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## CHEMICAL RECYCLING OF PRINTED CIRCUIT BOARD WASTE BY DEPOLYMERIZATION IN SUB- AND SUPERCRITICAL SOLVENTS

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**Abstract:** Disposal of waste printed circuit boards is regarded as a potential major environmental problem due to their heavy metal content. Therefore, recycling waste printed circuit boards represents an opportunity to recover the high value resin chemicals and the high value metals that are present. In this study, the solvo-thermal depolymerisation of waste printed circuit boards obtained from desktop computer monitors was carried out using water, ethanol and acetone between 300 – 400 °C. Alkalis (NaOH, KOH) were used as additives to promote the removal of the resin fraction of the printed circuit boards. At 400 °C, 94% resin removal was achieved when water was used as the solvent, in the presence of NaOH. The liquid produced in the process was analysed by GC/MS and the results showed that it was mainly composed of phenol, and some phenolic compounds, with up to 62.5 wt% present as phenol in the liquid phase.

**Keywords:** Recycling; Supercritical, Depolymerisation, Printed circuit board

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## 1 Introduction

Electrical and electronic equipment are widely used in our daily lives, with the technological developments and intense marketing of new products; from cell phones to laptops, TVs to kitchen appliances. This increasing production and utilization result in waste generation, due, in part, to the fast replacement of these electronic devices. Currently waste electrical and electronic equipment (WEEE or E-waste) is one of the fastest growing wastes especially in the European Union, therefore stringent regulations were introduced by the EU under the WEEE Directive in 2002 (Directive 2002/96/EC) for its management. In 2008, this Directive was revised as the projections for 2020 showed that more than 12 million tonnes of WEEE generation in the EU, so the new WEEE Directive 2012/19/EU was put into action to better regulate WEEE generation and disposal [1]. The aim of the regulations is to encourage the reuse and recycling of WEEE, to recover valuable metals and organic polymeric resin materials produced from non-renewable resources. The estimation for worldwide WEEE generation is estimated to be around 40-50 million tonnes, representing a considerable potential resource for metals and polymeric resin materials [2].

Printed circuit boards are the main waste stream in the WEEE, as they are present in every electronic device. However, because of their heterogeneity, it is a complicated process to treat printed circuit board waste. Mainly, they consist of organic resins (polymer fraction), metals and glass fibre. The resin is predominately a thermosetting polymer such as phenolic and creosol based epoxy resin, bisphenol A epoxy resin, or cyanate esters and polyamides. Generally the metal content is ~40 wt.% and copper, iron, nickel, gold and palladium are the valuable metals which commonly exist in printed circuit boards. [3, 4]. The typical metal content of printed circuit boards is given in Table 1 [4]. Copper is by far the most abundant component metal in printed circuit boards. Currently around 15 % of waste printed circuit boards are recycled with the conventional methods for management of these wastes are landfilling and incineration. However, because of the formation of hazardous compounds such as polybrominated dibenzodioxins and dibenzofurans (PCDD/Fs) and toxic brominated compounds from incineration; and the leaching of toxic compounds and heavy metals to groundwater due to landfilling, recycling of waste printed circuit boards are crucial to prevent such hazards to the environment [4].

The present technologies for recycling of printed circuit boards are mechanical processing, combustion and hydrometallurgical methods. Before any of such treatments of the waste, a pre-treatment step is necessary to dismantle the large components such as resistors, capacitors, etc., which can be hazardous or reusable materials [5]. Mechanical recycling of printed circuit boards is a physical separation process of the waste by grinding into fine particles. The final separation can be made via magnetic, electrostatic or density-based separation to produce a metal-rich fraction. The non-metallic fraction, which is the resin fraction of the board can find application areas as a filler for thermosetting resin composites [6], a reinforcing filler for thermoplastic resin composite materials [7], as a raw material for concrete [8-10], or as a modifier for viscoelastic materials [11]. Although mechanical separation processes appear an environmentally friendly, convenient and relatively simple process, the application area is highly influenced and limited by the final properties of the recycled polymer; as the clean separation of metals from the polymer should be achieved and the transfer of toxic and hazardous substances to the final product should be prevented. Also, to achieve high levels of recovery, very small particle sizes are ideally used, which increases the process cost.

