CHEM PUBLISH
Chempublish Journal
Journal Homepage: online-journal.unja.ac.id/chp
Department of Chemistry
Faculty of Science and Technology
Universitas Jambi
ISSN (Online): ISSN 2503-4588
No. 23/E/KPT/2019

Article

Study on The Ability of H_2O_2 and The Effective Concentration of $CuSO_4$ in Degrading Cyanide

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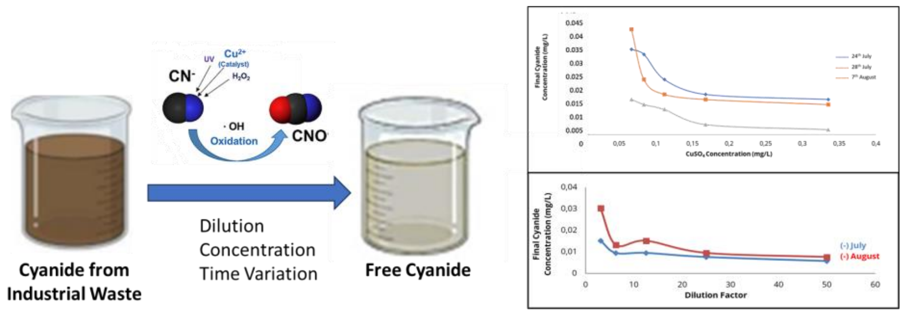
Abstract

PT. Aneka Tambang conducts gold processing using NaCN reagents, then from the gold processing process produces waste containing cyanide which can cause damage to the environment. One method used to reduce cyanide concentrations is the Degussa method. The Degussa method is a method to degrade free cyanide in waste by using H_2O_2 and Cu^{2+} ions as a catalyst source so that it becomes a harmless cyanide compound (cyanate ion). The purpose of the study was to determine the ability of hydrogen peroxide and determine the effective concentration of copper sulfate in degrading cyanide in the Degussa process and determine the level of stability of hydrogen peroxide oxidizers in the open air. Sample testing was carried out by varying the H_2O_2 retrieval time, variations in H_2O_2 dilution factor and copper sulfate concentration variations, then the sample was stirred with a jartes stirrer for 15 minutes. Cyanide is determined by spectrophotometer, the absorbance obtained is fed into the linear regression equation so that the final free cyanide concentration is obtained. From the experiment, it can be concluded that the concentration of cyanide degradation is effectively obtained when hydrogen peroxide and copper sulfate that have been mixed in the mixing tank are right out of use.

Keywords: *degradation, degussa method, gold processing, cyanide*

Graphical Abstract



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DOI: 10.22437/chp.v7i2.30438
Received 15 December 2023; **Accepted** 31 December 2023; **Available online** 31 December 2023

Introduction

PT. Aneka Tambang Tbk, is one of the State-Owned Enterprises (BUMN) under the Ministry of Mines and Energy. Currently PT. Antam has eight work units, one of which is the Pongkor Gold Mining Business Unit. The location of gold ore mining and processing is located in Sorongan Bantar Karet Village, Nanggung District, Bogor Regency, West Java.the mining power area is in the Du-893 exploration area of West Java [1].

Cyanide is a chemical used in the gold processing process, cyanide contains the risk of pollution and environmental damage resulting in a decrease in environmental quality [2], based on the Decree of the Coordinating State Minister for the Environment No. 202 of 2004 concerning effluent waste for industrial activities (wastewater quality standards for gold and / or copper ore processing activities), it has been determined that the concentration of cyanide discharged into the environment must be smaller than the environmental threshold i.e. 0.5ppm [3]. The initial concentration of cyanide in the tailings dam samples and seepage samples was 5.3 ppm (results of field checks) This is still above the permissible threshold [4], cyanide poisoning can have fatal consequences for human health and can even cause disability and loss of life in a person, the spread of cyanide in the environment will threaten environmental sustainability and the survival of living things in it [5].

One of the methods used at PT. Antam to reduce the concentration of cyanide that will be discharged into the environment, namely chemical degradation. Chemical degradation is considered effective as long as the right chemical sorting and concentration is carried out so as not to produce other harmful compounds [6]. The cyanide degradation method used is the Degussa method. The Degussa method is a method to degrade free cyanide in waste by using H_2O_2 and Cu^{2+} ions as a catalyst source so that it becomes a harmless cyanide compound (cyanate ion) [7].

