Competitiveness in removing copper, zinc and chromium trivalent in plating industrial effluent by using hydroxide precipitation versus sulphide precipitation

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Abstract: Plating industry wastewater is one of the processes that may contain various types of heavy metals. The most common heavy metals that mostly found are copper, nickel, cadmium, chromium, silver, zinc, and lead. In this study, three types of metals which are copper, zinc and chromium trivalent were removed using hydroxide precipitation and sulphide precipitation. Both performances of the method were observed and compared. A jar test was used to determine the optimum pH, coagulant dosage and precipitant dosage. The coagulant and precipitant used in this experiment was ferric chloride (FeCl₃), poly aluminium chloride (PAC), sodium hydroxide (hydroxide precipitant) and sodium sulphide (sulphide precipitation) respectively. In order to remove chromium trivalent, sodium metabisulfite was used to reduce chromium hexavalent to chromium trivalent. Based on the result, hydroxide precipitation can remove 86.61% of copper, 99.81% of zinc and 99.99% of chromium trivalent. While, for sulphide precipitation, 93.91% of copper, 99.37% of zinc and 99.99% of chromium trivalent were removed. Therefore, sulphide precipitation can remove better mixed heavy metals, thus it also produces less total suspended solid compared to hydroxide precipitation.

1. Introductions

Industrial effluent is any waste that generated from the manufacturing process such as electroplating, iron and steel industry, textile manufacturing and mining operation either in the form of liquid or wastewater, including the water treatment or any operation taking place at any industrial premises in compliance with the Industrial Effluent Regulations (IER) 2009. The metal plating industry process that produces an effluent that does not comply with the IER 2009 [1] standard will affect and harm human health as well as environment if does not treat it before it discharges. In accordance to Gunatilake, (2015) comprehensive review, it can be concluded that, in extreme cases, heavy metals can cause serious health effects to the human such as cancer, organ damage and also death [2]. It is because it can accumulate in the living organisms [3].

Heavy metals such that present in the wastewater can be a major environmental concern, thus can cause adverse health effect to the human if consumed in higher dosage. An excessive dosage of copper can lead to chronic effect such as Wilson disease, liver damage, and insomnia [4-5] as it only needed in trace amounts. Exceeding amount of zinc could affect several human body systemic systems such as...
neurology, respiratory and gastrointestinal system [6]. Chromium can cause problems to the human in excess dosage such as headache, diarrhea, nausea, vomiting and it is carcinogenic to humans [2]. Excessive chromium also causes eye irritation, skin irritation, respiratory tract, lung carcinoma in humans, and kidney dysfunction [7].

Hydroxide precipitation and sulphide precipitation are the most familiar [8-9], widely used precipitation and also an efficient method for the removal of heavy metal ions [3] from industrial effluent. Both of these methods however, have their own limitations in the treatment of mixed heavy metal effluents. For hydroxide precipitation, all metal hydroxides do not completely precipitate completely at a single pH because each type of metal hydroxide is favourable to precipitate at a certain pH range [8]. According to Ye et al., (2017) it also produces large volumes of relatively low density sludge that needed further treatment before disposal [8]. It is widely used because of its relative simplicity, easy to control pH and low cost [2-3].

Sulphide precipitation also constitutes an effective process for treating toxic heavy metal ions [3]. The advantages to compared with hydroxide precipitation are, the residual concentration of metals in the effluents is lower. The solubilities of the metal sulphide precipitation are substantially lower than hydroxide precipitation and it is not amphoteric [11]. Other than that, it also exhibits better thickening and it can achieve a high degree of metal removal over a wide pH range compared to hydroxide precipitation [3,8,11]. However, sulphide precipitation is costly and can emit hydrogen sulphide gas (H₂S) in acidic condition [3,11]. The purpose of this research was to identify the competitiveness of mixed heavy metals removal which is copper, zinc and chromium trivalent in industrial effluent by using hydroxide precipitation and sulphide precipitation.

2. Materials and Methods
2.1 Sample Wastewater
The raw effluent sample was obtained from the plating industry in Shah Alam. Approximately 15 liters of samples are needed for this study. The sample was stored in the container and kept in the water quality laboratory at room temperature to preserve the components of the sample. The sample was sent to wastewater characterization analysis.

