Recovery of metals from printed circuit boards by Goldrec 1 process

N.M. Ippolito[1], M. Passadoro, G. Pellei, F. Ferella, F. Vegliò

Department of Industrial and Computer Engineering and Economics, University of L'Aquila, L'Aquila, 67100, Italy

Abstract

The paper describes a small full-scale plant based on the Goldrec 1 process, designed and patented by the University of L'Aquila. The Goldrec 1 hydrometallurgical process can treat printed circuit boards (PCBs) and other gold-containing components of different grades. The first step is a mechanical treatment to reduce the size of the scraps below 2 mm. The extraction of base metals occurs in a first reactor, where elements like Cu, Sn, Al, Fe, and Zn are extracted by a sulfuric acid/hydrogen peroxide solution. After filtration, the solid is leached again with thiourea and ferric sulfate in a sulfuric acid solution to extract gold and silver. The insoluble materials, mainly the plastic fraction, are filtered off. This second solution is sent to an electrolytic cell where gold is recovered as metal powder. The resulting solution undergoes a second electrowinning, where silver is deposited on the cathode.

The first pregnant solution undergoes recovery of Cu and Sn. The simulation was developed using lab-scale trial results. The 350 tons PCB/year, running in a batch operating mode, produces around 43.8 kg/y of gold, 85.8 kg/y of silver, 42.4 tons/y of copper, and 7.2 tons/y of tin oxide. The results show that profitability is easily achieved when PCBs are directly collected without cost: the net present value is 10.7 MEuro, with an internal rate of return of 150 % and a discounted payback time of 2 years only.

Keywords: PCB, WEEE, recycling, gold, copper.

1. Introduction

The increasing generation of waste electronic and electric equipment (WEEE) reached a considerable amount in the last ten years, especially in economically developed countries. The growth of such wastes is directly proportional to the fast development of microelectronic technologies that quickly make personal computers (PCs), mobile phones, and other electronic devices obsolete [1]. The trend is unsustainable for two reasons: using natural resources to extract the necessary metals produces a considerable amount of harmful waste that must be disposed of properly. Metals on which these electronic technologies are based are not inexhaustible. In addition, ore deposits are located in a few countries like China, Russia, South Africa, and Australia: this is an urgent problem in terms of supply, also looking at the geopolitical tensions that have arisen in the last two years.

Hence, WEEE represents the best example of urban mining. It contains base and precious metals that can be extracted and reused; the additional advantage is that their concentration is usually much higher than in primary ores and requires less energy for processing [2]. Printed circuit boards (PCBs), connectors, and other from medical, military, and research devices contain a significant number of precious metals, in particular gold (Au), silver (Ag), palladium (Pd), besides copper (Cu), tin (Sn). Furthermore, PCBs are the sole WEEE whose recycling is feasible without any disposal fee due to the amount and value of the metals contained therein [3].

Nevertheless, PCBs contain around 70 %wt of plastic-ceramic materials, glass fibers, like epoxy resins, and brominated compounds used as flame retardants [2].

Spent PCBs are divided into categories depending on the particular WEEE they come from (mobile phones, PCs, military devices, TVs, washing machines, air conditioning units, copy machines, etc..). As a result, the precious metal content is quite different, making the profitability of PCB recycling almost variable [4,5].

The pre-treatment stages are crucial to separate the metallic fraction from the inert one in an efficient way and enhance the subsequent recovery [6,7].

The most used recycling techniques at a commercial scale are preferably based on pyrometallurgy, which removes the plastic board and concentrates metals in the char [8]. Nevertheless, several hydrometallurgical processes have also been studied: those using thiourea or other non-toxic mixtures like $HCl/H_2O_2/CH_3COOH$ [9-11] are the most common. Instead, thiocyanate-based systems are hazardous and require additional safety measures [9].

