RECOVERY OF SPENT IPA BY USING EVAPORATION PROCESS

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ABSTRACT

Spent Isopropyl Alcohol (IPA) that not recovered must be managed as schedule waste which is quite costly and give significant impact to the environmental issues. Thus, the main objective of this research is to suggest the reuse approach for spent IPA by using simple evaporation process because the IPA is easily to vaporize. The spent IPA recovery is studied by determine the effect of operating temperature and volume of spent IPA to the evaporation process. Besides, by analyzing the concentration of precious metals in the spent IPA, it will guide further research on precious metal recovery. The spent IPA taken from an electronic company will be recovered by using rotary evaporator that is easily to be handled. In the experiments, increasing in temperature will increased the evaporation rate because more heat is transferred to the spent IPA. Therefore, the reusable IPA vapour can easily released since the kinetic energy of spent IPA molecule is also increasing. In varying the spent IPA volume, 98.3% of the spent IPA can be recovered to reusable IPA where another 1.7 % will be in concentrated residual that consists of discarded solder paste in surface mount technology process. Besides, by using atomic adsorption spectrometer, metal concentration after leaching process is 5.214 ppm of copper and 0.8213 ppm of silver. In conclusion, high percentage of spent IPA can be recovered at the optimum temperature 80 ^{o}C, and concentration of metal in the spent IPA can be determined. Thus, it is recommended for the electronic company to optimize the usage of IPA.

ABSTRAK

Isopropil alkohol (IPA) yang telah digunakan akan diurus sebagai bahan buangan terjadual yang memerlukaan kos yang mahal dan memberi impak kepada isu-isu alam sekitar. Justeru, objektif utama kajian ini adalah untuk mencadangkan satu pendekatan untuk merawat IPA terpakai melaului proses penyejatan memandangkan IPA mudah meruap. Proses mendapatkan semula IPA yang boleh digunakan akan dikaji dengan mengnalpasti kesan suhu operasi dan isipadu IPA terpakai terhadap proses penyejatan. Selain itu, analisa yang dijalan terhadap kangdungan logam yang terdapat di dalam IPA terpakai akan membantu kajian seterusnya terhadap pengekstrakan logam berharga. IPA terpakai yang diambil dari sebuah syarikat elektronik akan dirawat menggunakan alat penyejatan berputar yang mudah dikendalikan. Dalam kajian ini, peningkatan suhu operasi akan meningkatkan kadar penyejatan IPA kerana haba yang dipindahkan kepada molekul molekul IPA juga akan meningkat. Justeru wap IPA dibebaskan dengan mudah oleh larutan IPA terpakai kerana tenaga kinetik molekul-molekul itu juga meningkat. Dalam pada itu, 98.3% daripada IPA terpakai dapat menghasilkan IPA yang boleh digunakan semula manakala selebihnya membentuk satu larutan pekat seperti gel. Selain itu, hanya 5.214 ppm kumprum dan 0.8213 ppm perak dapat dilarutkan oleh asid hidroklorik melalui proses larut lesap. Oleh itu, syarikat elktronik ini di sarankan agar mengoptimakan menggunaan IPA.

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LIST OF ABBREVIATIONS

Ag	Silver
AAS	Atomic Mass Spectrometer
IPA	Isopropyl Alcohol
Cu	Copper
OSHA	Occupational Safety and Health Association
HCl	Hydrochloric Acid
PCB	Printed Circuit Board
SAC	Stanum-Argentum-Copper
SMT	Surface Mount Technology
Sn	Tin
Pb	Lead

CHAPTER 1

INTRODUCTION

1.1 Research Background

Recently, Free lead Solder Paste is widely applied in Surface Mount Technology (SMT) assembly for printed circuit board manufacturing (PCB) in electronic industry. It is consist of definitely non volatile metal alloy and flux medium in which tin (Sn)-silver (Ag)-copper (Cu) alloy with term SAC alloy is the famous choice to replace Sn-lead (Pb) solder paste (Biocca, undated).