Combustion of printed circuit boards oxidises the polymer fraction of the waste and the remaining metals are subjected to further separation processes to recover the metals. The metals can be recovered by electro-refining. The main drawback of combustion is the potential production of toxic and hazardous materials such as PCDD/Fs and polybrominated biphenyl ethers (PBDEs) [12]. Hydrometallurgical treatment of printed circuit boards involves dissolution of the waste with typically an acid and to recover the metals from the solution via electro-refining or crystallization. For example, the leaching of nickel and copper is carried out selectively and gold can be recovered in high purity. However, the solutions are highly corrosive and hazardous, as concentrated nitric acid, hydrochloric acid and/or cyanide solutions are used for leaching over long dissolution times [13-15]. Destruction of the resin by dissolution in acid/alkali also represents a waste of non-renewable organic resources.

Currently studies on thermochemical methods to recycle waste printed circuit boards are being investigated to convert the resin fraction into monomers and to recover the metals. Pyrolysis processing has been investigated where the polymer can be thermally degraded to produce oils, and the metals collected in the char after the reaction [3]. However, the metals are often recovered together with the char, so that further processing via separation processes

becomes necessary. In addition for resins containing fire retardant bromine compounds, the liquid phase contains bromine, which thereby contaminates the product oil. Gasification has been investigated as a thermochemical treatment method which converts the organic resin into gas products, mostly CO and H<sub>2</sub>. However, high temperatures are used to gasify all of the polymer fraction [16].

Utilizing sub- and supercritical fluids to depolymerize the resin fraction of the printed circuit board waste represents a potentially cleaner and cheaper technology for recycling the waste, as it enables the recovery of metals in addition to converting the resin into valuable chemicals and/or fuels. The cleaned mixture of metals obtained would also become easier to process or separate. There are various studies with different solvents which have been investigated for the treatment via depolymerisation of plastic wastes reported in the literature. The most common solvents are water and alcohols, and the process has been mainly applied to pure polymers such as polyethylene terephthalate rather than printed circuit boards

In this study, water, ethanol and acetone were used as solvent to depolymerise printed circuit board waste produced from desktop computer monitors. Alkalis (NaOH, KOH) and acetic acid were investigated as promoters to remove the resin fraction from the waste to recycle the polymer fraction of the printed circuit boards and to produce a chemical feedstock in addition to the recovery of the metals.

## **2. Materials and Methods**

Printed circuit board waste from desktop computer monitors was used for the investigation. The capacitors, conductors, resistors and all removable materials were dismantled, and the boards were crushed into smaller particle size as shown in Figure 1. A further grinding process was applied to decrease the particle size below 2 mm to produce a more homogenous mixture. The sample with particle sizes <2 mm was characterized and used for the depolymerisation tests. Analysis was carried out to determine the volatile (resin) fraction. Four samples of 1 g waste were oxidized at 500 °C for 2 hours, and the results are shown in Table 2. These tests suggested that approximately 38 wt.% of the dismantled waste was composed of metals and the remaining part was the polymer.

Sodium hydroxide (pellets), potassium hydroxide (pellets), acetic acid and diethyl ether for the extraction of organic compounds in the liquid effluent and ethanol and acetone were obtained from Sigma-Aldrich, UK.

The sub-critical and supercritical solvent depolymerisation experiments were carried out in a 500 mL stainless steel reactor obtained from Parr Instruments Co. USA. For each experiment, 2.5 g of printed circuit board waste and 60 ml of solvent was fed to the reactor [17]. Alkalis and acetic acid were only used in the presence of water and when used, 1.0 g of NaOH or KOH was dissolved in the liquid. The reactor was then sealed, purge with nitrogen for 5 min and heated up to the designated temperature and corresponding pressure. The heating rate was set at 30 °C min<sup>-1</sup> kept constant throughout the experiments and the reactor was quickly withdrawn from the heater as soon as the designated conditions were reached. The reactor was then cooled to room temperature and the gaseous effluent sampled for analysis.