Many processes have been investigated worldwide to extract metals from PCBs, mainly on a lab scale and a few on a pilot scale [12-22]. Considering the circular approach of the whole recycling concept, even the recovery of

[1] Corresponding author: tel.: +39 0862 434238; fax: +39 0862 434203; e-mail: nicolomaria.ippolito@univaq.it

the non-metallic fraction of PCBs has been studied [1]. Tantalum capacitors are particular components better separated in advance for more efficient recovery of tantalum and the primary metals of the PCBs [23].
Economic analyses have already been carried out on recycling processes. D'Adamo et al. demonstrated that the net present value (NPV) varies from 6.8 M€ for medium-grade PCBs to 63.0 M€ for high-grade PCBs, whereas it is negative for low-grade ones [1]. Another research work, which assessed the Australian scenario, concluded that a plant with 10,000 tons/year capacity would be sufficient to treat the current annual collected PCB in Australia; a lower capacity would even result in higher economic feasibility [24]. Focusing on processes using thiourea for Au extraction, most of them have been applied to recover gold from primary ores; the final recovery has usually been carried out by electrodeposition [25-30].
The novelty of this paper can be placed on developing a new process and the relevant plant to recover the most valuable metals from spent PCBs, namely Cu, Sn, Au, and Ag. Apart from the technical simulation, a profitability analysis was also developed, using a modified version of the Goldrec 1 process patented by Birloaga et al. [31]. The original patented process indeed differs from this by the recovery methods used for Cu, Au and Ag. While metastannic acid is always recovered by precipitation with a polyamine flocculant, copper and the precious metals are precipitated together by cementation with powdered zinc. The process presented in this paper has been tested in the ambit of Treasure, a project funded by the European Commission under the Horizon 2020 program (grant agreement N.101003587).

2. Description of the process

The flow-sheet of the modified Goldrec 1 is shown in Figure 1. This process is characterized by a two-stage leaching section: in the first stage, base metals like copper (Cu) and tin (Sn) are extracted, whereas precious metals, i.e. gold (Au) and silver (Ag) are brought into solution in the second stage. This selective leaching is very helpful in separating metals already in the leaching section: this allows an efficient recovery of all of them, in terms of total mass and grade.

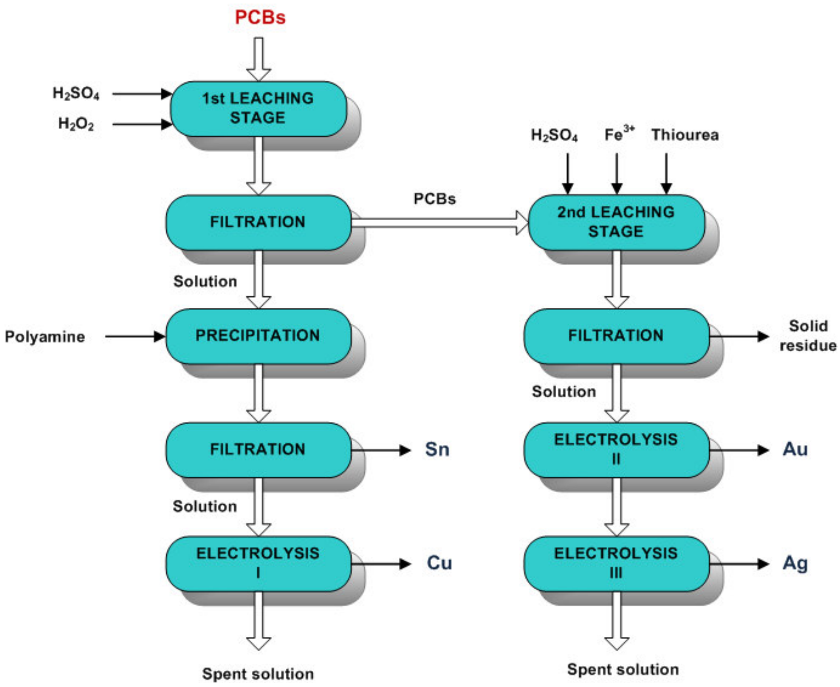

Fig. 1 Goldrec 1 recycling process flow-sheet

Tin is precipitated by flocculation with the aim of polyamine in the form of metastannic acid (H_2SnO_3), whereas copper is recovered by electrodeposition. The spent solution is partially recycled back to the leaching reactor, and the rest discharged after wastewater treatment to recover water. The second pregnant solution after the thiourea leaching undergoes sequential electrodeposition of gold and then silver, under different operating parameters. The spent solution is thus recycled in the second leaching stage, but a certain aliquot is sent to the wastewater treatment.