The advantage of using the Degussa method is the ability of hydrogen peroxide to not only oxidize cyanide ions, but also oxidize sulfide, thio cyanate and other metal ions at low oxidation conditions [8]. The purpose of this study is to determine the ability of hydrogen peroxide and determine the effective concentration of copper sulfate in degrading cyanide in the Degussa process and determine the level of stability of hydrogen peroxide oxidizers in the open air.

Sample testing is carried out by varying the H_2O_2 sampling time, variations in H_2O_2 dilution factor and variations in copper sulfate concentration, then the sample is stirred with a jartest stirrer for 15 minutes. Cyanide was determined using a spectrophotometer. The absorbance obtained is fed into the linear regression equation so that the final free cyanide concentration is obtained [[9]]. From the study, it was found that the concentration of cyanide degradation was effectively obtained when hydrogen peroxide in its handling was not in contact with open air and the use of hydrogen peroxide and copper sulfate that had been mixed in the mixing tank was right out of use.

Experimental Section

Materials and Equipments

Gold is a heavy metal with a characteristic yellow color, in powder form its reddish-brown color, gold metal melts at 1064.8°C. Generally, gold is found as a metal in the sand layer or soil containing gold mixed or as an alloy of metal with silver, and usually contains also iron, bismuth and platinum, gold is also found in the soil (sediment) of rivers that pass-through rocks containing gold. The ore found in pongkor gold mining is silica type seeds, silica type seeds are usually white to gray, and are not reducing or oxidizing.

HCN gas will form at a low pH (acidic), while at high pH cyanide ions will form. HCN is highly toxic, so alkaline conditions or in high pH are needed to form cyanide ions. The cyanide acid produced has very volatile properties with Ka 6×10^{-6}, the toxic nature of cyanide is a concern in analyzing industrial wastewater.

Based on government regulation number (Government Regulation number 82 of 2001), water pollution is identified as a decrease in water quality to a certain level which causes water to not function in accordance with its designation.

The materials used in the study were hydrogen peroxide ≥ 50%, copper sulfate 76%, sodium hydroxide 0.2 N, pyridine barbiturates, bufer acetate 25%, chloramine trihydrate 1% (Sigma-Aldrich), sodium cyanide and distilled water. The equipment used is a UV-Visible Spectrophotometer (Shimadzu UV-1700), flocculator, measuring flask (50,100) mL, volumetric pipette (1,10,100) mL, analytical balance, 10mL measuring pipette, watch glass, 1000mL trophy glass, tissue, bulb, mask and gloves

Method

The degussa method is one method to degrade free cyanide into products with concentrations below the environmental threshold of less than 0.5 ppm using hydrogen peroxide and copper sulfate catalysts so that they are not harmful to the environment. The experimental method consists of several stages, namely sampling, preparation, sample testing and data processing.

Sampling was carried out by taking samples of waste from tailings dams and absorption water from seepage tanks, then each sample was spiked with CN- 1.59%. Sample testing was carried out by determining cyanide levels in the initial sample and samples that had gone through the waste treatment process through variations in the exposure time of hydrogen peroxide to open air, variations in the concentration of adding copper sulfate and variations in dilution of hydrogen peroxide on the ability to degrade cyanide, then the sample was stirred with a jartest stirrer for 15 minutes.

Pipetted as much as 100mL hydrogen peroxide with a purity of ≥ 50% then each put into a 100mL beaker glass and a winkler bottle, for beaker glass left and for winkler bottles immediately closed. Prepared 6 pieces of 1000mL beaker glass containing 1000mL of seepage samples that have been spiked with CN^-. Within 0 minutes the seepage sample was added 0.33 grams of H_2O_2 from the beaker glass and then added CuSO4 67mg / L then stirred with a flocculator for 15 minutes, after that the seepage sample was ready to determine the siandanya level. Furthermore, this action is carried out consecutively with a time span of H2O2 collection, which is 15; 30; 60; 120 minutes. While in the 120th minute an addition of H_2O_2 as much as 0.33 grams was carried out from the winkler bottle, as well as the same thing was done in samples from the tailings dam.

