

Metals recovering from waste printed circuit boards (WPCBs) using molten salts

L. Flandinet^a, F. Tedjar^{a,b}, V. Ghetta^c, J. Fouletier^{a,*}

^a LEPMI, UMR 5279, CNRS – Grenoble INP-Université de Savoie, Université Joseph Fourier, BP75, 38402 Saint Martin d'Hères, France

^b RECUPYL SA, Rue de la métallurgie 38420 Domène, France

^c LPSC, UJF Grenoble 1 – CNRS/IN2P3 – INPG, 53 rue des Martyrs 38026 Grenoble Cedex, France

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ABSTRACT

Recycling of waste electrical and electronic equipments (WEEE) has been taken into consideration in the literature due to the large quantity of concerned wastes and their hazardous contents. The situation is so critical that EU published European Directives imposing collection and recycling with a minimum of material recovery [1]. Moreover, WEEEs contain precious metals, making the recycling of these wastes economically interesting, but also some critical metals and their recycling leads to resource conservation. This paper reports on a new approach for recycling waste printed circuit boards (WPCBs). Molten salts and specifically molten KOH–NaOH eutectic is used to dissolve glasses, oxides and to destruct plastics present in wastes without oxidizing the most valuable metals. This method is efficient for recovering a copper-rich metallic fraction, which is, moreover, cleared of plastics and glasses. In addition, analyses of gaseous emission show that this method is environmentally friendly since most of the process gases, such as carbon monoxide and dioxide and halogens, are trapped in the highly basic molten salt. In other respects, under operation without oxygen, a large quantity of hydrogen is produced and might be used as fuel gas or as synthesis gas, leading to a favourable energy balance for this new process.

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1. Introduction

Waste electric and electronic equipment (WEEE) is diverse and complex, in terms of materials and components, and is increasing dramatically. Electronic waste is made of a mixture of various metals, particularly copper, aluminium and steel, mixed with various types of plastics, glass fibre-reinforced epoxy resin and ceramics [1]. Electronic equipment has a relatively short average lifetime, i.e., 2–3 years; consequently, large quantities of electronic waste need to be discarded [2–4]. Many countries and organizations have drafted national legislation to promote re-use, remanufacturing, and recovery of materials by recycling techniques to reduce the quantity of waste. As an example, in Europe, according to the directive 2002/96/EC, since January 2007, the rate of recovery will be increased up to at least 75 wt.% and components, materials and substances re-use and recycling will be increased to at least 65 wt.% per Information Technologies and Telecommunications equipment appliance [5]. Incineration of electronic waste by traditional incinerator is also dangerous. For example, copper is a catalyst for dioxin formation when flame-retardants are incinerated. Recycling is important, not only from the point of view of waste treatment, but also from the aspect of recovering valuable materials.

The proportion of waste printed circuit boards (WPCBs) is about 3% of electronic waste [2,6]. Unlike other solid waste, WPCBs contain a variety of heavy metals and hazardous substances (i.e., lead, cadmium, mercury, PVC, halogenated flame retardants, etc.) that may seriously pollute the environment. The typical composition of PCBs is non-metal (i.e., plastic, resins, glass fibres, etc.) >70%, copper ~16%, solder ~4%, iron, ferrite ~3%, nickel ~2%, silver ~0.05%, gold ~0.03%, palladium ~0.01%, and so on [6]. It can be clearly seen that except for the hazardous substances, a lot of valuable materials contained in WPCBs make them worth being recycled: the precious metals make up more than 70% of the value and copper ca. 20%. Therefore, developing a non-polluting, efficient, and low-cost processing technology for recycling of WPCBs cannot only avoid environmental pollution, but also help recycle valuable resources. However, PCB waste is a heterogeneous mix of polymer, metal and fibreglasses, which makes its recycling difficult. For example, over the years, PCBs have evolved from uncomplicated single and double-sided plated-through-hole (PTH) to multi-layered PCBs.

The recovery of precious metals from electronic waste is now carried out by physical separation processes [7–9], pyrometallurgical processing, hydrometallurgical processing, and biometallurgical processing [10–15]. High temperature Pyrometallurgy, i.e., 1200 °C, as usual technology requires high investments. NaOH can be used as slag formation material to separate metal from slag and decrease the melting temperature [16]. Low temperature Pyrolysis can be considered as an alternative method of recycling WPCBs, because in the pyrolysis process (heating without oxygen),

* Corresponding author. Tel.: +33 04 76 82 66 51; fax: +33 04 76 82 67 77.
E-mail address: Jacques.Fouletier@lepmi.grenoble-inp.fr (J. Fouletier).

the organic material is decomposed into low molecular products (liquids or gases), which can be used as fuel or chemical feedstock [17–20]. Vacuum pyrolysis has several advantages over other pyrolysis methods because of the short organic vapour residence time in the reactor and low decomposition temperature, which reduces the occurrence and intensity of secondary reactions [21].