The gas samples were quickly analysed offline by means of two gas chromatographs for permanent gases and hydrocarbons, respectively [18]. Permanent gases, H<sub>2</sub>, CO, O<sub>2</sub>, N<sub>2</sub> and CO<sub>2</sub>, were analysed with a Varian 3380 GC with dual packed columns and dual thermal conductivity detectors (GC/TCD). Analysis for H<sub>2</sub>, CO, O<sub>2</sub> and N<sub>2</sub> was carried out with a 2m length by 2 mm diameter column packed with 60 – 80 mesh molecular sieve. The CO<sub>2</sub> analysis used a 2 m length by 2 mm diameter column with HayeSep 80 – 100 mesh packing material. A second Varian 3380 GC with a flame ionization detector (GC/FID) was used to analyse and determine the hydrocarbons (C<sub>1</sub> – C<sub>4</sub>). The column was 2 m length by 2mm diameter, packed with HayeSep 80 – 100 mesh.

The reactor contents, including liquid and solid residues were emptied into a holding beaker and separated by filtration washing several times with distilled water. The solid residue was dried to a constant weight in an oven at 105 °C to determine its weight. A portion of the liquid effluent was extracted with diethyl ether based on the method applied in our previous work and analysed on a Varian 380 GC fitted with a Saturn 2200 ion trap MS/MS according to the analytical procedure previously reported by Blanco et al. [19, 20]. The remaining aqueous phase was also analysed for the bromine content by ion chromatography.

The amount of resin removed from the printed circuit board waste was determined as;

$$R = \frac{F-X}{F_R} * 100 \quad \text{Eq. (1)}$$

Where R is the percentage of resin removal; F is the amount of the printed circuit board added to the reactor.  $F_R$  defines the amount of the resin in the original printed circuit board waste, which was found to be 62 wt% according to ash analyses. X is the amount of the solid residue, after the hydrothermal depolymerisation.

### 3. Results and Discussion

In this study, ethanol, acetone and water were used as solvents at their near critical conditions. When water was used, NaOH, KOH and acetic acid was investigated as promoters to increase the resin removal efficiency.

#### 3.1 The effect of solvent on resin removal

Ethanol was used to depolymerize the waste printed circuit boards sample at a temperature range from 200 to 400°C; the results are shown in Table 3. Below its critical point (241°C, 6.14 MPa) of ethanol, the resin removal was very low after three hours reaction time. As the temperature increased to 300 °C, the resin removal was improved, however, the highest achieved was only 56 %. The further increase in the temperature to 400 °C did not affect the resin removal. However, the gas yield increased to give an almost 10 fold increase in gas yield than that found at 300 °C (see Table 4), since the ethanol itself decomposed to produce more H<sub>2</sub>, CO and CH<sub>4</sub> at 400 °C, as shown in Figure 2. The effect of reaction time was also tested in the case of ethanol as the solvent; at 250 °C there was no significant change on the resin removal when the reaction time was increased from 3 to 6 hours. Although a relatively low depolymerisation was found here, ethanol as a solvent has proven to be effective in depolymerisation of some thermoplastics, for example, polyethylene terephthalate and the depolymerisation of thermosetting plastics [21, 22].

When acetone was introduced as the solvent, lower levels of depolymerisation were obtained compared to ethanol at the same reaction conditions. At 300 °C, after 3 hours reaction time, the resin removal was only 36.7 % (Table 5). When water was used as the depolymerisation solvent, at zero residence time at the 360 °C designated temperature, almost 75 % of the resin was removed, whereas ethanol was able to reach a resin removal of 59 % at

the same temperature (Table 5). At the critical point, water experiences unique changes in its properties such as decrease in dielectric constant, density, ion product, and it becomes an efficient solvent for organic materials [23]. Therefore, even at short residence times, high resin removals up to 85 % were achieved as the temperature was increased as shown in Table 6. To increase the depolymerisation efficiency, some additives to water were tested to determine their effect on the resin removal.