Measurement is carried out using a spectrophotometer, an optical spectrophotometer is an instrument with an optical system that is able to produce the distribution or (dispersion) of incoming electromagnetic radiation and can be measured the quantity of radiation transmitted or an intensity function. The basic principle of a spectrophotometer, the amount of light absorbed by a medium depends on the concentration, type of media and length of media through which the light passes. In general, spectrophotometers consist of six main components, namely light sources, monochromators, sample containers, detectors, ampliferes and data viewers.

Cyanide is determined using a spectrophotometer at λ: 578nm, the absorbance obtained is entered in the linear regression equation so that the final cyanide concentration is obtained [10].

Result and Discussion

Based on the results of research and data processing, the final CN- concentration results were obtained after undergoing the waste treatment process on the seepage example and tailings dam waste by UV-Vis spectrophotometer as follows.

Hydrogen peroxide oxidizer stability testing with time variation on seepage samples and dam tailings samples

Determination of the stability of the H_2O_2 oxidizer is carried out to determine the stability of the H_2O_2 oxidizer before use based on variations in contact time with free air. Test result data can be seen in table 1 and figure 1 for seapage samples. While in table 2 and figure 2 for tailings dam samples.

The longer hydrogen peroxide is in contact with open air, the less hydrogen peroxide is able to degrade cyanide [11]. Therefore, the H_2O_2 to be used should be conditioned so that it does not come into direct contact with air, in the H_2O_2 field contained in the mixing tank there is a gap that becomes a direct contact medium of hydrogen peroxide with air, this can be overcome by closing it. H_2O_2 has the property of very easily evaporating into oxygen and water.

$H_2O_2 \rightarrow H_2O + O_2$ .... [1]

In open conditions it will decompose into water and oxygen. Starting at the 60th minute the stability of the H_2O_2 oxidizer begins to decrease, this can be seen from the increase in cyanide concentration set until the 120th minute. In the H_2O_2 example in contact with open air, while at the 120th minute on the Winkler bottle the stability of the H_2O_2 oxidizer was stable.

Table 1. Oxidizer stability with time variation.

Sample	Time (Minutes)	H_2O_2 (mg/L)	$CuSO_4$ (mg/L)	Initial pH	CN^- (mg/L)
July	0	330	0.067	6.9	0.0076
	15	330	0.067	7.0	0.0076
	30	330	0.067	6.9	0.0076
	60	330	0.067	6.9	0.0095
	120	330	0.067	6.9	0.0095
August	120	330	0.067	6.9	0.0076
	0	330	0.067	6.9	0.0057
	15	330	0.067	6.9	0.0057
	30	330	0.067	6.9	0.0057
	60	330	0.067	7.0	0.0076
	120	330	0.067	7.0	0.0095
	120	330	0.067	7.0	0.0057

Description: Samples No. 1,2,3,4 and 5 are the addition of H2O2 from the glass beker while sample No. 6 is the addition of H2O2 from the winkler bottle