Hydrometallurgical processes require various steps, including a series of acid or caustic as leaching media for solid material, followed by various separation and purification procedures [22–25]. However, if hydrometallurgy is now demonstrated as sustainable route for waste processing, unfortunately, extraction of precious metals and mainly gold is only possible in cyanide media.

Therefore, the traditional technologies cannot meet the future requirements of industry because of environmental contamination, high cost and low efficiency. Two recycling processes at industrial scale, i.e., Pyrocom and Haloclean, are used presently [14,26–30]. Developing new clean technologies for recycling valuable resources from WPCBs is of great significance, with two main objectives: saving in energy (using recycled materials in place of virgin materials results in significant energy savings) and reduction in pollutions.

The challenge remains the access to encapsulated metals as we are in front of complex matrix containing non-ferrous metals, precious metals, ceramics, thermo-polymers and resins. Molten salts offer the best available technology to catch metals encapsulated in other matrices. The main objectives of this method are:

- recovering the precious metal fractions, without any dissolution or melting,
- increasing halides retention
- restricting CO₂ emission, and
- developing an economically viable process.

Molten salt oxidation (MSO) has been proposed for coal gasification and destruction of hazardous organic mixed oils, chlorinated organic solvents, chemical weapons, energetic materials, etc. [31–33]. In these processes, wastes are introduced in molten carbonate salts under oxidation condition using air injection. The salt, at temperatures in the range 900–1000 °C, catalyzes waste oxidation. Carbon dioxide and acid gases such as HCl and HF are trapped in the bed. The inorganic compounds contained in the wastes lead to final residues, which are retained in the molten salt bed. MSO has several advantages over incineration:

- Low cost environmentally acceptable process (no formation of dioxin or furan).
- Flameless oxidation process.
- Owing to high solubility of waste in molten carbonates, reduced volumes are necessary, with no effluents.
- Oxidation of most organic waste is exothermic; therefore the heat of oxidation can be used to keep the salt molten.
- Scale-up is adapted to small and medium size of installation while classical incineration is not economically feasible below a capacity of 100 000 tons/year.

The process can also be operated under reducing conditions, so that carbon monoxide and hydrogen gases are produced and can be used as fuel gas or as a synthesis gas.

The molten salt selected in the present work was a mixture of hydroxides, specifically NaOH–KOH, with eutectic composition ($T_{\text{melting}} = 170\text{ °C}$). The main attractive features of molten hydroxides are:

- The low melting temperature allowing working at about 250 °C, which reduces the energy cost and limits the corrosion of the installation.

- A wide electrochemical window.
- The high solubility of gases such as halogens and CO₂, which limits toxic emissions.
- A high solubility of oxides, glasses and plastics.
- The behaviour of metals in the salt mixture is described in the literature [14] and it is shown that metals like, copper, gold, nickel and silver are not oxidized in the absence of oxygen.
- Valuable metals can be recovered in the solid state.

The objective of this paper is to describe the WPCBs recycling method using molten KOH–NaOH eutectic, to evaluate the efficiency of this method, and to discuss its impact on the environment.

2. Materials and methods

2.1. PCBs characterization

PCB waste used in this study was obtained from IT equipment.¹ The samples were broken into pieces (20 mm × 10 mm) before experiments.

2.1.1. General structure of PCBs

PCBs are insulating substrates on which the connections between electronic components are made by copper tracks. There are several kinds of electronic waste: single and double-sided plated-through-hole (PTH) or multilayered printed circuits. Before experiments, the structure of PCB was analyzed by SEM, coupled with energy dispersive spectrometry (EDS).

2.1.2. Metal contents

The valuable metal contents are obtained by using inductively coupled plasma/atomic emission spectroscopy (ICP AES Perkin Elmer – Optima 3000XL). Before analysis, an acid leaching of the sample with aqua regia (3:1(V:V), 35% HCl:60% HNO₃) was carried out during 24 h. Then, part of the digestion solution was diluted with ultrapure water (Milli Q). For each analyzed metal (Fe, Ni, Au, Cu, and Ag), the mean concentration was averaged from five measurements.