2.1 Operating conditions

2.1.1 Characterization of PCBs

PCBs are manually dismantled, and specific components like capacitors, heat sinks, transistors, and cables are removed. Hence, the PCBs are cut with shear in pieces, with a size of around 2 x 3 cm. Thus, the small pieces are milled by a knife mill equipped with a sieving mesh of 2 mm (Retsch SM2000). The ground material undergoes acid digestion according to the procedure detailed in Ippolito et al. [9], thus determining the concentration of the metals by inductively coupled plasma optical emission spectrometry (ICP-OES, Agilent Technologies 5100). Several mixed samples from PCs, mobile phones and other devices have been characterized throughout the experimental activity, and the average composition is listed in Table 1.

Element	**Concentration**
Au	150 g/t
Ag	300 g/t
Cu	13.7 %wt
Al	4.6 %wt
Fe	4.9 %wt
Zn	4.7 %wt
Sn	2.3 %wt
Other metals	3.8 %wt
Inert (plastics, resins, fiberglass, ceramic)	~66 %wt

Table 1 Composition of the PCBs used in the simulation

2.1.2 Leaching section

The operating conditions of the first leaching stages are the following:

- H_2SO_4: 1.8 mol/L;
- H_2O_2: 1.96 mol/L;
- pulp density: 15 % wt/vol.
- reaction time: 1.5 h;
- temperature: 25°C;
- mixing speed: 250 rpm.

The reactions which take place are:

$$Cu + H_2SO_4 + H_2O_2 \rightarrow CuSO_4 + 2\ H_2O \tag{1}$$

$$Sn + H_2SO_4 + H_2O_2 \rightarrow SnSO_4 + 2\ H_2O \tag{2}$$

Copper has to be removed first, otherwise it would consume thiourea in the second leaching, competing with gold. Instead, in the first step, the precious metals are not leached.

The 1st leaching stage works in a countercurrent operation mode, as shown in Fig.2. The pregnant solution from the second stage is recycled back into the first reactor in order to increase the concentration of the base metals of interest; furthermore, this configuration results in a smaller volume of the spent solution generated at the end of the process and, hence, lower operating costs.

The concentration of copper is around 49 g/L as $CuSO_4$ (90 % extraction yield in both reactors), whereas tin is nearly 5 g/L as $SnSO_4$ (80 % and 50 % extraction yields in the first and second reactor, respectively) after the countercurrent 1st leaching stage.

After filtration, the PCBs undergo second leaching, with different operating conditions:

- H_2SO_4: 0.2 mol/L;
- $Fe_2(SO_4)_3$: 22.5 g/L;
- $CS(NH_2)_2$ - (thiourea): 20 g/L;
- pulp density: 10 % wt/vol.
- reaction time: 1 h;
- temperature: 25°C;

- mixing speed: 250 rpm.

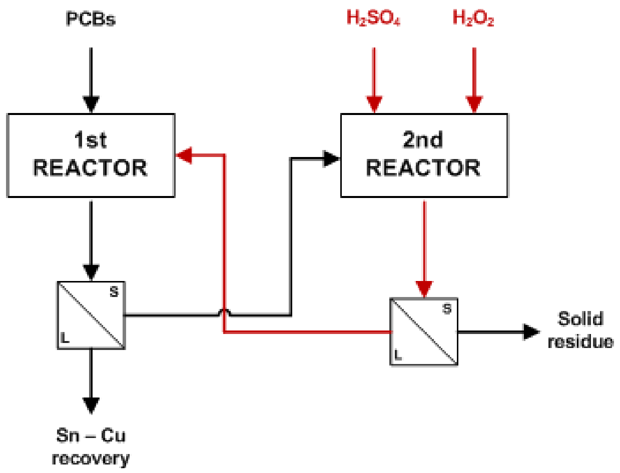

Fig. 2 Countercurrent leaching in the 1st stage.