In motherboard manufacturing, a totally naked PCB will be placed in screen printing machine to receive a pattern of solder paste. The metal mask is play the important role in giving the required pattern of solder paste in which it consist of small hole that will let the sticky solder paste printed on the PCB. After received solder paste, all components are stick by component placement machine and then get into reflow oven to firm the joint.

Isopropyl alcohol (IPA) then will be used to remove unwanted solder paste from metal plate and PCB in order to reuse the metal plate and maintain the quality of the final product that might be affected by unsuitable solvent. There is some method that had been used to clean the metal plate using IPA includes ultrasonic equipment. A vented room is provided in the SMT area for cleaning process because IPA can give effect to human health because over exposure will affect the central nervous system.

1.2 Problem Statement

Pure IPA had been used by many industries to meet the desired of customer demand on the high quality of product. Spent IPA may contain metals alloy because it has been used as cleaning agent to remove solder paste and flux medium in PCB. Spent IPA recovery will give significant impact to the environment since it will reduce the amount of schedule waste. Besides, spent IPA recovery is required to overcome the issues that might occur due to solvent shortage potential, solvent cost and schedule waste disposal cost. Thus this spent IPA need to be treated from the existence of metals and flux medium by some solvent recovery methods so that it can be reused again.

1.3 Research Objective

The objectives of this research are:

a) To suggest the reused approach for spent IPA by using simple evaporation process.
b) To analyze the effect of operating temperature and volume of spent IPA to evaporation rate.
c) To identify the existence of precious metals in spent IPA for further research on heavy metal recovery.

1.4 Research Scope

The sample of spent IPA is taken from electronic company at the SMT area where the spent IPA is predicted to contain metal alloy and flux medium. The simple evaporation process had been used to recover the reusable IPA because pure IPA has relatively low boiling point compared to the boiling point of solder paste. The existence of water can be neglected since no portion of water is added in removing cleaning process. Thus, once again, simple evaporation can be used because no azeotrope of IPA-water will form. Rotary Evaporator is choose to run the experiment because it is easy to operate and can give better heat transfer.

The effect of operating temperature to evaporation process will be determined by heating 300 ml of the Spent IPA at different temperature ($50^{o}C$, $55^{o}C$, $60^{o}C$, $65^{o}C$ $70^{o}C$, $75^{o}C$, $80^{o}C$ and $85^{o}C$) for 20 minutes. Meanwhile, the volume of spent IPA will be varied (0.2 L, 0.4L, 0.6L, and 0.8L) to analyze the amount of concentrated residual mass and reusable IPA volume.

In leaching process, only hydrochloric acid (HCl) is used as the cleaning agent in which, as stated by Seng (2006), HCl can give above than 90% of leaching percentage.

In order to determine the concentration of metals in spent IPA, the mass of concentrated residual will be varied (0.2g, 0.4g, 0.6g, 0.8g). Atomic Adsorption Spectrometer (AAS) is used to determine the concentration of copper (Cu) and silver (Ag) that had been leached from the concentrated residual since the spent IPA is taken from SMT area.

CHAPTER 2

LITERATURE REVIEW

2.1 Solvent Recovery Method

Many industries keep using solvent although it is costly because solvent is having a great influence in producing high quality of final product. Thus, it is difficult to find a replacement for solvent over the year. Unrecovered waste solvent will be managed as schedule waste, thus solvent recovery is very important in chemical industries to help in reducing the amount of schedule waste and this will reduced disposal cost of waste solvent (Fox and Knox, undated.). Solvent recovery is also done for Occupational Safety and Health (OSHA) compliance to save the environment. In addition, focusing on solvent recovery will become more important because of solvent price and potential shortage of solvent in future (Mhd Said, 2008).