2.2. Treatment of PCBs in molten KOH–NaOH eutectic

2.2.1. Experimental reagents

Treatments were carried out with pure sodium hydroxide and potassium hydroxide (NaOH purity >98% and KOH purity >85%). The experimental salt had the eutectic composition, i.e., 41 wt.% NaOH–59 wt.% KOH. 30 g of hydroxide mixture was used for each run.

2.2.2. Experimental setup

The experimental setup, as shown in Fig. 1, consists of a stainless steel sealed cell, flown through by pure argon, and allowing the recovery of the output gases. Crushed PCBs samples were placed in an iron melting pot containing the salt. The waste was mixed with solid granules of hydroxides so that $w_{\text{PCBs}}/w_{\text{salt}} = 0.3$ (for a total of ca. 40 g). The cell was heated in a tubular electric furnace. After melting of the salt, the temperature was kept at 300 °C during about 1 h. Pure Argon, with a flow rate of 2 L h⁻¹, was used as flowing gas.

2.3. Analyses of reaction products

After cooling, the solid mixture was lixiviated in ultrapure water and the solid residues were separated from the solution by

¹ *PCB waste provided by Company VMA, Pontcharra, France.

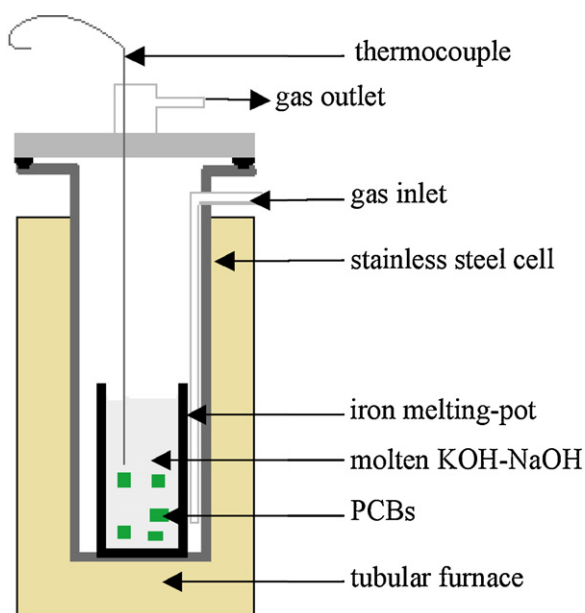


Fig. 1. Experimental set-up.

filtration. After filtration, two types of products were extracted from the molten salts:

- a metal fraction, i.e., plates, pins, foils, etc.,
- a brown powder.

2.3.1. Characterization of the metallic fraction

The metallic fraction recovered after the treatment was observed by SEM, and analyzed by ICP AES, after a leaching operation with aqua regia.

2.3.2. Characterization of the brown powder

The brown powder was analyzed by SEM and powder X-ray diffraction (PXRD), using a PANalytical X'pert Pro MRD diffractometer with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$), in the Bragg–Brentano geometry from 8 to 102° in 2θ (0.017° in 2θ step, 45.1 s as a counting time).

2.4. Characterization of gaseous emissions

To check the efficiency of the process with respect to environmental protection, two analyses of gaseous emissions were performed.

2.4.1. Analyses of halides

During treatment, the outlet gas was bubbled through ultrapure water (Milli Q), so that the ionizable halogens were dissolved in water to form halides, which were analyzed by ion chromatography.

The analyses of halides in solution were carried out by using a Metrohm ion chromatographic instrument (881 Compact IC Pro, Metrohm) attached to an autosampler (863 Compact). Detection was performed with a Conductivity Detector (881 Compact IC Pro 1) from Metrohm. Separation was carried out using a Metrosep A Supp 15-250/4.0 column. All the measurements were performed at 45 °C (column temperature) under the following elution conditions: 3.5 mM sodium carbonate/3.0 mM sodium hydrogen carbonate at 0.80 mL min⁻¹ as mobile phase.

Table 1
Metal contents and intrinsic values in PCBs.

Metal	Composition (g/kg)	Metal value ^a (€/kg)	Intrinsic value (€/kg)
Ni	2.17	16.38	0.04
Cu	255	6.50	1.66
Au	0.632	33020.52	20.87
Ag	0.306	672.04	0.21
Fe	2.61	0.11	0.0003

^a Metal values are based on November 2010 Market Values.