2.2 Heavy Metals Concentration Measurement
Inductively Coupled Plasma Spectrometer (ICP-OES) Thermo Scientific iCAP 6000 series was used to trace the concentration of heavy metals in the effluent sample which is zinc, copper, and chromium trivalent. Heavy metals concentration before and after treatment is measured and compared. 10 mL of the sample was taken for the characterization of the element content.

2.3 pH Measurement
pH meter was used in this experiment was Knick 765 Laboratory pH Meter Calimatic. The pH meter must undergo a calibration to minimize any measurement uncertainty by ensuring the accuracy of the equipment. After calibrating, immerse the electrode and temperature probe into the sample and wait until the number displays show constantly.

2.4 Turbidity Measurement
ITS DM-TU Digimed Turbidity Meter was used for measuring the turbidity or cloudiness of the sample caused by the suspended solid particles. Calibrate the equipment before using it to ensure the accuracy of the equipment. A representative sample was collected and the sample was filled into a 10 mL sample vial. The sample cell was gently inverted and then inserted into the instrument cell. The result will be in unit NTU.

2.5 Chemical Oxygen Demand (COD) Measurement
Chemical oxygen demand (COD) was measured with HACH DR2800 Spectrophotometer and Orbeco Hellige TR 125 Reactor. Power on and preheated Orbeco Hellige TR 125 Reactor to 150°C. For sample
preparation, the vial was held at an angle of 45° and 2.00 mL of sample was added to the vial by using a pipette. For blank sample, hold the vial at an angle of 45° and 2.00 mL of deionized water was added to the vial by using a pipette. The vials were put in the preheated TR 125 Reactor and the lid was closed. Heated the vials for 2 hours. Next, power off the reactor and let the vials cool down to 120°C or less in the reactor for about 20 minutes. Inverted each vial several times while it’s still warm. Put the vials in a tube rack to cool at room temperature. By using HACH DR2800, start the Program 435 COD HR. Clean and insert the blank sample into the cell holder. Push ZERO and the display show 0 or 0.0 mg/L COD. Clean and inserted the prepared sample cell into the cell holder and push READ. The results show in mg/L COD. The concentration before and after treatment is measured and compared.

2.6 Total Suspended Solids Measurement
The weight of a dry filter paper was determined and filter the sample by using Whatman 1 filter paper. The residual was dried at 35°C – 40°C in an oven (MMM Medcenter Venticell Drying Oven). Then, cool the sample and by using ADAM Nimbus analytical balance, the new weight of the filter paper plus dried residue is then taken. Equation 1 shows the calculation of total suspended solids (APHA 2540 D Method).

\[
\text{Total suspended solid (mg/L)} = \frac{(A - B)}{V} \tag{1}
\]

Where:
- \(A\) = mass of filter paper + dried residue (mg)
- \(B\) = mass of filter paper (mg)
- \(V\) = total volume of the sample (L)

2.7 Jar Test
The type of jar tester used during this process is the Stuart SW6 Flocculator jar tester. The sample was rapidly mixed at 150 rpm. The chemical substances used were 1 M ferric chloride (FeCl₃), 5% poly aluminium chloride (PAC), 1 M sodium hydroxide (NaOH), 1 M sodium sulfide (Na₂S) and 0.05 M sulphuric acid (H₂SO₄).

2.7.1 Conversion of chromium hexavalent (Cr⁶⁺) to chromium trivalent (Cr³⁺).
The Cr⁶⁺ was converted to Cr³⁺ by using sodium metabisulfite [12-13]. Sulphuric acid (H₂SO₄) and sodium hydroxide (NaOH) were used in pH adjustments. The condition of the sample is at the pH of 2 [13] in order to optimize the condition of Cr⁶⁺ reduction, and the ORP value is 280 mV. Knick 765 Laboratory pH Meter Calimatic was used to set the pH and ORP value. It was mixed by using flocculator at 150 rpm.

