review & editing. **Prashant Ram Jadhao:** Conceptualization, Methodology, Writing – review & editing. **S.N. Naik:** Supervision, Funding acquisition, Writing – review & editing. **K.K. Pant:** Supervision, Funding acquisition, Writing – review & editing. **Vivek Kumar:** Supervision, Funding acquisition, Writing – review & editing.

## Declaration of Competing Interest

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

## Data availability

Data will be made available on request.

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## Appendix A. Supplementary material

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.seppur.2022.122314>.

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