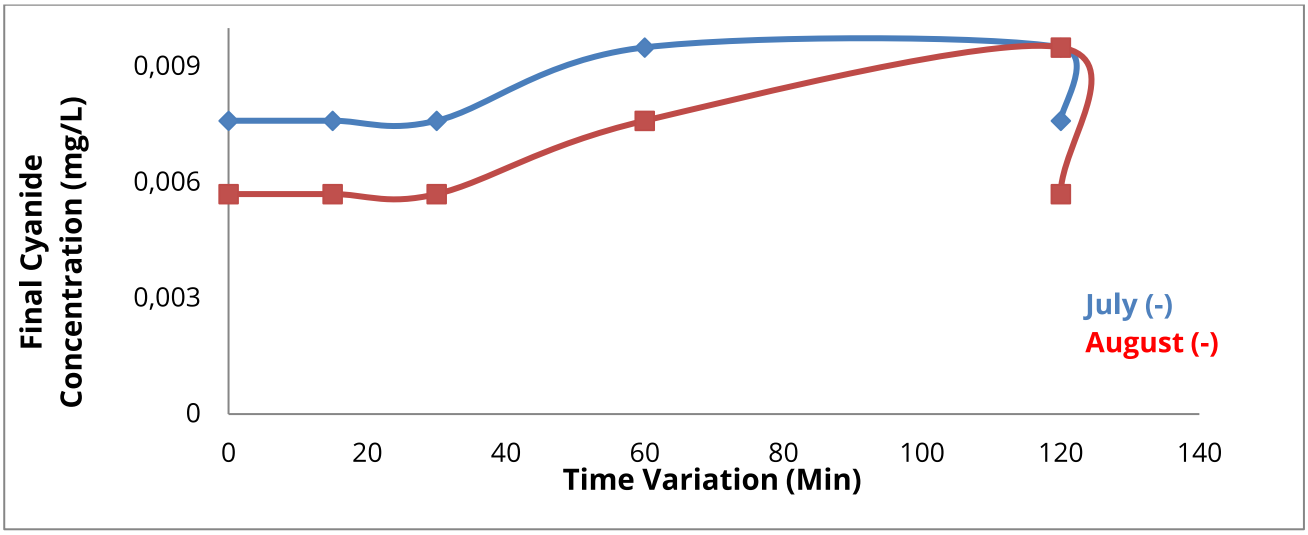

Figure 1. Graph of H_2O_2 oxidizer stability determination with time variation in seapage samples.

Table 2. Results of determination of H_2O_2 oxidizer stability with time variations in samples from dam tailings

Time (Minutes)	H_2O_2 (mg/L)	$CuSO_4$ (mg/L)	Initial pH	Initial CN^- Concentration (mg/L)	Final CN^- Concentration (mg/L)
0	330	0.067	8.8	5.3	0.051
15	330	0.067	8.8	5.3	0.049
30	330	0.067	8.8	5.3	0.055
60	330	0.067	8.8	5.3	0.059
120	330	0.067	8.8	5.3	0.070
120	330	0.067	8.8	5.3	0.040

Description: Beaker Glass No 1,2,3,4,5 for H2O2 in beaker glass, no 6 H2O2 on winkler bottle

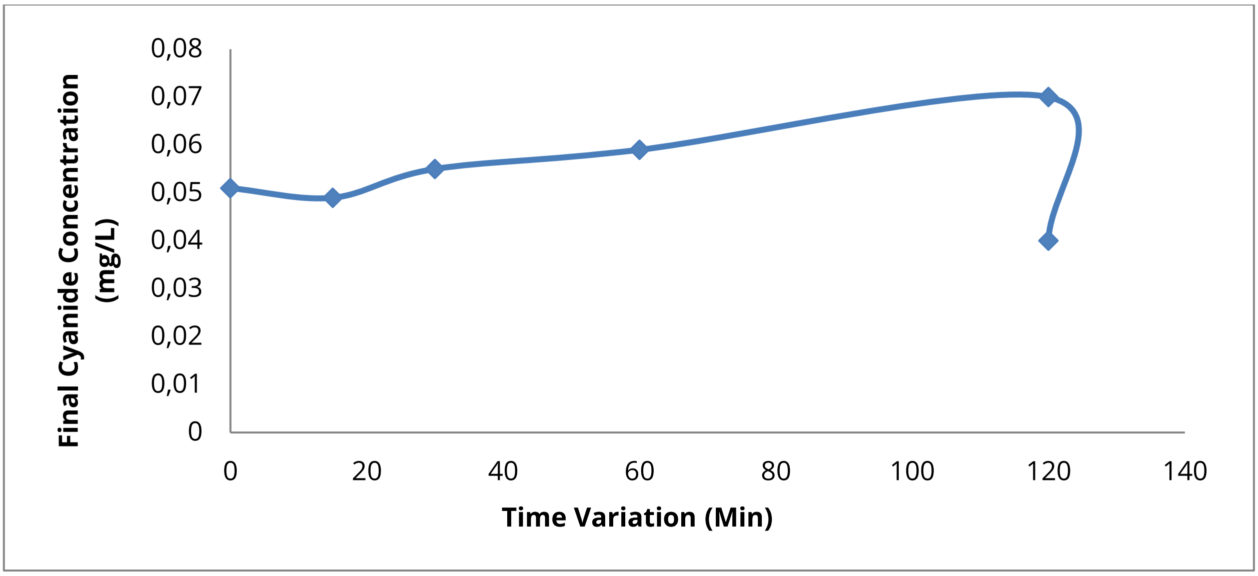

Figure 2. Oxidator stability setting graphic H_2O_2 with time variations in samples from tailing dam.