2.7.2 Determination of Optimum pH.
Five units of 500 mL beaker were used in this experiment. Every beaker was set up with 300 mL of sample for different pH setting. The pH of the samples is adjusting to a different value which is 6.5, 7.5, 8.5, 9.5, and 10.5 for hydroxide precipitation and 8.5, 9.0, 9.5, 10.0, and 10.5 for sulphide precipitation by using H₂SO₄ or NaOH. Turn on the flocculator at 150 rpm and add the same dosage of FeCl₃ (0.5mL) to each beaker. The samples will be mixed for 3 minutes. Add 0.5 mL of PAC and continue to mix it at the same speed. Turn off the mixer and allow the flocs to settle for about 30 minutes [14]. Pipet the sample and put it into 10 mL vial. Lastly, turbidity was measured by using a turbidity meter, and the pH value with the lowest turbidity value will be selected as the optimum pH [8].

2.7.3 Determination of optimum coagulant dosage.
Five units of beakers of the sample were prepared and were adjusted to the optimum pH, which is pH 10.5 (hydroxide precipitation) and pH 10.0 (sulphide precipitation) by adding H₂SO₄ or NaOH. Rapid
mixed each beaker and different doses of FeCl$_3$ were added to each beaker, 0.2 mL, 0.4 mL, 0.6 mL, 0.8 mL, and 1.0 mL respectively for each method. Mixed the sample for 3 minutes and added 0.5 mL of PAC to each beaker. Continue mixing the sample and turn off the mixer and allow the flocs to settle for 30 minutes for sedimentation [14]. By using a pipette, take the sample and placed it into 10 mL vial in order to measure the turbidity. Coagulant dosage that contributes to the lowest turbidity value is selected as the optimum coagulant dosage [8].

For the determination of PAC dosage, the experiment was repeated by using five units of beaker of the sample and adjusted to the optimum pH and optimum dosage of FeCl$_3$ which is 0.8 mL (hydroxide precipitation) and 0.2 mL (sulphide precipitation). Rapid mixed each beaker and added different doses of PAC which is at 0.2 mL, 0.4 mL, 0.6 mL, 0.8 mL, and 1.0 mL respectively.

2.7.4 Determination of optimum precipitant dosage.
Five units of beakers were prepared with an adjusted optimum pH. Rapid mixed each beaker and added optimum dosage of FeCl$_3$ (0.8 mL) and mixed the sample for 3 minutes. Then, added 1.0 mL of PAC to each beaker and continue to mix. After that, different doses of NaOH that act as precipitant was added to each beaker, 0.2 mL, 0.4 mL, 0.6 mL, 0.8 mL, and 1.0 mL respectively. Continue mixing the sample and allow the flocs to settle for 30 minutes. By using a pipette, take the sample and placed it into 10 mL vial in order to measure the turbidity. The lowest turbidity was chosen as an optimum precipitant dosage. Repeat the step for sulphide precipitation by using Na$_2$S.

2.7.5 Hydroxide precipitation and sulphide precipitation.
Turn on the flocculator and set the pH to pH 10.5 for hydroxide precipitation. Then, added the optimum dosage of FeCl$_3$ (0.8 mL) and PAC (1.0 mL) into the sample. After dosing the sample, set the speed of stirrer to 150 rpm and let it stir for 3 minutes [8]. After that, added precipitant (NaOH) [10,15-16] into the sample and continue stir for 5 minutes. Continue to mix and add PAC. Turn off the mixer and allow the flocs to settle for 30 minutes [14]. The sample was filtered by using filter paper. Lastly, the residual concentration of Cu, Zn, and Cr$^{3+}$ were measured and the total suspended solid produced was observed. Both of chemical precipitation was triplicated. The step was repeated by using different chemical precipitants which is Na$_2$S for sulphide precipitation. The optimum pH (pH 10.0), FeCl$_3$ (0.2 mL) and PAC (0.8 mL) was set. Performance results from both precipitants was compared.

2.8 Statistical Analysis
The comparison between hydroxide precipitation and sulphide precipitation was analysed by using two-factor replication in ANOVA analysis in Excel Software.

