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Extraction and Stripping Behaviour Study on Gold Recovery from Printed Circuit Board

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Abstract. Single extraction was used to investigate the suitable condition parameters of temperature and agitation speed in extracting gold, Au (III) from printed circuit board (PCB). Extractant di-(2-ethylhexyl) phosphoric acid (D2EHPA) was mixed with thiourea leaching solution. From the extraction system, the optimised conditions are the temperature of 45°C and the agitation speed of 300rpm and obtained 88% of percentage of extraction (%E). In order to enhance the recovery of Au(III), three samples underwent stripping process, the results from three samples which are sample 1,2 and 3 showed the percentage of stripping efficiency (%S) were 50.37%, 29.58% and 25.64% respectively. The results of FTIR showed the presence of C-H and C-O bond in waste cooking oil and after addition of D2EHPA into the diluent the P-O-H, P-O-C and O-H vibration band were appeared.

Keywords: extraction; D2EHPA; Au(III); temperature; stripping

1. Introduction

Solvent extraction or also known as liquid-liquid extraction is a mass transfer operation which is commercially used in metallurgical industry for selective extraction of metal ions from an aqueous solution. Solvent extraction becomes one the mostly used refinery process of heavy metals because of its advantages such as shortening processing time, high product yield, high selectivity and flexibility and controlled recovery [1]. Various industries including pharmaceutical, biochemical, analytical separations and wastewater treatment are also practising this method. In solvent extraction process, two layers of immiscible liquids are put into contact, where the valuable metal in an aqueous solution will be captured and bind to the extractant in the organic phase. The desired metal ion is purified and concentrated during the process. An extractant is capable of binding metal ions and dissolved in an organic type diluent. The extractant has an active hydrophilic group bound to a long chain hydrocarbon molecule in order to reduce solubility in the organic phase. Among the commercially used extractants, di(2-ethylhexyl)phosphoric acid (D2EHPA), an organophosphorus acids which is the most used extractant because of its chemical stability, high complexing ability and availability in the market.



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A diluent is an important element in solvent extraction process. It is required to reduce the viscosity of the extractant and to ease the flow of the fluids since certain fluids are too viscous or too dense to move from one particular point to another. Waste cooking oil is chosen as the diluting agent in this research due to a non-petroleum based which is safe to the environment, cheap and available everywhere. In order to enhance the recovery of Au (III), stripping process is studied which had not been widely done by other researchers. Stripping process is the back-extraction of recovered metal ion into a new aqueous phase by using sulphuric acid (H_2SO_4). H_2SO_4 is suitable acid acts as the stripping agent because it is non-volatile and commercially available [2]. It is a single-stage process for stripping metal from a loaded-liquid cation exchanger with a dilute mineral acid while simultaneously precipitating a hydrolysed phase [2]. Previous study showed that stripping of gold from quaternary amines with acidic thiourea and air sparging are found to be quite effective [3]. In hydrometallurgy, leaching process is significant to separate components. For example the metal can be recovered from PCB by contacting with soluble liquid solvent. This process converts PCB into liquid forms and undergo solvent extraction. Leaching process is a good method to extract metals by transforming it into cationic species in low pH acid and high concentration [4]. High extraction rate of gold can be achieved at optimum condition with the presence of iron and oxygen as the oxidant [5]. However, in this study, only synthetic Au (III) is used as the replacement of the real PCB due to the constraints of time.

2. Materials, Equipment and methods

2.1 Materials

Materials and chemicals used in this study are gold (III) chloride trihydrate ($\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$) (Sigma-Aldrich, $\geq 49\%$ purity), di-2-(ethylhexyl) phosphoric acid (D2EHPA) (Acros Organics, $\geq 99\%$ purity), iron(Fe^{3+}) (HACH, $\geq 92\%$ purity), thiourea (Sigma-Aldrich, $\geq 99\%$ purity), sulphuric acid (H_2SO_4) (Sigma-Aldrich, $\geq 98\%$ purity) and sodium hydroxide (NaOH) (Sigma-Aldrich, $\geq 98\%$ purity), and waste cooking oil.

2.2 Equipments

Incubator orbital shaker (Lab Companion SK300) is used to mix aqueous and organic phase together while pH meter (Hanna Instrument HI 3221) is used to check the pH value of aqueous phase solution. Atomic absorption spectrophotometer (AAS) (Shimadzu AA-6800) is used to analyse the concentration of gold recovery of aqueous phase after the extraction and FTIR is to examine functional groups in organic phases before and after solvent extraction.

