# Simulation studies of n-heptane/toluene separation by extractive distillation using sulfolane, phenol, and NMP

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## Abstract

Simulation studies of n-heptane/toluene system were performed using Aspen Plus. The comparative study of different entrainers: N-Methyl-2-Pyrrolidone (NMP), Phenol, and Sulfolane for separation of this system was done. Sensitivity analysis was carried out to optimize flowsheets. The criteria to determine the appropriate solvent for the said system included, the total cost calculated by the Aspen Process Economic Analyzer, amount of the solvent consumed, cost of the solvent, and purity obtained during the separation process. For the given system, sulfolane was found to be the appropriate solvent.







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## AI based Sustainable Approach for Metal Extraction from E-Waste: A Comprehensive literature review

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Abstract— Due to the overwhelming increase in the electrical and electronic products every year the rate of discarding older or damaged electrical or electronics products has also increased. Proper disposal or recycling of the discarded products of Electrical and Electronic waste is of major concern for research as it contains some harmful substances. There are several literatures being reported for extraction of electronic waste using different methods. Electro chemical, Pyrometallurgical, Hydrometallurgical and Bioleaching are the most enhancing methods to extract metals. Among all these methods, electrochemical is one of the most efficient method reported. (80-95) % of the metal extraction is reported in former mentioned method by using different electrolytes. Additionally, different combinations of electrodes are used to separately extract the metals from E-Waste after the pretreatment and chemical leaching of the Waste. To identify the best efficient electro chemical methods and its challenges for extraction of metals from e-waste, a literature review of research papers has been carried out during the period between 2010 to 2019. This work will conclude the comparative analysis of all these methods for extraction of metals from e-waste based on some factors like: Energy efficiency, Environment friendliness, Economical and Efficient extraction of metals.

Keywords—Electrolysis, Electrochemical, Electrowinning, E Waste

#### I. INTRODUCTION

With reference to the advancement of technology use of Electrical & Electronic products has increased rapidly which has enhanced the Productivity as well as abundant rate of Waste materials. World produces 50 million tonnes of E-Waste in a year which will go up to 120 million by 2050. But presently only 20% of the waste is recycled [1][2][3]. The primary element for almost all the electrical and electronic products are Printed Circuit Board. Composite materials for PCB contains 40% resin, 30% glass fiber, 30% metal [4][5]. The E Waste is classified into Used and non-functioning but repairable Electrical & Electronic Equipment, Used and non-functioning and nonrepairable wasted electrical and electronic

products. [6]. Handling of E Waste is of foremost priority because diverse hazardous materials exists in it. It contains several toxic elements such as Lead, barium, beryllium cadmium, PAHs and mercury [6][7]. These hazardous contents have both environmental & human health impact. Heavy metals are present in several effluents of chemical processing industries. Metals need to be separated before discarding it in the environment. Apart from effluents from industries, a large amount of heavy metals are present in ewaste. If these metals can be recycled successfully, it can be a huge savings in the production as well as for the natural resources. Electro chemical [2], Pyrometallurgical, Hydrometallurgical and Bioleaching [8][9][10] are the mechanism used for extraction of metals from E Waste shown in Fig. 1. The Electrolysis process for extraction of metals is analyzed in this work with various research publications. In literature it has been found that for the separation of metals from printed circuit boards, the PCBs are broken into pieces and chemical leaching is applied to release the metals in the acids or any other reagents.

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Fig 1. Pretreatment methods for E-Waste. [10]

The pretreatment of E Waste is almost similar for all the methods. After manual segregation of waste PCBs &CPU Slots, they are considered for recovery of metal. The pretreatment follows some steps as shown in Fig. 1 [11][12]. These pretreatment methods help to separate metal and nonmetal from the waste powder. Nonmetallic parts can be separated using Eddy Current method, Electrostatic separator or Density separation [13][14]. Electrostatic separation is commonly used for segregation of Metallic Parts from nonmetallic parts. Crushed PCB Boards will undergo the chemical leaching process so that all the metals get dissolved in the solution.

After pretreatment wasted printed circuit the powdered waste will be used for recovery of metals. Table 1 showcases the presence of Metals in PCB Board [15].

#### TABLE 1: % METAL CONTENT [14]

Copper	48.8%
Tin	21%
Lead	12%
Nickel	2%
Zinc	1.5%
Gold	0.08%
Silver	0.04%
Other	14.56%

It is evident from the diagram that majorly present material is Copper, Tin, Lead. Metal concentrations are determined by Inductively Coupled Plasma Optical Emission Spectrometry(ICP-OES).[3] [15][16].