This can be seen in the experiment conducted on July 29, which is up to a 30-minute time variation in the final cyanide concentration after going through the waste treatment process is:

CN Concentration = 0.379 x Ab x Fp ....[2]

At the 60-minute time variation there was an eventual increase in cyanide concentrations become:

CN Concentration= 0.379 x Abs x Fp ....[3]

While in experiments carried out with a time variation of 120 minutes where hydrogen peroxide is tightly closed and not Contact with the open air can be seen that there is a decrease in cyanide concentration to:

CN concentration= 0.379 x Abs x Fp .... [4]

pH measurements are carried out using pH meters because the pH range must be maintained, the optimal pH of hydrogen peroxide in degrading cyanide into cyanate ions is in the pH range of 7-11 [[12]], if the pH is too acidic, HCN gas will form while if in alkaline pH conditions it will not form cyanate ions [[13]].

The absorbance used in measurements using a UV-Visible spectrophotometer is at a wavelength of 578.0 nm with a greenish-yellow absorption color and a violet observed color, and the blank absorbance in the spectrophotometer used as a correction factor is 0.000.

Cyanide levels in seepage samples and samples from dam tailings with H_2O_2 dilution

Four series of 50% ≥ H_2O_2 dilution with 2x dilution factor; 4x ; 8x ; 16x, then weighed H_2O_2 0.33 grams H_2O_2 ≥ 50% (Fp 1x), 0.66 grams (Fp 2x), 1.32 grams (Fp 4x), 2.64 grams (FP 16x) then each inserted into a 1000mL beaker glass containing 1000mL seepage samples. Each sample was added 1mL $CuSO_4$ with concentration 67 mg / L and then stirred with a flocculator for 15 min, the sample was ready to determine the cyanide concentration, carried out the same steps as the sample from the Tailings Dam.

From the research conducted, it can be concluded that the greater the dilution factor, the less the ability of hydrogen peroxide to degrade cyanide. Test result data can be seen in table 3 and figure 3 for seepage samples, while in table 4 and Figure 4 for tailings dam samples. The final cyanide concentration was obtained after going through the waste treatment process with variations in hydrogen dilution (1,2,4,8,16) times the dilution gave the difference results that insignificant, dilution can be done as long as the number of moles is the same and the final cyanide concentration obtained is still below the environmental threshold [14], in the field before H_2O_2 is added to the tailings sample dam in the mixing tank, H_2O put first into the mixing tank, dilution is done by inserting seepage water containing cyanide with a small concentration, this causes H_2O_2 before degrading cyanide in the tailings dam, the first degrades the cyanide contained in the seepage water used in dilution [[15]], if the cyanide concentration in the waste is high it will have an effect, but if the concentration of waste is small this has no effect.

Table 3. Cynaide levels in seepage samples with dilution factors.

Date	Sampling	H_2O_2 Concentration (%)	$CuSO_4$ Concentration (mg/L)	Initial pH	Initial CN^- Concentration (mg/L)	Final CN^- Concentration (mg/L)
July	1	50	0.067	6.9	5.30	0.0057
	2	25	0.067	6.9	5.30	0.0076
	4	12,5	0.067	6.9	5.30	0.0095
	8	6,25	0.067	6.9	5.30	0.0095
	16	3,12	0.067	6.9	5.30	0.0152
August	1	50	0.067	6.9	5.30	0.0076
	2	25	0.067	6.9	5.30	0.0095
	4	12,5	0.067	6.9	5.30	0.0152
	8	6,25	0.067	7.0	5.30	0.0133
	16	3,12	0.067	7.0	5.30	0.0303