Alkalis (NaOH, KOH) and acetic acid were added to water which was added to the reactor followed by depolymerisation at 400 °C, the results are shown in Table 7. The table shows that acetic acid had no significant effect on the depolymerisation of the resin, showing similar depolymerisation levels to water alone. However, the addition of NaOH and KOH resulted in a significant increase in the depolymerisation of the resin, increasing from ~81% to ~94% in the presence of the alkalis (Table 7).

### 3.2. Product Distribution

Amongst all the solvents used in this study, only water was able to reach high resin removal efficiencies, especially in the presence of alkalis. Mainly, the resin was converted into liquid products, as around 85 wt% of the organic material was detected in the liquid effluent when KOH was used as the additive. The gas composition and the total grams of gas produced per gram of waste is shown in Table 8. The gas chromatographic analysis of the gases in the presence of water showed that the major composition of the gas products consisted of H<sub>2</sub>, CO and CO<sub>2</sub>. The addition of the alkalis resulted in a marked increase in hydrogen concentration, this is suggested to be due to the enhancement of the water-gas shift reaction, between H<sub>2</sub>O and CO resulting in H<sub>2</sub> and CO<sub>2</sub>, the CO<sub>2</sub> in turn is captured as the sodium or potassium carbonate.

The composition of the organic content of the liquid obtained from depolymerisation was also highly influenced by the introduction of KOH and NaOH into the reaction. When they were used as the additives, the major organic compounds detected by the GC/MS analytical system were phenol and phenolic compounds as shown in Figure 3. Phenol had the largest concentration in the liquid effluent, at 62 wt%, which was a six fold increase compared to that of with water alone (Table 9). When NaOH was introduced with the water, 80 % of the resin was converted into liquid and which improved to a value of 86 % in the presence of KOH. Quantification of the organic components in the liquid product in Table 9

shows that methyl-phenols (o-cresol, p-cresol, 2,4,6-trimethylphenol) and ethyl-phenols were the second major products identified, as a result of the degradation of the phenolic thermosetting resin.

While no bromine was detected in the gas phase, a trace amount of bromine in the form of bromophenol was detected in the organic liquid phase, at a level of ~0.03 ppm. However, around 60 mg bromine per gram waste was detected in the aqueous phase collected from the reactor, according to the ion chromatography analysis. This confirms that the bromine compounds were dissolved in the water after the hydrothermal treatment, which results in producing a clean, almost bromine free oil. These results confirm earlier work [18] in relation to the hydrothermal treatment of wastes containing brominated flame retardants where, acrylonitrile-butadiene-styrene containing bromine flame retardant (Br-ABS) and high impact polystyrene containing bromine flame retardant (Br-HIPS); where the bromine content of the brominated plastics ended up mostly in the aqueous phase due to dissolution in the water medium [18].

To residues recovered after the hydrothermal treatment, were further processed by mild oxidation. For example, Figure 4, shows the solid residues after drying, before and after oxidation; Figure 4(a) shows the residue from depolymerisation with ethanol at 400 °C; Figure 4(b) shows the residue from depolymerisation with water at 400 °C (c); Figure 4(c) shows the residue from depolymerisation with water and NaOH at 400 °C. When the resin removal efficiency was low as in the case of depolymerisation with ethanol, there was a high level of residual resin present in the solid residue and oxidation resulted in significant weight loss from the solid residue before and after oxidation (Figure 4(c)). However, where water with alkali was used as the solvent, there was only low levels of residual resin on the solid residue (Figure 4(c)). The clean residue could be further processed for recovery of valuable metals, such as copper, silver, gold, palladium, etc.

#### **4. Conclusions**

The hydrothermal depolymerisation of printed circuit board waste obtained from desktop computer LCD monitors was investigated, to remove the resin fraction from the waste in order to recover metals, and also to recycle the resin as a chemical feedstock. At 400 °C, 81% of resin removal was achieved when water alone was used as the solvent, and this was further improved in the presence of NaOH and KOH, which led to 94% resin removal. However, acetone and ethanol were not able to depolymerize the waste completely; only up

to 56 % resin removal was achieved at 300 °C after 3 h reaction in ethanol. Further increase in the temperature caused ethanol to decompose to produce H<sub>2</sub> and CH<sub>4</sub> rich gas, while the higher temperature had no effect on the resin removal. The liquid produced in the aqueous processes was mainly composed of phenol, and phenolic compounds, which were the precursors of the original thermosetting resin. Most of the bromine content, which was derived from the flame retardant additives in the printed circuit boards, was found in the aqueous phase, which results in oil recovery with near-zero bromine content. Addition of alkalis to the water solvent increased the phenol yield up to 62.5%, and the residues were recovered in a clean state, ready for metal separation.