II. C	COMPARISI	ON OF VARIOU	JS METHOI	DS
TABLE 2 D	IFFERENT	METHODS TO	EXTRACT	METALS

Ref. No.	Methods to extract Metal	Process	Remarks
[9]	Hydrometallurgical	The e-waste first dissolved into the alkali solutions and then extracted metal using precipitation, Cementation and Solvent Extraction.	The Process is similar to extract metals from natural ores but due to the complex behaviour of E Waste makes the process more complex.
[10]	Pyro metallurgical	The E-Waste is melted with various flux components as slag formatives. The molten metals are collected to recover metal from it.	Pyrolysis of E- Waste gives rise the toxic gases which needs to deal carefully.
[8]	Bioleaching	This is a bacterial leaching process where several microorganisms used.	The process is very slow compared to the other processes.
[15]	Electrolysis	After the Chemical leaching of E-Waste, the metals were segregated using electrolysis process.	Reuse of Electrolytes can be done to extract metals.

The Comparison of Various methods [17] shown in Table 2. The Hydrometallurgical and Pyro metallurgical process which is more similar to the process of extraction of metals from natural ores. But due to the complex nature of E-Waste the process is more complex. Acidithiobacillus ferrooxidans, Acidithiobacillus ferrooxidans microorganisms [8] used to extract metals in the Bioleaching process. The process is more ecofriendly compared to the other process but the process speed is very slow in this case. The Electrolysis process can be done in a ecofriendly way to extract metals from E-Waste.

#### III. MATERIALS AND METHOD

#### A. Sample Preperation

The PCB Powder obtained from mechanical pre-treatment methods first dried to  $105^{0}$ C temperature [3]. In few literature where a particular component has taken, PCB Powders are dried to  $70^{0}$ C temperature [16]. The process is shown in Fig 2.

Selection of Electrodes for Electrolysis
Analyze the different combination of component presents
Digested liquid was analyzed by Inductively Coupled Plasma Optical Emission Spectrometer
₹۶
Digested with different electrolyte System
Dried at 105° C for 24 hour
Pretreated Waste Powder

Fig: 2: The process to prepare the sample

As shown in Fig 2 the E Waste which obtained by the pretreatment method will undergone for electrolysis process to extract metal from it. Selection of the electrodes will be based on the metal to be extracted.

TABLE 3 DIFFERENT CONCENTRATION OF ELECTROLYTES

Various Electrolytes are used to dissolve the Grinded PCB Board. The experiments were carried out at different concentration of solvents [16] to find out more efficient concentration for better extraction. The solvents generally used to dissolve the e-waste are HCl, HNO<sub>3</sub>, NaCl & H<sub>2</sub>O<sub>2</sub>. From Table 3 it can be identified that fourth system HNO<sub>3</sub>-H<sub>2</sub>O<sub>2</sub>-HF as the best extraction rate when compared to other system. For this system is the concentration maintained between 90%-100% then the metal extraction is almost 99% as reported.



Fig. 3: % Concentration vs. % Metal Extraction

From the Table 3, % Concentration of Solvent Vs. % Metal Extraction graph shown in Fig. 3. The Graph clearly shows the highest metal recovery at 100% of concentration. The solvent used in [17] & [18] gives the extraction efficiency around 100% which consists of HNO<sub>3</sub>.

#### B. Electrolysis Process

For the same four systems of Table 3, the electrode combinations checked with the efficiency of current. In most of the case polytetrafluoroethylene electrochemical reactor of approximate 100 cm<sup>2</sup> area is used. The Selection of electrodes are based on the metals to be extracted. Membrane cell is used separate the Anode and the cathode chamber. As a membrane cell filter cloth is used in maximum cases. The extracted metal is collected from the cathode chamber.

Table No. 4 shows the summarized analysis for the above four systems. Metal Extracted in all the cases are copper. It is seen from the table 4 that to extract a particular metal the same material cathode enhances the extraction rate.

The cell potential is maintained in such a way that the ion

Paper Refere nce	System	Electrolyt e	A	В	A	В	A	В
[15]	HCI- HNO3 – HClO4 - HF	30% NaCl, 55% HCL and 5 % H <sub>2</sub> O <sub>2</sub>	90	88	95	91	100	95
[3]	HNO3 - HF- H2O2	40% HNO <sub>3</sub> & 60% H <sub>2</sub> O <sub>2</sub>	90	83	95	86	100	90
[17]	HNO3 - HF- HClO4	3:1 HCL & HNO <sub>3</sub>	90	98	95	98	100	99
[18]	HNO3 - H2O2 - HF	30% HNO <sub>3</sub> & 70% H <sub>2</sub> O <sub>2</sub>	90	99	95	99	100	99



exchange from Anode to cathode should be according to the electro chemical series for a particular metal to be extracted.