There are some methods in waste solvent recovery including thermal regeneration which involve evaporation and distillation process. Recent decade, membrane technology quite popular and this will involve hybrid distillation or called as pervaporation as mention by Araki, S et al. (2011) and Singha, N.R. et al. (2009). Pervaporation can be use in separating azeotropes for close boiling point mixtures and thermally sensitive compounds. This method has less energy consuming, but it is complex to operate. Thus, pervaporation are not the best choice for effective solvent recovery from waste solvent for certain solvent that might require simple process such as spent IPA with neglecting water composition.

2.1.1 Evaporation Process

It is known that evaporation is a thermal separation process which is widely used to vaporize the volatile component. According to Geankoplis (2003, p.4), evaporation process refer to evaporation of volatile substance from a non volatile solute for example; removal of water from salt solution. Similarly, GEA Process Engineering (2011) stated that concentrated solution can be determined by boiling out the volatile substance from the liquid where in many cases, concentrate resulting from the evaporation process is the final product. For some cases, such as in solvent recovery, the volatile component is the main product. According to GEA institution, the evaporation processes fall into two general categories which are film type evaporation and suppressed boiling type evaporation.

The generation of a thin product film over a heat transfer surface is involved to enhance minimum resistance to heat transfer in film type evaporation. The optimum film boiling occurs within the evaporator body is one of the important parameter. Besides, it is important to take caution to ensure the wetting rate of continuous film hydraulic and to prevent nucleate boiling; if not, the rate of heat transfer will fall off dramatically, and the rate of scaling on the heat transfer surface will significantly increase. Meanwhile, suppressed boiling type evaporation involves superheating a product above its boiling point while maintaining a backpressure within the system to prevent boiling in the evaporator body. This is commonly referred to as a "heat and flash" principle.

Below are the most important requirements that stated by GEA to build evaporation plant:

a) Product characteristics, including heat sensitivity, viscosity, corrosiveness, foaming tendency, fouling and precipitation, boiling behaviour
b) Capacity and operation data, including quantities, concentrations, temperatures, annual operating hours, change of product, controls automation which required operating media, such as steam, cooling water, electric power, cleaning agents, spare parts

c) Capital and other financial costs
d) Personnel costs for operation and maintenance
e) Standards and conditions for manufacture, delivery, acceptance
f) Choice of materials of construction and surface finishes
g) Site conditions, such as space, climate (for outdoor sites), connections for energy and product, service platforms
h) Legal regulations covering safety, accident prevention, sound emissions, environmental requirements

2.2 Isopropyl Alcohol

Known as rubbing alcohol, isopropyl alcohol (IPA) is widely used as cleaning agent and solvent in many industries. In pharmaceutical, the amount of water is more than 70% in waste solvent make Mhd Said (2008) studied on the IPA-water system. On the other hand, in some electronic industries there is less and almost none of water composition in waste solvent. This make the simple separation process can be used to recover the IPA.

Regarding on Safety Data Sheet of IPA by Kinetronic Corporation (undated.), it is a flammable liquid and vapour. It is harmful if swallowed or inhaled and causes irritation to eyes and respiratory tract. If IPA is adsorbed trough skin, it may also cause irritation to skin. Too much exposure then will affect central nervous system. Due to this health issue, Spent IPA has been classified as scheduled waste and need to be threaten prior to disposal. Applying spent IPA recovery will reduce the amount of disposal waste and make the recovery process more environmental friendly.

a) Skeletal View b) 3D View

Figure 2.1: Structure of IPA

Table 2.1: Properties of IPA

Chemical and Physical Properties	
Appearance Clear	Colourless liquid
Odour	Rubbing alcohol
Appearance	Clear
Boiling Point	82 ºC
Melting Point	-89 ºC
Vapour pressure	Vapour Pressure 44 @ 25 ºC (mm Hg)
Vapour Density (Air = 1)	2.1
Specific Gravity	0.79 @ 20 ºC / 4ºC
Evaporation Rate	2.83 (BuAc =1)ACTIVITY
Solubility in Water	Miscible in water
Volatile by Volume	100% at 21 ºC
Reactivity	Data
Stability	Stable
Incompatibility	Heat, flame, strong oxidizers, acetaldehyde, acids, chlorine, ethylene oxide, isocyanates.
Hazardous Decomposition Products	Carbon dioxide and carbon monoxide may form when heated to decomposition.
Conditions to Avoid	Heat, flame, ignition sources and incompatibles
Hazardous Polymerization	Will not occur