2.3 Methods

2.3.1 Preparation of aqueous and organic phase

A gold solution is prepared by dissolving the 0.2 g of gold (III) chloride trihydrate ($\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$) in 1.0 L distilled water in a volumetric flask. The aqueous phase solution is then made by leaching the gold solution in the thiourea acid and Fe(III) solution. The concentration of gold in aqueous solution should always be smaller than of D2EHPA in the organic phase. The organic phase solution is prepared by mixing measured amount of waste cooking oil with D2EHPA 250 mL conical flask. H_2SO_4 and NaOH are added to control and maintain the pH of the solution.

2.3.2 Extraction procedures

Extraction gold is carried out by simple bench-top studies involving 'shake out tests'. A volume of 10 mL of organic phase is mixed with the prepared gold-containing aqueous phase at 1:1 organic to aqueous volume ratio in a glass-stoppered bottle. The flask is shaken by the orbital shaker at 100 rpm and 30 °C. After that, the mixture is allowed to settle down for a few minutes until the presence of two layers

solution. The pH is adjusted using 1 M of H_2SO_4 or/and 1 M of NaOH to obtain the optimum condition at pH 1.

The pH adjustment and shaking steps are repeated until the desired pH obtained. When pH 1 is achieved, the mixture is transferred into a separating funnel for phase disengagement. Desired compound which appears in aqueous layer prior to extraction will be extracted and transported to organic solvent layer at the top. The aqueous sample is analysed using AAS while chemical bonds in organic phase is determined by using FTIR prior to appropriate filtration and dilution. The percentage of extraction (%E) of gold is calculated from the initial concentration of gold in the aqueous phase $[\text{Gold}]_{i,\text{aq}}$ and after extraction $[\text{Gold}]_{f,\text{aq}}$ by using Eq. (2.1) [4]

$$\%E = \frac{[\text{Gold}]_{i,\text{aq}} - [\text{Gold}]_{f,\text{aq}}}{[\text{Gold}]_{i,\text{aq}}} \times 100 \% \quad (2.1)$$

Where $[\text{Gold}]_{i,\text{aq}}$ is the initial Au(III) concentration in aqueous phase and $[\text{Gold}]_{f,\text{aq}}$ is the final Au(III) concentration in aqueous phase. The experiments are repeated with different agitation speed, 200 rpm and 300 rpm. Next, the experiment is done by fixing agitation speed of 200 rpm and varies the temperature between 25 °C, 35 °C and 45 °C

2.3.3 Stripping procedures

The procedure of stripping process is similar to the extraction process except the use of H_2SO_4 as the new aqueous phase and pH adjustment is omitted in this procedure [5]

$$\%S = \frac{[\text{Gold}]_{f,\text{S}}}{[\text{Gold}]_{f,\text{aq}}} \times 100\% \quad (2.2)$$

3. Results and discussion

3.1 Effect of temperature

The percentage extraction (%E) is measured with the variation of three temperatures and the result is obtained as shown in Figure 3.1 below. The aqueous phase containing 100 ppm of Au(III) is mixed with thiourea and Fe(III) solution, while the organic phase used is waste cooking oil loaded with single extractant, D2EHPA. The extraction was done at pH 1 with the ratio of organic to aqueous is 1:1 and orbital shaker agitation speed was set at 200 rpm for 15 minutes.[6]