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Figure 3 GC/MS analysis of the liquid from the depolymerisation of printed circuit boards with water and NaOH at 400 °C

Figure 4 Solid residues after drying, before and after oxidation; samples from depolymerisation (a) with ethanol at 400 °C (b) with water at 400 °C (c) with water and NaOH at 400 °C.



Figure 1.

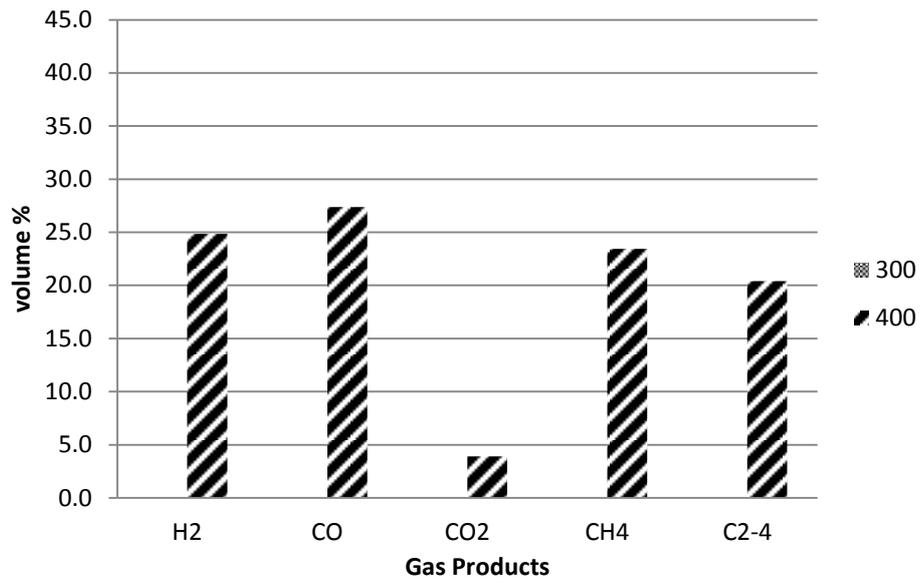


Figure 2.