Source: LookChem (2008)

2.3 Solder Paste

Solder pastes is an alloy which is melt when heated and flow onto the space between two close fitting parts and creating a soldered joint that has strength and corrosion resistance (Wikioedia 2010). It is produce by mixing the alloy powder and pasty flux that will release first upon heating to prevent surface oxides. The powder filler metal then melts and flow into the joint area to form a reliable, but with recent development of SAC alloy; a proper soldered joint can be achieved.

Figure 2.2: Solder Paste

Resource: NYP Trinity Limited (2009) and Jim Hisert (2008)

SAC alloy usually consist of 96.5 % of Sn, 3 % of Ag and 0.5% of CuO. This solder paste is widely applied in the SMT assembly in which, it also contain flux medium that will have the adhesive propertied to make it stick to the PCB and have firm joint. Because the ingredient of solder paste flux is a tread secret, Biocca (undated.) had summarized the basic ingredient in the solder paste flux as below:

a) Resins solid and liquid types
b) Activators, organic acid and/or hydrohalide
c) Solvents and co-solvents
d) Gelling agents
e) Surfactants
f) Chelating agents

Figure 2.2 show that; when different SAC solder paste were run through a reflow oven at 180 ^{o}C, Paste B has better hot slump properties compared to Paste A which more likely cause bridges, solder balls or mid-chip balling (types of defect associate with lead-free reflow soldering). This proves that, it is important to select lead-free paste with a heat stable gelling agent for fine pitch component.

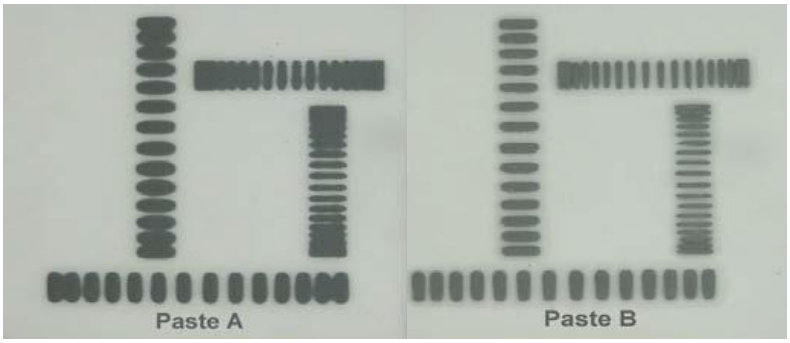


Figure 2.3: Different Type of SAC Solder Paste

2.3 Metal Recovery

Metal recovery also become more important over the year due to exhausted of source, pricing and due to environmental issues. It is stated in The Washington post (2012), Copper for March delivery gained 2.3 cents to $3.7525 per pound silver for March delivery raised 40.8 cents to $30.543 an ounce. Leaching process is one of the methods had been used to recover the metal in semiconductor waste. According to Tan (2006), increasing in HCl concentration will increase the percentage leaching for both copper and silver. At the end, 4M of HCl will leached the highest percentage of metal leaching for both copper and silver as shown in Figure 2.3.