2.4.2. Analysis of carbon monoxide and dioxide (CO and CO₂) and volatile organic compounds (VOCs)

The outlet gases were collected in a Tedlar bag and were analyzed by micro gas chromatography coupled with mass spectrometry (gas chromatograph M3000/mass spectrometer quadruple HP MSD5973).

3. Results and discussion

3.1. PCBs characterization

3.1.1. General structure of PCBs

As shown in Fig. 2 (SEM observation using BSE mode) the PCBs were made of a fibreglass core (referred to as (1) in the figure), onto which a copper sheet is laminated (referred to as (2), the white phase) and the whole is covered with an epoxy resin (3). The fibreglass matrix, according to the orientation of its fibres, forms a kind of woven plate, made of aluminium, silicon and calcium (see EDS spectrum in Fig. 2). Almost 75 wt.% of the waste consists of non-metallic components, such as plastics, resins or fibreglass. The EDS analysis indicated that the metallic layer was mainly made of copper.

3.1.2. Metallic content of studied PCBs

Table 1 gives the composition in metals obtained by ICP AES analyses after dissolving the waste in aqua regia. Copper is the predominant metal, i.e., ca. 25 wt.% compared with nickel, ca. 2 wt.%. As shown in Table 1, the metal recovery, including precious metals, is economically interesting.

3.2. Treatment of PCBs in molten KOH–NaOH eutectic

As illustrated in Fig. 3, after treatment in molten KOH–NaOH eutectic, followed by cooling, the solidified mixture was lixiviated in ultrapure water and both a metallic fraction and a brown powder were recovered.

3.2.1. The metallic fraction

The metallic fraction appears like a tangle of wires and foils (Fig. 4). Fibreglass, resins, and plastics are totally eliminated even within the core of the boards. After dissolution in aqua regia, the analysis of the solid metallic phase was carried out by ICP, as shown in Table 2. Comparison with the mean intrinsic composition of PCBs, indicate that copper nickel and all the precious metals, i.e., gold, and silver, are recovered. These results are in agreement with various studies of the solubility of metals as a function of pO_2 [34,35].

Table 2
Metal composition of the metallic fraction.

Metal	Metallic fraction in g/kg in the waste after treatment
Ni	0.950
Cu	287
Au	0.725
Ag	0.238

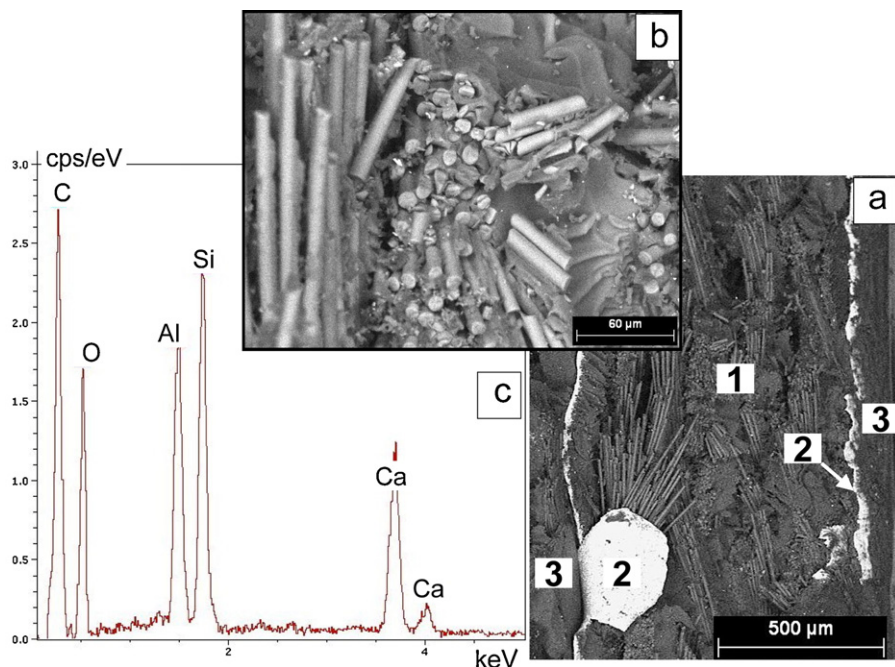


Fig. 2. SEM analysis of the studied PCBs. (a) SEM micrograph of PCB cross-section in BSE mode (1) fibreglass core, (2) copper sheet, (3) epoxy resin. (b) Magnified micrograph of epoxy resin. (c) EDS spectrum of the fibreglass matrix.