3. Results and Discussion
The characterization of the effluent from the plating industry is carried out according to Standard B of Industrial Effluent or Mixed Effluent, Environmental Quality (Industrial Effluent) Regulations 2009. From the result obtained, the sample is in the basic condition which is in the range of pH 11.76 to pH 11.78 and it is in compliance with the Standard B which is in pH 5.5 – 9.0. The initial turbidity value of the sample is 6.55 NTU. The optimum condition for each treatment is shown in Table 1. The initial concentration of heavy metals and chemical oxygen demand is shown in Table 2.

| Table 1. The optimum condition for hydroxide precipitation and sulphide precipitation |
|-----------------------------------------------|--------------|-----------------|
| Optimum pH                                   | 10.5         | 10.0            |
| Optimum FeCl$_3$                             | 0.8 mL       | 0.2 mL          |
| Optimum PAC                                  | 1.0 mL       | 0.8 mL          |
| Optimum precipitant (NaOH or Na$_2$S)         | 1.0 mL (NaOH) | 0.8 mL (Na$_2$S) |
Table 2. Initial concentration of effluent from plating industry

| Parameter               | Initial concentration (mg/L) |
|-------------------------|------------------------------|
| Copper                  | 0.0017                       |
| Zinc                    | 1.0613                       |
| Chromium trivalent      | 22.0657                      |
| Chemical oxygen demand  | 159                          |

3.1 Effect of pH

By varying the pH during pH adjustment process between pH 6.5 until pH 10.5 with an interval of 1.0 for hydroxide precipitation and pH 8.5 until pH 10.5 with an interval of 0.5 for sulphide precipitation and at a constant dosage of FeCl₃ and PAC at 0.5 mL, the graph of turbidity removal against pH is plotted. Then, the optimum pH of each treatment was obtained by choosing the highest turbidity removal. Figure 1 shows the effect of pH on the turbidity removal in the jar test process.

![Figure 1. Effect of pH of wastewater on turbidity removal](image)

Overall, sulphide precipitation can remove better in term of turbidity than hydroxide precipitation as it has an ion of the oppositely charged to the colloid [17]. Based on the graph, it shows that turbidity removal increasing with increasing pH for each treatment. According to previous research [18], by increasing the pH value (alkaline condition) will reduce the turbidity value. At higher pH value, there is a reduction of turbidity because of the tendency toward sedimentation due to the opposite charge of an ion of the colloid. However, for sulphide precipitation, there is slightly decreased in turbidity removal at pH 10.5, 96.64%. It is due to some stable colloid cannot neutralize as stable colloid possess a negative charge that repels other colloid particles colliding with each other. Based on the result obtained, the optimum pH that was chosen for hydroxide precipitation and sulphide precipitation is at pH 10.5 and pH 10.0 respectively as it has the lowest turbidity value.

3.2 Effect of coagulant dosage

Figure 2 shows the effect of FeCl₃ dosage on the plating effluent on the removal of turbidity in the coagulation process. As shown in Figure 2, sulphide precipitation can remove better turbidity than hydroxide precipitation. For sulphide precipitation, as coagulant increased from 0.2 mL to 0.8 mL, the removal decreased. But, at 0.1 mL of FeCl₃, the turbidity removal was significantly decreased about 27.63% from 87.94% (0.8 mL) to 60.31%. It is due to the insufficient or too much coagulant added to the wastewater in which can affect the turbidity of the water and the size of the coagulated particles.
could also be affected by pH, which in turn determines the density, tendency and the rate of settling of the flocculated slime [17]. It is apparent that at 0.2 mL of FeCl$_3$ is chosen as optimum coagulant dosage as it produces the lowest value of turbidity. It is in line with the previous research stated that lower rates of using FeCl$_3$ can show an appropriate result in turbidity removal.

FIGURE 2. Effect of FeCl$_3$ on turbidity removal at different doses

Meanwhile, the result obtains for hydroxide precipitation is contrary to sulphide precipitation. From the graph above, the trend for hydroxide precipitation shows the increment of FeCl$_3$ dosage at a certain amount can enhance the removal of turbidity inside the wastewater. However, as the coagulant dosage was low, the efficiency of turbidity removal decreased as the pH of wastewater still at alkaline condition due to the hydroxide group present in wastewater. But, at 1.0 mL of FeCl$_3$, the turbidity removal was minimally decreased at 49.01%. By using too much FeCl$_3$, it can cause a reversal in change by forcing the suspended solids to destabilize and complicating the entire treatment process. The optimum FeCl$_3$ for hydroxide precipitation is at 0.8 mL as it removes the highest percentage of turbidity.