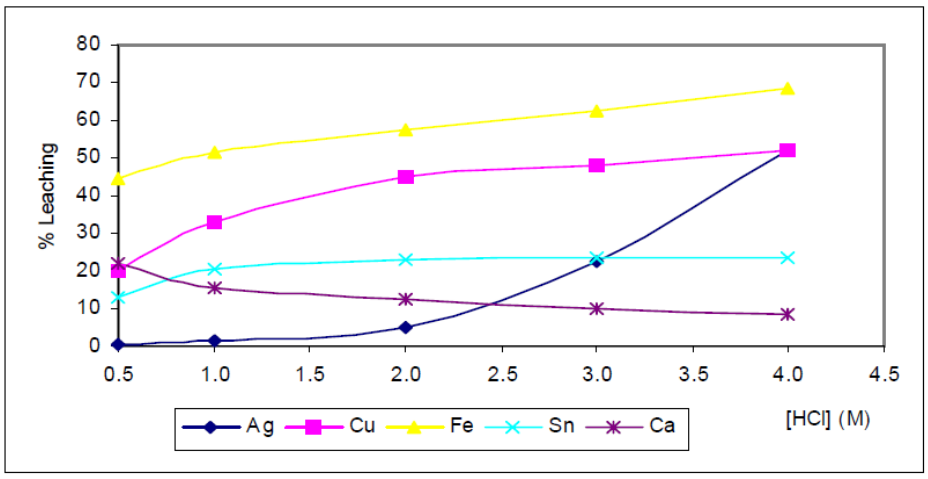


Figure 2.4: Effect of HCl Concentration on the Metal Leaching

Resource: Tan (2006)

2.3.1 Copper

According to SinceView, 'copper' names is take from the Roman name “aes Cyprium” because of the huge amount. Environmentally, copper exist naturally in the air, water and soil which are significant in protecting the environment and health. Maybe not many people realized that copper can also be found in milk, nuts, oysters, and seeds chickpeas. In human health, copper can help maintaining a healthy heart and liver, bone strength and brain development. However as cited by Lenntech (2011), long-term exposure to copper can cause irritation of the nose, mouth and eyes, and it causes headaches, stomachaches, dizziness, vomiting and diarrhea. In addition by taking intentionally high copper may cause liver and kidney damage and even death.

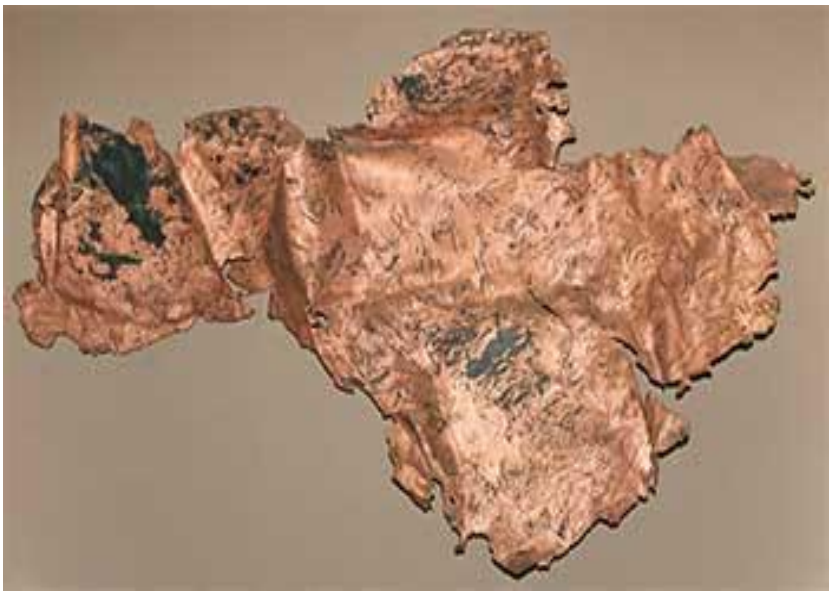


Figure 2.5: Pure Copper

Resource: SciencView.com

Table 2.2: Properties of Copper

Physical Properties	Specific Properties
Malleable and Ductile	Chemical Symbol: Cu
Excellent Electrical Conductor	Atomic Number: 29
Excellent Alloying Characteristics	Atomic Weight: 63.54
Non-Magnetic	Density: 8960 kg m(-3)
Essential Nutrient to Life	Melting Point: 1356K
Resistant to Corrosion	Specific Heat at 293K: 0.383kJkg(-1)K(-1)
Machinable	Thermal Conductivity: 394W m(-1) K(-1)
Formable	Electrical Conductivity (%International Annealed Copper Standard): 100%
Excellent Heat Transfer Characteristics	Electrical Resistivity: 1.673x10(-8) ohm-m
Durable	Crystal Structure: Face Centered Cubic
Recyclable	