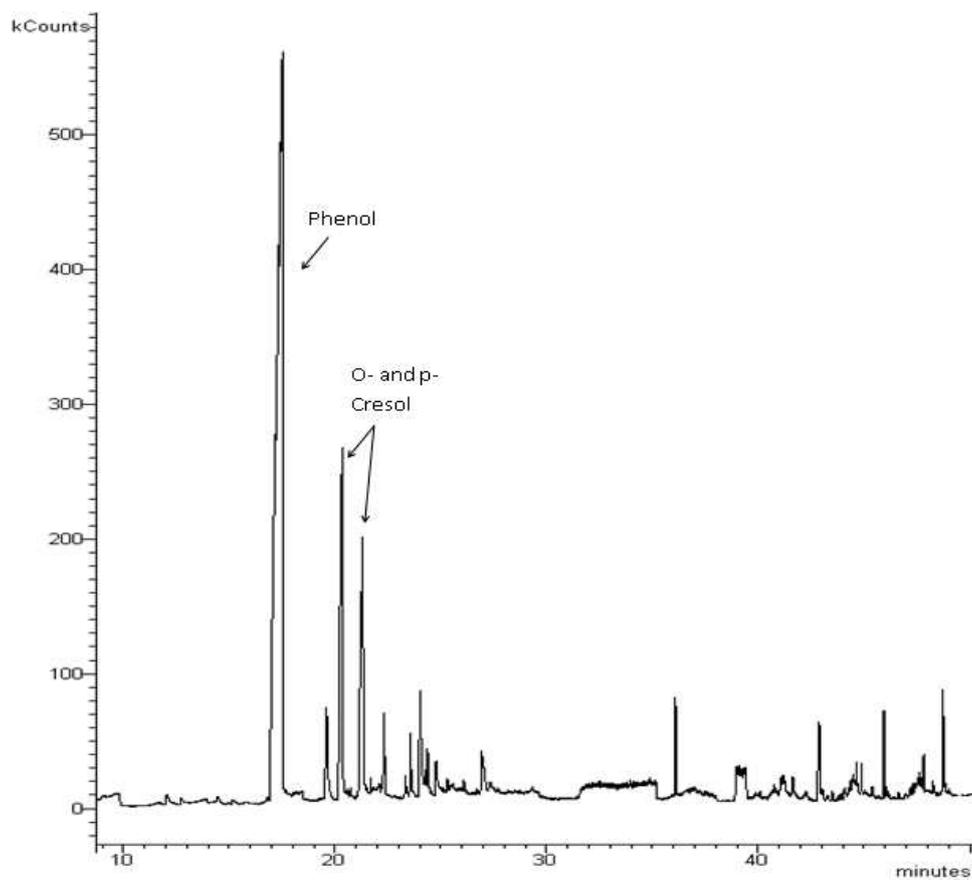


Figure 3.

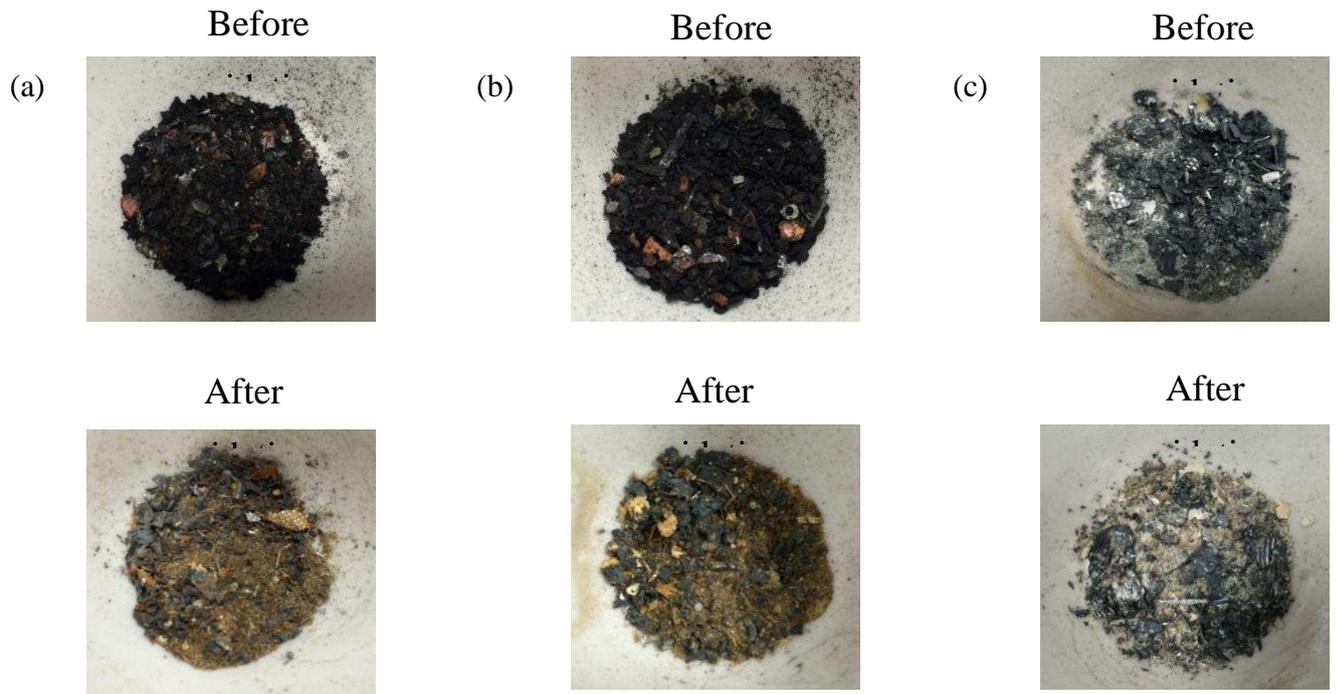


Figure 4.