Fig. 3. Photographs of reaction products after lixiviation of the solidified salt, i.e., metallic fraction (left) and brown powder (right), respectively.

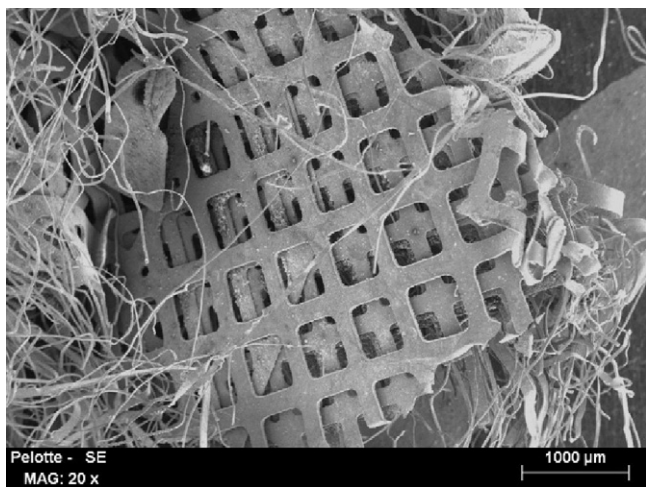


Fig. 4. SEM micrograph of the metallic fraction.

3.2.2. The brown powder

EDS analysis (Fig. 5) showed that the powder is a mixture of calcium carbonate and calcium silicate, which result from the dissolution of fibreglass, epoxy resin and plastics. The main components of fibreglass, i.e., silica (ca. 70%), CaO (10%) and Na₂O (14%) are dissolved in the highly basic hydroxide melt. The organics are converted to CO₂ and H₂O. These results confirm that carbon dioxide formed during the molten salt oxidation of resin and plastics is trapped in the melt by forming molten carbonates and silicates.

3.3. Characterization of gaseous emissions

Pollutant concentration in the off-gas emitted from the reaction chamber were analyzed, i.e., carbon monoxide, carbon dioxide, halogens, and volatile organic compounds (VOCs). The comparison between the halide concentrations in the molten salt after PCB treatment and in the downstream solution after bubbling the flue gas indicates that most of the halogens react with the hydroxides and are converted to sodium and potassium halides. As shown in Table 3, 99.8% of fluorides, 99.3% of chlorides and 99.99% of

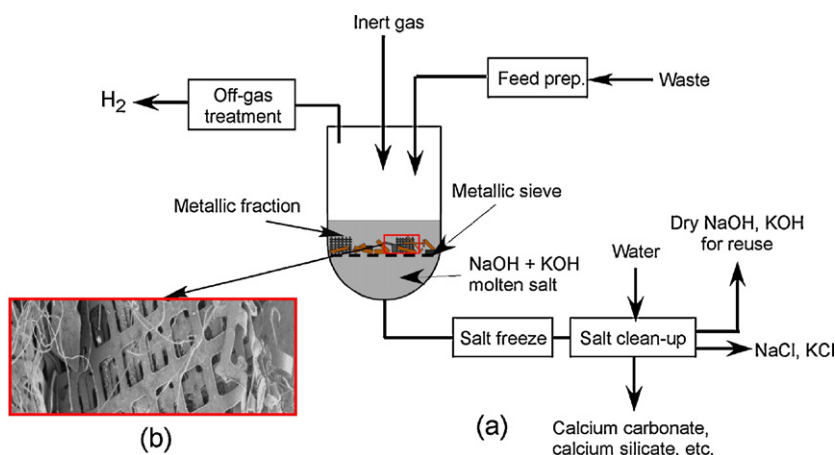


Fig. 6. Schematic recovery process. (a) Process functional flow diagram. (b) Recovered metallic fraction.

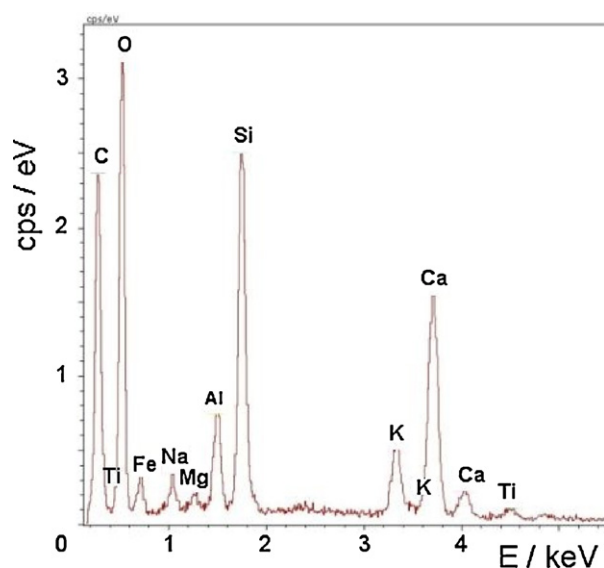


Fig. 5. EDS spectrum of the brown powder.