Fe^{3+} ions are necessary as oxidants and thiourea as a complexing agent, whereas sulfuric acid assures that the pH remains acidic, avoiding iron precipitation. The simplified reactions that involve precious metals are:

$$2\ Au + Fe_2(SO_4)_3 + 4\ CS(NH_2)_2 \rightarrow [Au(CS(NH_2)_2)_2]_2SO_4 + 2\ FeSO_4 \quad (3)$$

$$2\ Ag + Fe_2(SO_4)_3 + 6\ CS(NH_2)_2 \rightarrow [Ag(CS(NH_2)_2)_3]_2SO_4 + 2\ FeSO_4 \quad (4)$$

The leaching mechanism of gold and silver leaching by thiourea in a sulfuric medium and the presence of Fe^{3+} ions leads to the oxidation of thiourea to formamidine disulfide [9]; the latter is not stable in acid solution and oxidation conditions and decomposes irreversibly to thiourea, cyanamide, and sulfur. Hence, if thiourea is not added in excess, the such reagent will not be sufficient for the reactions reported in Eqs. (3) and (4) [31].
After the second stage, the concentration of the Au- and Ag-thiourea complexes is around 48 and 169 mg/L, respectively, the same concentrations of the pregnant solutions entering the related electrowinning processes. The extraction yield of the two precious metals is in the 85-90 % range.

2.1.3 Recovery of Sn and Cu from aqueous solution

The pregnant solution from the 1st reactor undergoes tin precipitation by adding a cationic flocculant, i.e., polyamine, with a concentration of 2.5 mL/L of solution. The polyamine has to be diluted with distilled water to a concentration of 10% vol/vol. The solution is slowly mixed at 50-100 rpm, at room temperature, for 30 min., a sufficient time to precipitate the metastannic acid, which is separated by filtration and dried at 100°C for 2 h [32]. The precipitation yield is nearly 90 %. The metastannic acid can be oxidized at 650°C for 1 h to obtain high-grade tin oxide.
The solution coming from filtration and containing copper sulfate undergoes electrowinning. The cathode is made of copper, whereas the anode is made up of titanium alloy. The current density is 250 A/m^2, voltage 1.5 V, and the deposition time 1 h, room temperature. While copper is recovered at the cathode by reducing Cu^{2+} ions, water is oxidized at the anode, generating H_3O^+ ions. Thus, the spent solution contains acid, most of which can be recycled back to the first leaching stage. This allows the recovery of the remaining copper that cannot be discharged completely, not to lower the current efficiency. The calculated electrical consumption is 2.1 kWh/kg, with a recovery of copper of around 98 % and a current efficiency of 90 %.

2.1.4 Recovery of Au and Ag from aqueous solution

Au and Ag are recovered by two sequential electrowinning stages, with the following operating conditions:

- Gold anode: titanium alloy;
  cathode: graphite;
  current density: 50 A/m^2;
  cell voltage: 1.2 V;
  time: 2 h;
  temperature: 25°C;

- Silver anode: titanium alloy;
  cathode: graphite;
  current density: 75 A/m^2;
  cell voltage: 1.4 V;
  time: 2 h;
  temperature: 25°C;

The spent solution from the silver cell can be reused back in the 2nd leaching stage. The number of reusing cycle has to be determined, as other metal impurities like Fe, Zn, Ni tend to concentrate.
The recovery of Au and Ag by the electrodeposition is in the 85-95 % range, and the electrical consumption is 4.1-4.4 kWh/kg per each process. Unfortunately, the current efficiency is rather low, i.e. 15 % for gold and 12 % for silver. These efficiencies can certainly be improved with higher concentration of the precious metals, that could be achieved by a countercurrent thiourea leaching stage or processing PCBs with greater precious metal content.