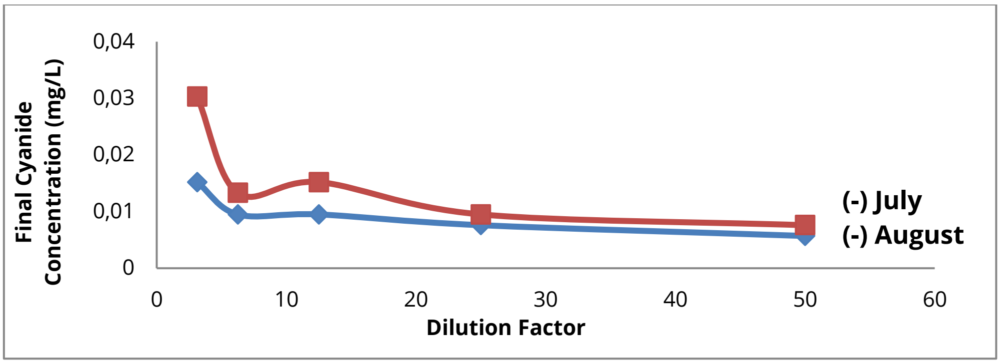

Figure 3. Comparison graph of cyanide concentration with dilution factor.

Table 4. Results of determining cyanide levels in samples from tailings dam with dilution factors.

FP	H_2O_2 concentration 50% (%)	$CuSO_4$ concentration (mg/L)	Initial pH	Final pH	Abs	CN- Concentration (mg/L)	
						Initial	Final
1	50	0.067	8.8	7.9	0.004	5.0	0.004
2	25	0.067	8.8	7.9	0.003	5.0	0.006
4	12,5	0.067	8.8	7.9	0.002	5.0	0.008
8	6,25	0.067	8.8	7.9	0.002	5.0	0.008
16	3,12	0.067	8.8	7.9	0	5.0	0.008

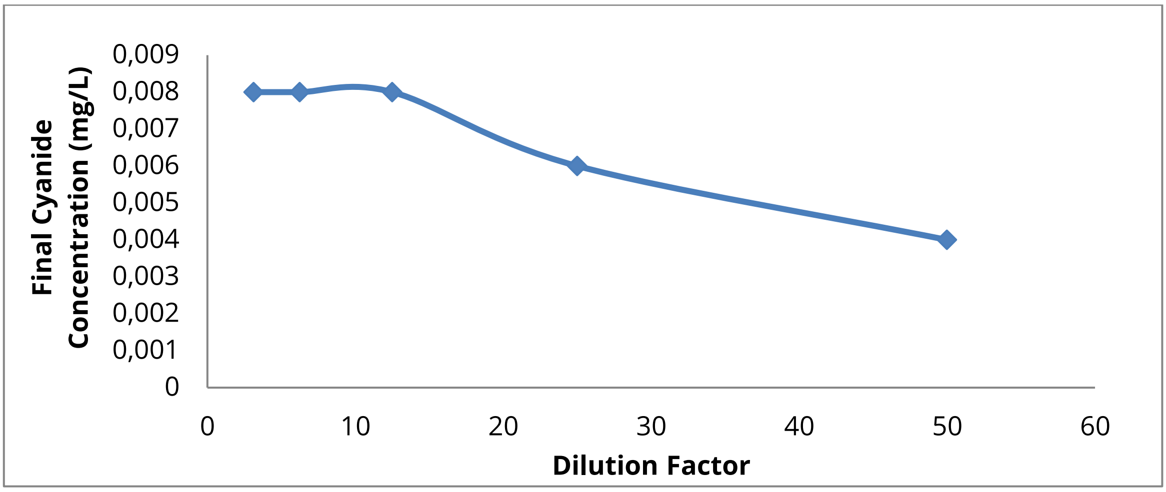

Figure 4. Comparison graph of initial cyanide concentration with dilution factor.

Cyanide levels in seepage samples and samples from dam tailings by varying the concentration of copper sulphate

Seepage samples were prepared with CN- concentrations of 1000mL each into 1000mL beaker glass, each beaker glass was added 0.33 grams of hydrogen peroxide, each solution was added 67mg/L copper sulfate solution as much as 1; 1.25; 1.67; 2.5; 5 mL. Then stirred with a flocculator for 15 minutes, the sample is ready to determine the cyanide level, carried out the same steps as the sample in the Tailing Dam. Variations in copper sulfate concentration were carried out to see the effective concentration of copper sulfate in degrading cyanide [[16]]. The test result data can be seen in Table 5 and Figure 5 for seepage samples, while in table 6 and Figure 6 for tailings dam samples.