Source: International Copper Association

Figure 2.6: Application of Copper

Source: International Copper Association

3.2 Silver

As cited by Lenntecth (2011) pure silver is white, lustrous, soft, very ductile, and malleable and it is the highest electrical conductivity of all metals. However, its greater cost has prevented it from being widely used for electrical purposes. Silver can be attacked by nitric acid (forming the nitrate) and by hot concentrated sulphuric acid but it is not a chemically active metal. Silver compounds can be slowly entering the body tissues, with the consequent bluish or blackish skin pigmentation (argiria) and may cause irritation and allergic dermatitis. At high exposure, it may cause dizziness, breathing difficulty, headaches or respiratory irritation. Meanwhile, extremely high exposure may cause drowsiness, staggering, confusion, unconsciousness, coma or death.

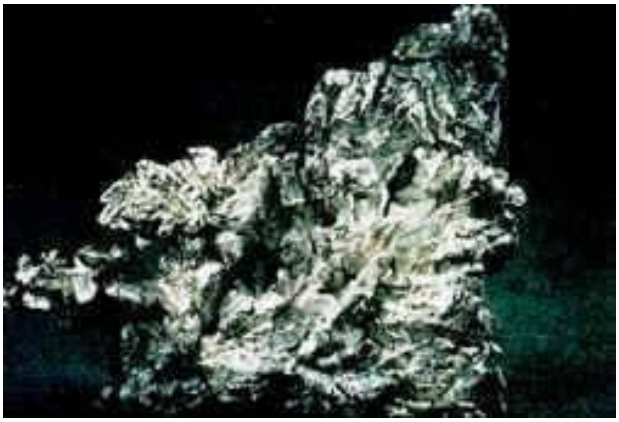


Figure 2.7: Pure Silver

Source: Lenntech (2011)

Table 2.3: Properties of Silver

Physical and Chemical Properties			
Atomic number	47	Ionic radius	0.126 nm
Atomic mass	107.87 $g.mol^{-1}$	Isotopes	11
Electronegativity according to Pauling	1.9	Electronic shell	[Kr] $4d^{10}$ $5s^{1}$
Density	10.5 $g.cm^{-3}$ at 20°C	Energy of first ionization	758 $kJ.mol^{-1}$
Melting point	962 °C	Energy of second ionization	2061 $kJ.mol^{-1}$
Boiling point	2212 °C	Discovered by	The ancients
Vanderwaals radius	0.144 nm		

Souce: Lenntech (2011)

Figure 2.8: Application of Silver

Source: The Silver Institute

CHAPTER 3

METHODOLOGY

3.1 General Procedure

This research will involve evaporation process for solvent recovery and leaching process to determine the concentration of metal for future research on precious metal recovery. Figure 3.1 show the general procedure that has been implemented during the experimental procedure.