2.2 Plant description and simulation

The entire process, tested on a lab-scale, was simulated in semi-industrial conditions by the software package SuperPro Designer v.9.5 (Intelligen, USA). The capacity of the plant is 350 tons/year in a batch operating mode (2 tons/batch); nonetheless, this capacity could be fully industrial as the number of PCBs available on the market is not high, considering a thousand collectors already sharing the market, primarily black, and that the breakeven capacity to make the investment profitable can be low. The feedstock composition used for simulation is the same as in Table 1.
The leaching section is composed of two stages: the first one, as said, is composed of two reactors (18 m^3 each) and the relevant plate and frame (P&F) filters in a countercurrent configuration. The volume of the reactor of the second stage is 19 m^3, whereas the P&F filter is equal to the previous two. Tin precipitation is carried out in a fourth reactor (18 m^3), but filtration is performed with a Nutsche filter, more efficient for metastannic acid, which is thus dried in a tray dryer.
The volume of the electrolytic cell for copper is 14 m^3. The pregnant solution from the second leaching stage undergoes electrodeposition of gold in another 12 m^3 electrolytic cell. Afterward, the spent solution is stored in a tank, whereas the operating parameters and cathodes are changed. Thus, the solution is transferred into the same cell for the electrodeposition of silver. The solid residue from the thiourea leaching stage is dried in a small belt dryer fueled with natural gas.
The plant is completed with storage tanks for reagents like water, sodium hydroxide solution, sulfuric acid, and hydrogen peroxide, mixed tanks for storage of intermediate and treatment of waste solutions, pumps, grinder, mill, scrubber, some conveyor belts, etc.. Solid reagents are stored in their original plastic bins or big bags.
The simulation was carried out using the operating conditions reported in the previous paragraph and the reaction yields obtained in the laboratory trials. The H&M balance of the entire plant and the economic analysis were carried out by Microsoft Excel, using data from SuperPro Designer.
The economic analysis was developed according to Peters et al. [33]. It has to be pointed out that the analysis is developed for a plant that does not consider the purchase cost of spent PCBs. Hence, purchasing PCBs represents represents an additional and significant annual operating cost that must be considered when performing the profitability analysis of the recycling plant.
The plant has an estimated useful life of at least 15 years, a construction time of 8 months, and the opportunity cost of capital quantified at 5%. The capital received from third parties is considered, with a 10-year loan and an interest rate of 3%. The plant is supposed to be built in an Eastern European country, and the total level of taxation is set at 25%. The depreciation period for a recycling plant is usually set at 10 years [33].
The following profitability indexes were calculated: net profit, return on investment (ROI), and payback time. Nevertheless, these indexes do not consider the time value of money. The Discounted Cash Flow (DCF) method considers a project's cash flows and assesses the value of money over time. The indicators used for this analysis are the Net Present Value (NPV) which measures the profit achieved by implementing the project. The Profitability Index (PI) measures the wealth achieved per unit of investment, and the Discounted Payback Time (DPBT), quantifies how long it takes for the project to return from the initial investment cost. Besides those indexes, the Internal Rate of Return (IRR), which measures the project's economic return, is also provided.

3. Results and discussion

3.1 Economic analysis

3.1.1 Capital expenditure

The main equipment purchase cost is estimated using commercial offers from suppliers or estimating them from indirect methods indicated in [33]. The auxiliary and unlisted equipment includes a boiler, scrubber, compressor, pumps, cyclone, grinder, mill, belts, and storage tanks. The other items are estimated as a percentage of the equipment purchase cost and proportional to the plant capacity and material treated. The total plant indirect cost (TPIC) items are also estimated as a percentage of the total plant direct cost (TPDC). Thus, the direct fixed capital (DFC) invested in this project is estimated to be 3,516,958 Euro. The capital expenditure (CAPEX) details are reported in Table 2.