Table 1. Typical metal content of printed circuit boards [4].

Material	Composition (wt%)
Metals (Typically 40 wt%)	
Cu	10 – 26.8
Al	1.33 – 4.78
Pb	0.99 – 4.19
Zn	0.16 – 2.17
Ni	0.28 – 2.35
Fe	1.22 – 8.0
Sn	1.0 – 5.28
Sb	0.06 – 0.4
Au	80 – 1000 (ppm)
Pt	4.6 – 30 (ppm)
Ag	110 – 3301 (ppm)
Pd	10 – 294 (ppm)

Table 2. Analysis of printed circuit board residual ash.

Sample	Weight loss (%)	Standard Deviation (%)
1	64.1	1.4
2	58.6	2.5
3	65.8	2.6
4	59.9	1.6
Mean	62.1	2.4

Table 3. The influence of temperature on the depolymerisation of printed circuit boards with ethanol

Temperature (°C)	Time (min)	Resin Removal (%)
200	180	17.3
250	180	45.2
250	360	43.3
300	180	55.9
400	180	50.3

**Table 4. Gas yield from the depolymerisation of printed circuit boards with ethanol**

Solvent	Temperature (°C)	Time (min)	Gas Produced (g/g waste)
Ethanol	300	180	0.24
Ethanol	400	180	2.82

Table 5. The influence of ethanol and acetone solvent on the depolymerisation of printed circuit boards

Solvent	Temperature (°C)	Time (min)	Resin Removal (%)
Ethanol	300	180	55.9
Acetone	300	180	36.7
Ethanol	360	0	59.1
Water	360	0	74.6

Table 6. Influence of temperature on the depolymerisation of printed circuit boards with water

Temperature (°C)	Resin Removal (%)
360	74.6
380	76.3
400	81.0
420	85.4

Table 7. Influence of the presence of additives on the depolymerisation of printed circuit boards with water

Solvent	Temperature (°C)	Resin Removal (%)
Water	400	81.0
Acetic acid/Water	400	81.9
KOH/Water	400	93.6
NaOH/Water	400	94.1

Table 8. Gas composition from the depolymerisation of printed circuit boards with water in the presence of additives

Gas Component	Yield (vol.%)		
	NaOH	KOH	Water
Hydrogen	35.2	28.3	7.1
Carbon monoxide	3.3	1.1	13.9
Carbon dioxide	58.4	68.4	77.1
Methane	2.2	1.7	1.4
C <sub>2</sub> – C <sub>4</sub> Hydrocarbons	1.4	0.5	0.5
Total produced gas (g/g waste)	0.30	0.34	0.28

Table 9. Organic composition of the liquid produced from depolymerisation of printed circuit boards at 400 °C with water in the presence of NaOH, in the presence of KOH and without any additives (water).

Compound	Yield (mg organic/g waste)			Compositions (wt.% organic)		
	NaOH	KOH	Water	NaOH	KOH	Water
Phenol	387.2	386.0	68.7	62.4	62.2	11.1
o-Cresol (2-methylphenol)	50.9	69.3	22.1	8.2	11.2	3.6
p-Cresol (4-methylphenol)	40.7	48.8	9.0	6.6	7.9	1.5
4-ethylphenol	6.9	14.0	5.9	1.1	2.3	1.0
2-ethylphenol	3.7	5.7	1.5	0.6	0.9	0.2
4-isopropylphenol	2.1	4.6	7.4	0.3	0.7	1.2
2,4,6-trimethylphenol	0.3	1.4	0.4	0.1	0.2	0.1
Dimethylanisole	0.9	-	0.5	0.1	-	0.1
2-methylbenzofuran	0.2	0.3	0.1	0.1	0.04	0.05
Total	493.6	530.2	116.1	79.5	85.4	18.7