Ion Cu is used as a catalyst in the Degussa method [[17]], in experiments obtained the greater the addition of copper sulfate catalyst the greater the cyanide degraded [[18]], this is seen in the graphs in experiments on July 24, July 28 and August 7 and the August 20 experiment for tailings dam samples, then the final cyanide concentration was measured using a UV-Vis spectrophotometer.

CN Concentration= 0.379 x Abs x Fp................. [5]

In addition to 5 mL $CuSO_4$, there is a decrease in the concentration of final cyanide (cyanide that has gone through the waste treatment process into 0.0133 mg/L.
CN Concentration= 0.379 x Absorbance x Fp .. [6]

Table 5. Result of $CuSO_4$ concentration variation experiments.

Date	Samples	H_2O_2 concentration (mg/L)	$CuSO_4$ concentration (mg/L)	Initial pH	Initial CN- Concentration (mg/L)	Final CN- Concentration (mg/L)
24th July	1	330	0.067	7.1	5.3	0.0322
	2	330	0.084	7.1	5.3	0.0303
	3	330	0.112	7.0	5.3	0.0208
	4	330	0.168	7.0	5.3	0.0152
	5	330	0.335	7.0	5.3	0.0133
6th August	1	330	0.067	6.9	5.3	0.0398
	2	330	0.084	6.9	5.3	0.0208
	3	330	0.112	6.9	5.3	0.0152
	4	330	0.168	6.9	5.3	0.0133
	5	330	0.335	6.9	5.3	0.0114
7th August	1	330	0.067	7.0	5.3	0.0133
	2	330	0.084	7.0	5.3	0.0114
	3	330	0.112	7.0	5.3	0.0095
	4	330	0.168	7.0	5.3	0.0038
	5	330	0.335	7.0	5.3	0.0019

Data: 21st August

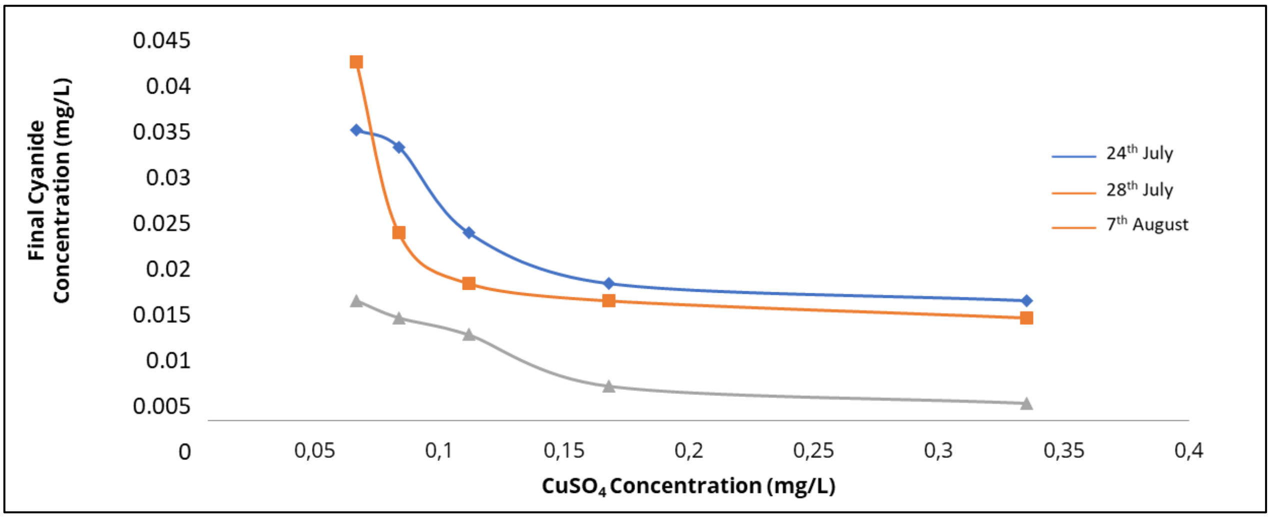

Figure 5. Seepage concentration variation graph after stirring with a flocculator for 15 min.