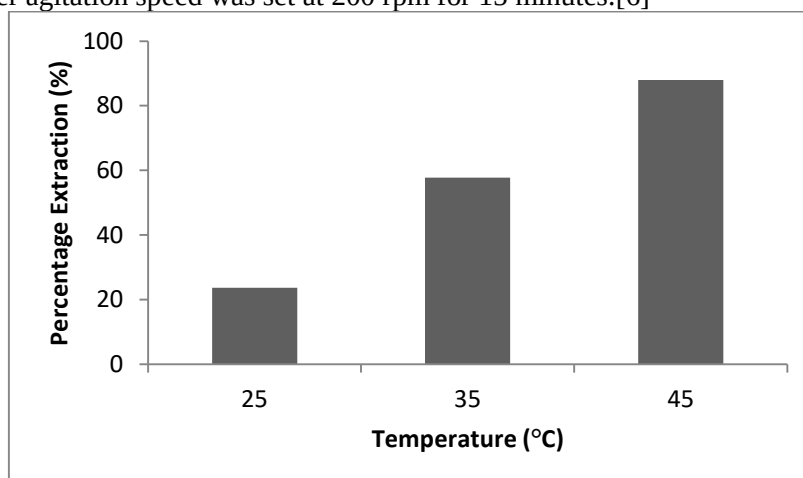


Figure 3.1: Temperature against percent extraction at pH 1, 200 rpm and 1:1 ratio of organic to aqueous.

Figure 3.1, shows the trend of increasing percentage of extraction (%E) as the temperature increases. The extraction at 25 °C showed the lowest %E of 23.63% in comparison with other temperatures condition. At temperature 35 °C, the %E of Au (III) increases to 57.73% while at 45 °C, the %E continues to rise to 88%. High temperature tends to reduce the viscosity and surface tension of organic phase subsequently increases the diffusion rate and mass transfer of solutes in the organic phase [7]. This condition is suitable for high viscous organic phase likes waste cooking oil. This explains why the higher temperature is able to obtain high percentage of extraction of Au (III). According to the chemical kinetics theory, higher temperature is conducive reaction process. Therefore, the extraction of gold is increasing with the raising of temperature [8]

3.2 Effect of Agitation Speed

The effect of agitation speed on percentage extraction is studied under constant optimum pH 1, temperature at 35 °C, and 1:1 ratio of organic to aqueous phase. The result is presented as in Figure 3.2.

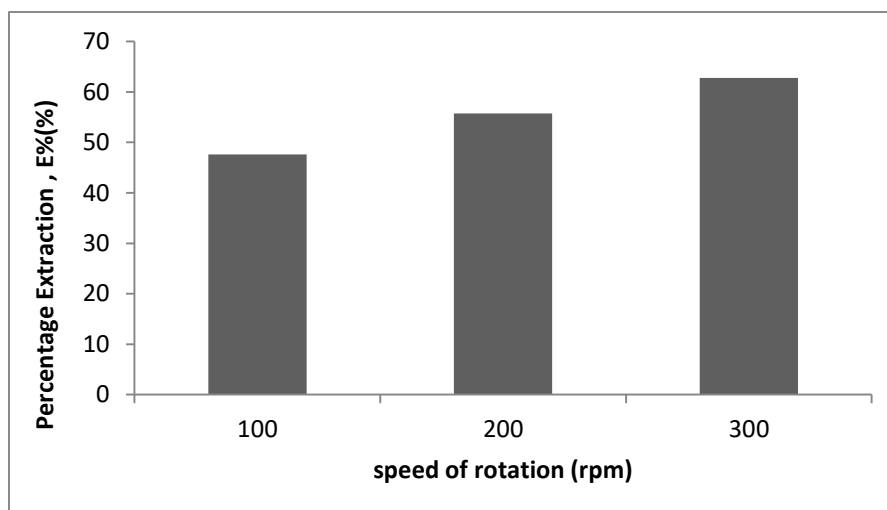


Figure 3.2: The percent of extraction at different speed of rotation

From the figure 3.2, it can be seen that in comparison with temperature, the agitation speed give less effect to %E of Au (III), however as agitation speed increases, the E% is also increases. It was found that the extraction at 100 rpm produces the lowest %E of 47.62%. With agitation speed at 200 rpm and 300 rpm, the %E of Au (III) increases to 55.73% and 62.79% respectively. These results can be explained by the fact that the increase of the agitation speed, improves the diffusion of Au (III) ions into the organic phase which is waste cooking oil [9]. An increase in level of agitation would increase interfacial area and mass transfer coefficient, k_{La} in the aqueous feed phase [10]. It is proved that by increasing speed of agitation from 100 to 300 rpm would result in increasing the rate of extraction. This also indicates that a higher shaking rate produces better mixing of the mixture and it is sufficient to ensure that all the binding sites are made readily available for Au (III) uptake.