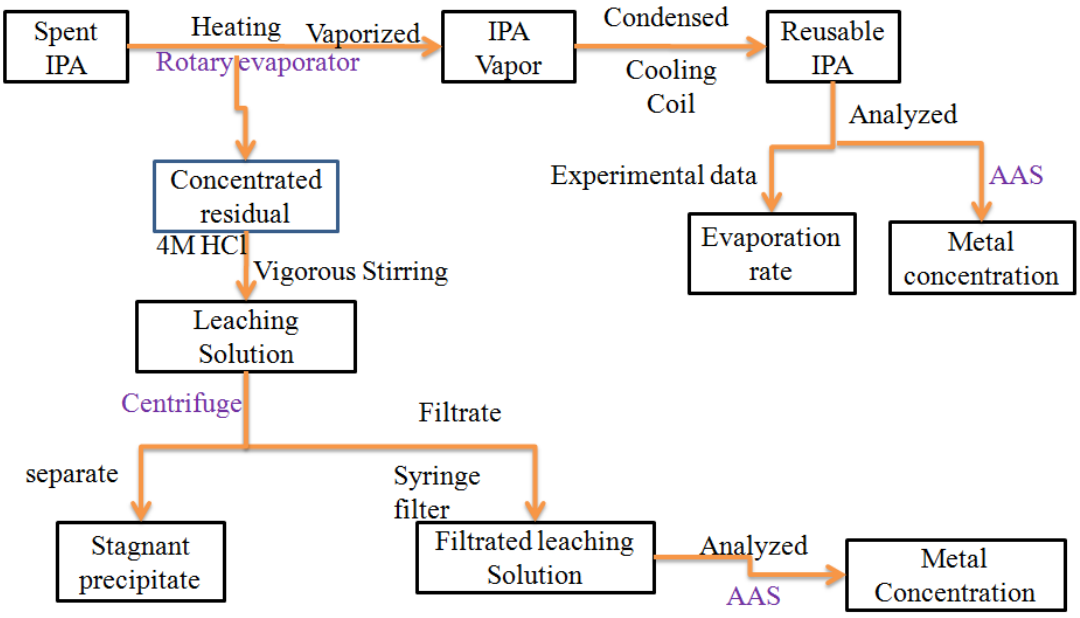
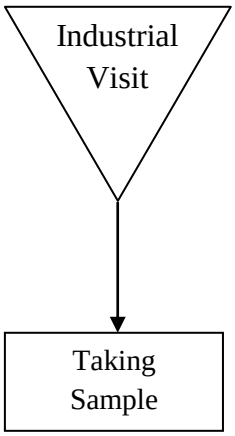


Figure 3.1: General Procedure

3.2 Preliminary Procedure

An industrial visit has been done in electronic company at Bandar Pusat Jengka, Pahang to observe the usage of IPA as cleaning agent. The representative of that company shows the researcher the PCB manufacturing process and cleaning process using IPA. It can be seen that workers who handling the cleaning process are wearing mask and glove. Besides, there is a vented room where the metal plate and metal mask are cleaned by using pure IPA solution. This vented room also place ultrasonic equipment for the cleaning process. An appropriate personal protective equipment (PPE) and good ventilation system are needed because overexposure to IPA can give significant effect to human health. Trough this visit, researcher manage to get the spent IPA from Surface Mount Technology (SMT) area to fulfill the objective of this research.

Table 3.1: Preliminary Procedure

Process Flow	Description
	• The electronic company has good practice in handling IPA solvent and good ventilation system • Lead free of solder paste is used in most area • The composition of solder paste used at SMT area are Sn:96.5, Ag:3% and CuO:0.05%
	• Spent IPA which is taken from SMT area is used as sample • The spent IPA is put into closed container and placed in ambient temperature because it is easily to vaporize and high flammability

3.3 Laboratory Preparation

Under laboratory preparation, the chemical is prepared as mention in Table 3.2. Appropriate PPE such as face mask and rubber gloves are needed because IPA can give irritation to skin and eyes. Ventilation chamber should be in a good condition because IPA is a volatile substance.

Table 3.2: Laboratory Preparation

Process Flow	Description
Chemical ordering ↓ Preparation on Equipment	• Acid - Nitric acid • Leaching Agent - 37% HCL solution • Diluents - Deionized water and ultra pure water • Standard Solution - 1000 ppm copper (Cu) aand 1000 ppm silver (Ag)
	Major Equipment • Lab-scale Rotary Evaporator - To vaporized reusable IPA from spent IPA • Atomic Spectroscopy, AAS - To analyze Cu and Ag concentration Other Equipment • Centrifugation - To remove precipitate • Magnetic Stirrer - To leach metal from concentrated residual achieve and make that aqueous phase achieve equilibrium • Electronic Weighing - To measure concentrated residual mass