Figure 3 shows the experimental data on the effect of PAC to the turbidity in the wastewater. A comparison of the two types of treatment obtained in Figure 3 reveals that sulphide precipitation has the highest percentage in removing turbidity than hydroxide precipitation. By adding PAC to the wastewater by coagulation process, it can contribute to the decreasing value of turbidity in the wastewater. For sulphide precipitation, the turbidity removal increased as the dosage of PAC increasing. This is because PAC is presumed to be more advantageous than conventional coagulants due to their higher removal of particulate and organic matter. It is also less alkalinity consumption and less production of suspended solid [19]. However, at 1.0 mL of PAC, the turbidity removal slightly decreased by 0.3%. With regard to hydroxide precipitation, it shows the increasing value of turbidity removal at 0.2 mL (64.27%) and 0.4 mL (68.24%) of PAC, but it decreased to 67.33% at 0.6 mL and increased again at 0.8 mL (72.98%) and 1.0 mL (84.58%). Therefore, 1.0 mL of PAC was chosen as the optimum value of PAC for hydroxide precipitation. This finding is contrary to the previous studies which stated that PAC required lower doses to react [20].
3.3 Effect of precipitant dosage

Overall, sulphide precipitation has the highest percentage of turbidity removal compared to hydroxide precipitation. The graph in Figure 4 shows that there gradual decreased in the percentage of turbidity removal from 0.2 mL to 0.8 mL and then slightly rise at 1.0 mL (91.45%). Meanwhile, for hydroxide precipitation, the graph shows a steady increase in the percentage of turbidity removal from 0.2 mL to 1.0 mL of NaOH. Thus, the suitable dosage of chemical precipitant for sulphide precipitation is 0.2 mL of Na$_2$S and 1.0 mL of NaOH for hydroxide precipitation. Therefore, it is important to determine the suitable dosage of precipitant to ensure its effectiveness in removing heavy metals.

3.5 Comparison performance between hydroxide precipitation and sulphide precipitation

All the optimum condition was set up to identify the performance of hydroxide precipitation and sulphide precipitation in removing heavy metal (Cu, Zn, Cr$^{3+}$). Final turbidity value, total suspended solid produces and the average heavy metals removal (Cu, Zn, Cr$^{3+}$) is plotted against types of chemical precipitation used. Figure 5 reveals the effect of hydroxide precipitation and sulphide precipitation on the production of total suspended solids and turbidity removal. Based on Figure 5, sulphide precipitation is more efficient than hydroxide precipitation in the production of total suspended solid which is 300
mg/L. This means that sulphide precipitation produces less total suspended solid compared to hydroxide precipitation. It is consistent with the literature, which is hydroxide precipitation tends to produce more total suspended solid than sulphide precipitation [3,8,11]. Hence, it also has better thickening and dewatering properties than the respective metal hydroxide of total suspended solids. Furthermore, sulphide precipitation also has the highest turbidity removal than hydroxide precipitation at 95.27%.

**Figure 5.** Effect hydroxide precipitation and sulphide precipitation on turbidity removal and total suspended solid

Sulphide precipitation produces a higher concentration of COD than hydroxide precipitation at 189 mg/L, but it is still not exceeding the acceptable condition for the discharge of mixed effluent Standard B which is 200 mg/L. It is due to the lower usage of FeCl₃ in sulphide precipitation which is 0.2 mL than hydroxide precipitation 0.8 mL as the higher dosage of FeCl₃ can remove better COD [21]. Higher COD level in water indicates that in the certain wastewater there is a greater amount of oxidizable organic material in which it can lower the dissolved levels of oxygen.

Based on the result shown in Figure 6, copper concentration is reduced about 88.24% for hydroxide precipitation, while about 94.8% of copper is removed via sulphide precipitation. For the removal of zinc, about 99.81% are removed via hydroxide precipitation and 99.37% removed by sulphide precipitation. Based on the result obtains, the concentration of chromium is reduced about 99.99% by both methods which are hydroxide precipitation and sulphide precipitation. Although it has the same concentration, sulphide precipitation is more efficient as it uses lower coagulant dosage compared to hydroxide precipitation. These results are in accord with recent studies indicating that sulphide precipitation can remove a high degree of metals compared to hydroxide precipitation due to the lower solubility of metal sulphide over a wide pH range precipitation [3,8]. All of the heavy metals concentration is complying with the Standard B of acceptable conditions for the discharge of industrial effluent or mixed effluent after the treatment, especially for chromium.
3.6 Two-factor ANOVA analysis on the removal of mixed heavy metals via hydroxide precipitation and sulphide precipitation