3.3 Effect of Stripping

Stripping process is carried out to enhance the recovery of Au (III) which was not recovered during extraction method. The effect of stripping is investigated by using the same organic phase taken from the previous experiment on the effect of temperature to the percent of extraction. The organic phase from this study effect is chosen instead of from the effect of agitation speed is because of its more obvious trend. To avoid confusions, the samples are renamed as follows; organic phase at 25 °C (sample 1), 35 °C (sample 2) and 45 °C (sample 3).

The new aqueous phase used in this study is 1M of sulphuric acid, H_2SO_4 which acts as the stripping agent. The mixture is kept under optimum condition at pH of 1, temperature of 35 °C, 1:1 ratio of organic to aqueous phase. The result is presented in Table 3.1.

Table 3.1: Percentage stripping of Au (III) using 1M of H_2SO_4

Sample	Extracted amount of Au (III) (ppm)	Back extracted amount of Au (III) (ppm)	Percentage stripping (%)
1	61.638	31.048	50.37
2	71.835	21.252	29.58
3	72.879	18.683	25.64

Table 3.1 indicates the results of stripping process of Au (III) of the back extracted Au (III) from the organic phase. From the result, it can be seen that, sample 1 achieved the highest percentage of stripping efficiency (%S) of 50.37% compared than other samples. On the other hand, sample 2 and 3, showed 29.58% and 25.64% of %S respectively.

H_2SO_4 is a strong acid and capable to dissociate easily into hydrogen ion (H^+) in the stripping phase more than other mineral acids, hence H_2SO_4 contributes to higher stripping percentage of each sample [11].

3.4 FTIR Spectroscopy Analysis

The chemical properties examinations were investigated to study the existence of interactions in organic phase between D2EHPA and waste cooking oil using FTIR spectroscopy. The FTIR spectrum of waste cooking oil is shown in Figure 3.3. The C-H bond in waste cooking oil identified at 2922.16 cm^{-1} shows the presence of alkane functional group in oil. While the C=O bond exist at 1743.65 cm^{-1} shows the ester functional group.

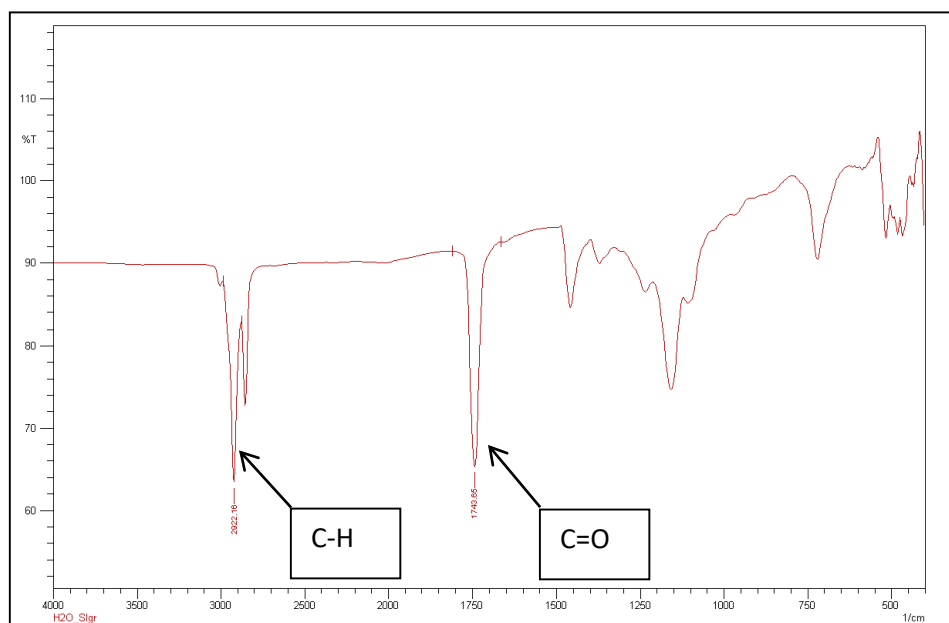


Figure 3.3: Infra-red spectrum of waste cooking oil

Figure 3.4 shows Infra-red spectrum of waste cooking oil with the addition of D2EHPA. The peak at 1033 cm^{-1} is identified as the stretching vibration of P-O-C and P-O-H bond after the addition of D2EHPA. At 2100 cm^{-1} , the spectrum shows the appearance of the peak of O-H stretching vibration [12]. The existence of O-H peak in D2EHPA shows the hydrogen bonds between each two D2EHPA molecules known as dimeric D2EHPA [13].