TABLE 4	DIFFERENT	ELECTRODE	ANALYSIS	FOR	METAL
EXTRACTIO	N				

Ref.	Anode	Cathode	Membrane Cell	%Curre nt Efficienc y	Metal Extractio n %
[15]	Rhodium- plated titanium plate	High- purity titanium plate	Acid resistant filter cloth	88	95
[3]	Graphite stick	Titanium mesh	Installed porous ceramic (50 µm)	92	90
[17]	Ruthenium- plated titanium	Titanium	Anti-Acidic filter cloth	95	99.1
[18]	Rutheniumco ated titanium sheet	Copper sheet with 99.5% purity	Double- layered filter cloth.	95	99.3

#### IV. CALCULATIONS

The metal powder which get stuck into the cathode is collected after every experiment. The collected metal powder is washed with absolute ethanol for many times, after which it gets dried in vacuum[15]. The dried metal powder is then dissolved by the electrolyte system and analyzed by ICP-OES. To obtain the character phases and morphology of the collected metal powders scanning electron microscope was used. Following calculations was used to calculate extraction rate and efficiency:

The influence of utilizing the electrolyte again for the electrolysis process, the extraction rate of metal (X) can be determine by using the following equation:

$$X = (M_A + M_B) / M_X * 100\%$$

where

M<sub>A</sub> is the extracted mass (g) of metal;

 $M_B$  is the mass (g) of the metal present in the electrolyte;

 $M_X$  is the weight (g) of the total metal in waste CPU slots.

Separation rate =  $(M_1 + M_2 + M_3)/Metal$  present in wasted powder \* 100 %

where M1 is the extracted mass (g) of metal from cathode chamber;

M2 is the mass (g) of the metal present in the electrolyte; and M3 is initial mass (g) of the cu at the start of the experiment.

Efficiency of energy can be calculated by the efficiency of current of copper (Y) as copper is the principal extracted element from the wasted powder.

 $Y = M_4/M_5 * 100 \%$ 

Where

M4 is the extracted mass (g) of the copper in the cathode chamber which possess some charge; and

M5 is the theoretical calculated electrode decomposition mass of copper (g) by Faraday's law for the similar quantity of charge as M4, g;

Where  $M_5 = I * t * C$ 

I is the value of current during electrolysis, A;

t is the electrolysis time, h;

C is the electrochemical equivalent of Current efficiency.

#### V. EFFECT OF CURRENT DENSITY

During the metal decomposition the initial current density is greater than the limiting dissemination current density. It is also observed that the efficiency of the current will be low when the density of the current is at the lower side. This is because of the discriminatory nature of hydrogen evolution. On the other side if the current density is increased the efficiency of the current will also increase and the metal decomposition in the cathode chamber will increase.

VI. ARTIFICIAL INTELLIGENCE TECHNIQUES FOR EXTRACTION

A mathematical model is proposed to predict the recovery of Cu from E-Waste using Genetic Algorithm [18] with a correlation coefficient of 91.46%. For optimize the process conditions response surface methodology was used.

Artificial Intelligence Techniques can be used to predict the recovery of metal. With reference to the study [19] conducted for the prediction of Cu recovery, the author generated two Cu recovery prediction models using the leaching method. For both the models, more than 95% of the prediction quality is achieved.

#### DISCUSSION

It has been seen from all the work that precious metals are not frequently recovered using Electrolysis. Mainly all the research work focuses on recovery of copper. More than 30 metals are present in PCB Boards. It will not be economically viable to extract only one or two metals instead all available metals should be recovered. Further research work needs to be done for this.

The Metal to be extracted should be used as cathode to get maximum extraction of the metal.

The Current efficiency is maintained around 80-85 % which indeed effects the energy balance of the process. It can be increased beyond 90% to regulate the power balance.

Artificial Intelligences method can be used to predict the recovery of copper for better extraction efficiency of metals.

#### CONCLUSION

Electrochemical is the most efficient method as it can extract more than 95% of the metal. Using chlorine component in the solvent makes the extraction faster. Using Artificial Intelligence Methods Extraction rates can be predicted which will help to identify proper concentration of electrolyte for metal extraction.

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