Two-factor ANOVA analysis was performed to analyse the comparative performance between hydroxide precipitation and sulphide precipitation in removing Cu, Zn and Cr\(^{3+}\) from plating wastewater. The two-factor with replication ANOVA analysis was conducted based on the experiment of chemical precipitation by using the Excel Software. The ANOVA analysis for each chemical precipitation was presented in Table 3 and Table 4.

### Table 3. Descriptive analysis for the heavy metal removal

|                  | Cu     | Zn     | Cr\(^{3+}\) |
|------------------|--------|--------|-------------|
| Hydroxide precipitation | 86.61  | 99.81  | 99.99       |
| Sulphide precipitation   | 93.91  | 99.37  | 99.99       |

### Table 4. Two-factor ANOVA analysis

| Source of variation | df | F      | p-value | F crit |
|---------------------|----|--------|---------|--------|
| Sample              | 1  | 2.90508| 0.11403 | 4.74723|
| Columns             | 2  | 22.38736 | 0.00009 | 3.88529|
| Interaction         | 2  | 3.49467 | 0.06368 | 3.88529|

In accordance with Table 4, the p-value for the chemical precipitation is 0.11403, which is p > 0.05, hydroxide precipitation and sulphide precipitation does not affect the removal of Cu, Zn and Cr\(^{3+}\). In other words, there is no significant difference when considering hydroxide precipitation and sulphide precipitation by itself. As shown in Table 3, the average between two types of chemical precipitation in removing heavy metals is close to each other which is 95.47 % for hydroxide precipitation and 97.76% for sulphide precipitation. There is a significant difference when only considering the different type of heavy metals removal which is 0.00009 (p < 0.05). However, in terms of interactions, there is no
significant difference when considering hydroxide precipitation and sulphide precipitation and its relationship to the type of heavy metals removal.

4. Conclusion
The comparative performance between hydroxide precipitation and sulphide precipitation through the coagulation process by using jar test for removal of Cu, Zn and Cr\(^{3+}\) was investigated. The optimum pH by both of the chemical precipitation was studied on the effect of turbidity removal to the wastewater. It has shown that the optimum pH for hydroxide precipitation and sulphide precipitation are pH 10.5 and pH 10.0 respectively. Meanwhile, there are two types of coagulant used in this experiment which is FeCl\(_3\) and PAC. The optimum FeCl\(_3\) for hydroxide and sulphide precipitation was 0.8 mL and 0.2 mL respectively. The optimum dosage of PAC is 1.0 mL for hydroxide precipitation and 0.8 mL for sulphide precipitation. Furthermore, the optimum dosage of NaOH used was 1.0 mL, while the optimum dosage of Na\(_2\)S was 0.2 mL.

It can be concluded that sulphide precipitation can remove heavy metals more efficient than hydroxide precipitation. For sulphide precipitation, 94.81% of copper is removed while 88.24% of copper is removed by using hydroxide precipitation. For zinc, about 99.37% is removed via sulphide precipitation and about 99.81% is removed via hydroxide precipitation. Sulphide precipitation and hydroxide precipitation can remove 99.99% of chromium respectively. It is in line with the previous study stated that sulphide precipitation can remove more mixed heavy metals, thus, it also produces less total suspended solid compared to hydroxide precipitation. Sulphide precipitation produces 300 mg/L of total suspended solid compared to hydroxide precipitation, which is 400 mg/L. In terms of chemical oxygen demand (COD), there has shown the decreasing value from the initial value which is 156 mg/L for hydroxide precipitation. While for sulphide precipitation, there shows an increasing value of COD which is 189 mg/L from the initial value which is 159 mg/L, but it is does still not exceed the acceptable value in Standard B. Though, the objective is achieved.

For the next research, it is recommended to use heavy metals removal as an indicator to choose the best optimum value instead of choosing the lowest turbidity value. It is also recommended to use a flocculant in the coagulation-flocculation process to promote the larger flocs formation.

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