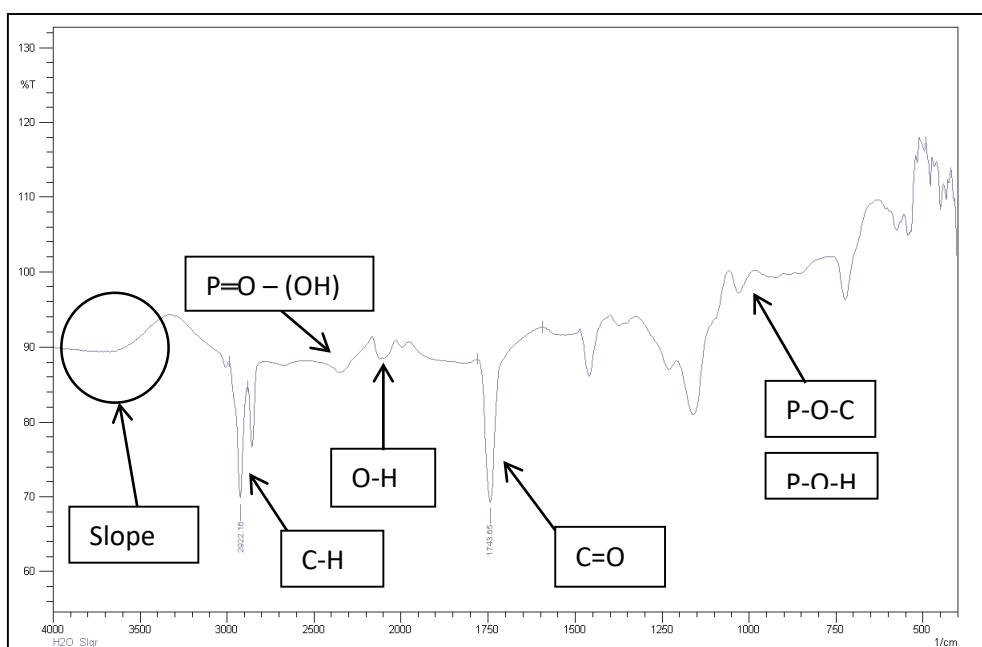


Figure 3.4: Infra-red spectrum of waste cooking oil with D2EHPA

The bands in the region $2300 - 2400\text{ cm}^{-1}$ are due to P=O-(OH) vibrations between intermolecular hydrogen bonding in the dimeric form [14]. A slope can be observed at 3300 cm^{-1} after the complexation

which describes the vibration of the free –OH group due to dissociation dimer to monomer. The peak position of C-H and C=O bond in both waste cooking oil with and without D2EHPA are identical. However, the peak with addition of D2EHPA appears to be shorter in length which equals to higher percentage of transmittance (%T). The %T indicates the effectiveness of the surface of a material in transmitting radiant energy. The higher the amount of light passes through, the larger the value of the transmittance and the higher energy been transmitted.

4. Conclusion

Studies on solvent extraction and stripping method on recovery of gold have been performed to investigate the best parameters and conditions in order to obtain the best performance as desired. The organophosphorus compounds, D2EHPA is an excellent metal ion extractant that contributes to solvent extraction process in this research. Since waste cooking oil is non-petroleum based, it has been chosen as the diluent in this study due to its environmental friendliness as well as low in cost. For the extraction process, the parameters of temperatures and agitation speeds were investigated to study their effects towards the %E of Au (III) recovery. The other parameters were fixed at 100 ppm initial concentration of Au (III), pH 1, 1:1 organic to aqueous ratio and 15 minutes of contact time during the mixing process. It can be concluded that the most suitable conditions for the single D2EHPA extraction system are temperature of 45 °C and agitation speed of 300 rpm. The maximum %E of Au (III) obtained at these conditions are 88% and 62.79% respectively. It is shown that, increasing extraction temperature would promote the mass transfer of Au (III) and also reduce the solvent viscosity and surface tension and hence, high %E of Au (III) were obtained. In this study, stripping process is able to enhance the recovery of Au (III) by back extraction of the metal using the same organic phases that were used in the extraction system by %S of 50.37% of sample 1, 29.58% of sample 2 and 25.64% of sample 3.

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