bromides were captured in the bath. μ GC–MS analyses of the escape gases (see Table 4) showed that volatile organic compounds, only in the ppm range, were emitted. On the other hand, the flue gas contained a very small mole fraction of CO and CO₂ (less than 20 ppm) and a high content of hydrogen (in the order of 30%) which can be used as fuel or chemical feedstock.

3.4. Towards an industrial process

Molten salt oxidation processes have been proposed for the treatment of various types of wastes [36–40]. Fig. 6 shows a schematized process system for metals recovering from WPCBs using molten NaOH–KOH eutectic mixture, which includes the waste preparation equipment; the molten salt reactor, the off-gas treatment system and a salt recycle system. As a comparison

Table 3

Comparison of the quantity of halogens emitted in gaseous emissions and trapped in molten salts, in mg/kg of treated waste.

Anions	Bubbling solution (mg/kg)	Salts solution (mg/kg)
Fluoride	0.262	166
Chloride	22.6	3089
Bromide	0.822	9159

with the MSO process using carbonate mixtures, one of the main advantages of the proposed recovery treatment concerns the low operating temperature, i.e. 300 °C compared with 850 °C. Consequently, the structural materials will be less sensitive to corrosion and thermal management processes will be much simpler. A shredder has to be used for size reducing of the PCBs before feeding to the molten salt reactor. A metallic sieve with appropriate mesh, set in the molten salt reactor will retain the metallic fraction of the PCBs that can be easily retrieved. It has been shown in the present work, that if neutral atmosphere is maintained in the reaction vessel, the produced gas is mainly hydrogen. The off-gas treatment requires trapping the traces of acid gases (however, most of chlorides compounds are trapped within the molten salt), to condense water vapour and to capture the particles in the off-gas. Spent salt can be recycled: after dissolution in water, the solid residues are removed by filtration (as shown in the present work, the solid residues are mainly calcium carbonates and silicates). After removing of sodium and potassium chlorides from the solution, and drying, the solid NaOH–KOH mixture can be reused. The sodium and potassium

Table 4

Analyses of gaseous emissions by μ GC–MS.

Compounds	Averaged concentration (%)
Hydrogen	27.8
Oxygen	0.3
Nitrogen	2.0
Methane	1.8
CO ₂	13 ppm
CO	<20 ppm

Compounds	Averaged concentration (mg/(n)m ³)
THF	1123
IPA	706
Benzene	564
1-Butanol	427
Allene	419
Propyne	413
Propene	377
Acetone	186
2-Butene	174
2-Butanone	128
Toluene	123
Hexamethyltrisiloxane	116
1,3-Cyclopentadiene	108
1,3-Dioxolane-2-methyl	103
Disiloxanehexamethyl	95
2-Butanol	69
Chlorobenzene	15
Xylenes	14
Styrene	13

hydroxides can be concentrated using a multi-effect evaporator: the salt precipitates during the concentration of the caustic soda. Anhydrous caustic product can be obtained in a rising film evaporator, and under a partial vacuum.

4. Conclusion

A new process having no negative impact on the environment was investigated to recover valuable metals from waste printed circuit boards (WPCBs). Non-metallic components, i.e., fibreglass, epoxy resin; plastics were dissolved in the molten KOH–NaOH eutectic at 300 °C. With this method, the metallic fraction, which contained copper, and all the precious metals present in the waste, were recovered, with no dissolution or melting phenomena. Moreover, molten hydroxides, due to their high basicity, dissolved and trapped many gases such as halogens or carbon monoxide and dioxide. Finally, the flue gas contained almost 30% of hydrogen, a gas which can be collected as fuel gas or as a chemical feedstock. It should be pointed out that, to reduce the consumption of fresh salt, a recycling system can be included. In a pilot plant, the metallic fraction can be easily separated from the molten salt, using an appropriate sieve, and the molten NaOH–KOH mixture be transferred to the reaction vessel.

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