Item		**Cost (€)**
Main equipment		1,490,000
Auxiliary and unlisted equipment	30%	447,000
Drums/big bags for packaging		10,000
Laboratory equipment		250,000
Equipment Purchase Cost		**2,197,000**
Installation	12%	263,640
Piping	3%	65,910
Instrumentation	7.5%	164,775
Insulation	1.5%	32,955
Spare parts (2 years)	1%	21,970
Electricals	5%	109,850
Buildings	8%	175,760
Total Plant Direct Cost (TPDC)		**3,031,860**
Engineering, Procurement, Construction	10%	303,186
Supervision, start-up & training	5%	151,593
Contingencies	1%	30,319
Total Plant Indirect Costs (TPIC)		**485,098**
Direct Fixed Capital (DFC)		**3,516,958**

Table 2 Evaluation of CAPEX

3.1.2 Operating expenditure

The operating expenditure (OPEX) is composed of several items listed in this paragraph. The reagents and raw materials required to carry out the process are reported in Table 3. The H&M balance is crucial to estimate reagents, product streams, energy consumption, and all the other running cost throughout the year.

Raw material	**Consumption (tons/year)**
Sulfuric acid (98 %wt)	489
Hydrogen peroxide (30 %vol)	202.7
Ferric sulfate (s)	37.1
Thiourea	26.3
Polyamine (l)	0.57
Water	677
NaOH(s)	2.80
CaO (s)	5.25
PCBs	350

Table 3 Raw materials used in the recycling plant

The total water consumption is around 4183 tons/year, but around 3500 are recycled back into the process after the wastewater treatment or as spent solutions from the electrolytic cells. The rest is discharged into the sewage network to the industrial wastewater treatment plant (WWTP). Hence, the net consumption is 677 tons/year.
The total cost of raw materials is 218,400 Euro/year. Another important annual cost is due to utilities, mainly represented by electrical energy and natural gas. The electrical consumption is nearly 227,000 kWh/y, for a total installed power of 208 kW. Hence, a power station of 215 kW is required to the plant. The two small dryers consume natural gas: the total consumption amounts to 51,794 Euro/year. Other utilities consist of technical and instrument compressed air produced on-site by one compressor.
The plant runs in batch mode, whose net duration is 10 h. The personnel comprises of one technical director with administrative tasks, four skilled workers with maintenance duties, and a laboratory analyst for routine analysis and quality control. The annual cost is 248,430 Euro/year. The waste item accounts for the disposal of the PCB residue from the second stage leaching and some sludge and powder from the scrubber and dust filters. The wastewater treatment takes into account the cost of the treatment of the spent solution, carried out on-site and the fee for discharging a small amount of treated water into the sewage network. Consumables include replacing cathodes and anodes consumed and other components like gaskets, filters, lubricants, etc.
The greatest expense is the loan payment, which amounts to 343,351 Euro/year (3 % interest rate). The complete list of the operating costs is shown in Table 4.

Item	**Cost (€/year)**
Raw Materials	218,400
Labor-Dependent	248,430
Loan payment	343,351
Laboratory/QC/R&D	20,000
Consumables	60,000
Waste Disposal	38,325
Wastewater treatment	42,061
Utilities (electric energy + methane)	51,794
Transportation	15,000
Miscellaneous	10,000
Running Royalties	-
Maintenance & Insurances	36,418
Annual operating costs	**1,083,779**

Table 4 Evaluation of OPEX

3.1.3 Economic evaluation report

The economic evaluation summary is reported in Table 5. The revenues were calculated according to the market prices of metal commodities, i.e., copper, gold, and silver [34]. The plant produces 42.4 tons/year of copper, 7.2 tons/y of tin oxide, 43.8 kg/year of gold, and 85.8 kg/year of silver, all commercial-grade metals. They are indicated in Figure 2.
Working capital is the investment in temporary or consumable materials, i.e., tied-up funds required to operate the business. It is the sum of raw material, labor-dependent cost, waste disposal, and utilities, i.e., the minimum money to run the plant during the first year. The tax rate is 25% of the gross profit (gross profit = revenues - operating costs). Net profit = gross profit - taxes + depreciation [33]. The profitability indexes are calculated as follows:

$$Gross\ margin = \frac{Gross\ profit}{Revenues} \tag{5}$$

$$ROI = \frac{Net\ profit}{Total\ investment} \tag{6}$$

$$Payback\ time = \frac{Total\ investment}{Net\ profit} \tag{7}$$

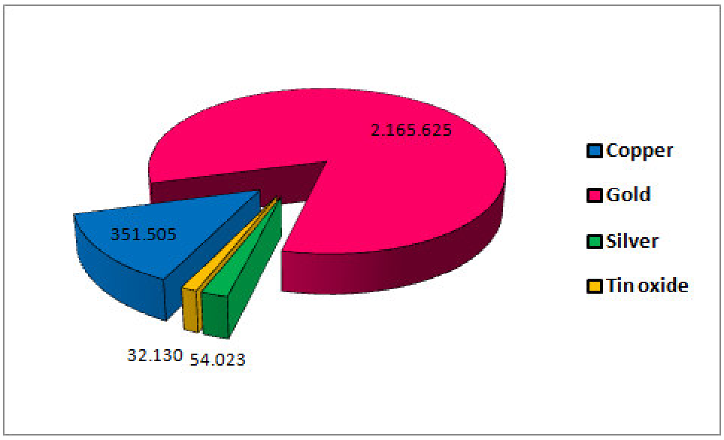

Fig. 2 Annual revenues of the plant (Euro)

The gross margin is 58.4%, with a good return on the investment (ROI), amounting to 27.8 %, and a payback time of 3.6 years. The overall project based on the recycling of PCBs is highly profitable.

Item		
Direct Fixed Capital	3,516,958	€
Working Capital	556,949	€
Start-up cost	15,000	€
Up-front R&D	10,000	€
Total Investment	4,098,907	€
Revenues	2,603,283	€/year
Annual Operating Costs	1,083,779	€/year
Gross Profit	1,519,503	€/year
Taxes	379,876	€/year
Net Profit	1,139,627	€/year
Gross Margin	58.4	%
ROI	27.8	%
Payback Time	3.6	years

Table 5 Profitability summary

Gross profit, ROI, and payback time are indexes that do not consider the time value of the money. Hence, the DCF analysis is also developed (detailed calculations are not reported here). To be conservative, an annual inflation rate of 1.5 % is considered when calculating the operating costs; nevertheless, a 2 % annual increase in the commodities price is also considered [35]. The depreciation period for a recycling plant is usually set at 10 years [33].

The profitability index is 2.6 and is calculated as the ratio of the present value of the after-tax cash inflows to the present value of the cash outflows or capital items. This means that the business generates 2.6 Euro of profit for every Euro invested in the project. The DPBT was also calculated and resulted in 2 years, i.e., the year in which the sum of the discounted cash flow becomes positive.

The IRR is the interest or discount rate for which the project's net present value (NPV) equals zero. In the present analysis, the IRR is around 150 %. Moreover, the NPV is about 10.7 MEuro. Overall, also this alternative analysis confirms the outstanding profitability of the PCB recycling business. Considering the economy of scale, the indexes calculated above even enhance with the increase of the plant's capacity.

Nevertheless, as already pointed out, the analysis was developed considering that PCBs are directly collected from WEEE collection points. In case PCBs have to be bought on the market, the profitability analysis still shows good indexes for purchasing costs lower than 1500 Euro/ton, which results in an additional operating cost of 525,000 Euro/year. The investment is not profitable for PCB purchasing prices greater than that threshold.

4. Conclusions

The present paper focused on the techno-economic analysis of a plant that recycles PCBs, the most interesting WEEE in terms of metals and intrinsic economic value. The analysis was developed for a plant with 350 tons/year capacity. The technical analysis was based on an industrial process whose main stages were tested on a lab-scale. The profitability analyses, developed in case of no cost for collecting PCBs, demonstrated the overall benefit of investing in such a recycling business. The DPBT is only 2 years, the NPV at 5% of interest is 10.7 MEuro, and the profitability index is 2.6. Nevertheless, if PCBs have to be purchased on the market, the profitability of the investment is guaranteed for prices lower than 1500 Euro/ton.

Acknowledgments

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