Table 6. Data on $CuSO_4$ concentration variation in samples from tailings dam.

Sample	H_2O_2 concentration (mg/L)	$CuSO_4$ concentration (mg/L)	Initial pH	Final pH	Konsentrasi CN^- Awal (mg/L)	Final CN- Concentration (mg/L)
1	330	0.067	8.4	7.7	5.0	0.044
2	330	0.084	8.4	7.7	5.0	0.041
3	330	0.112	8.4	7.8	5.0	0.036
4	330	0.168	8.5	7.8	5.0	0.034

5	330	0.335	8.5	7.8	5.0	0.028
1	330	0.067	7.7	7.7	5.0	0.040
2	330	0.084	7.7	7.7	5.0	0.038
3	330	0.112	7.8	7.8	5.0	0.036
4	330	0.168	7.8	7.8	5.0	0.034
5	330	0.335	7.8	7.8	5.0	0.030

Date: 20th August

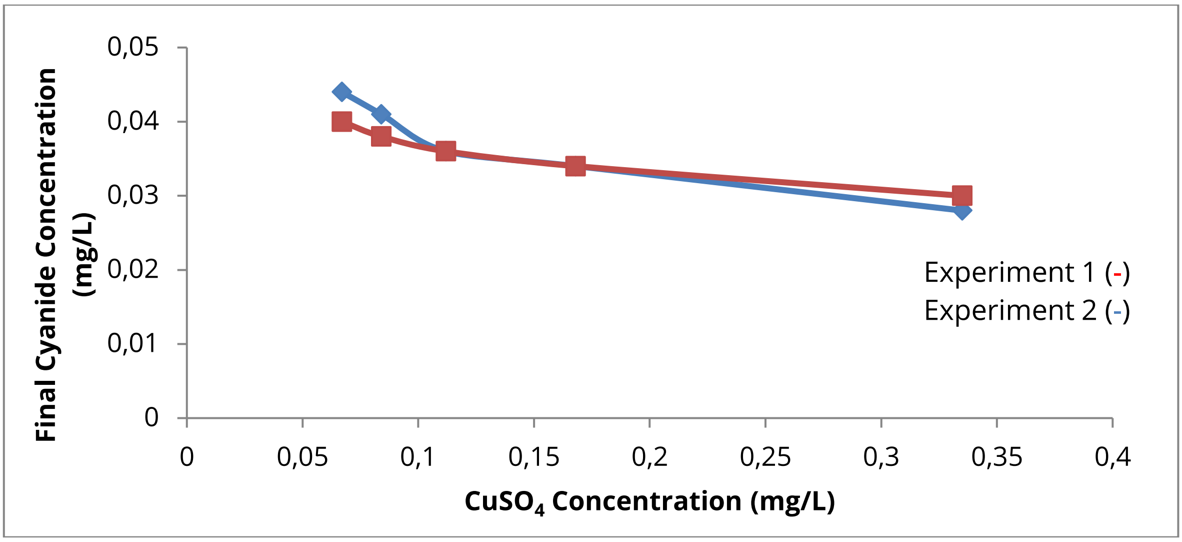

Figure 6. Graph of $CuSO_4$ concentration variation, in dam tailings sample**.**

The effective concentration of adding $CuSO_4$, is by adding 1 mL $CuSO_4$. The addition of 1 mL the concentration of degraded cyanide is already below the environmental threshold. It can be reduced the addition of $CuSO_4$ reagents, so that PT ANTAM UPBE Pongkor can more efficiently use $CuSO_4$ reagents and save costs in cyanide waste management.

The analysis results show that increasing pH and increasing CuSO4 concentration can reduce cyanide levels. This was also reported in several other studies, copper cyanide can be replaced by copper sulfate in the electroless brass bath and the deposition rate is maintained. This allows substitution the use of environmentally friendly brass plating, since wastewater is more simplified [19,20].

Conclusion

Based on research that has been done can be concluded at a concentration of hydrogen peroxide 330 mg / L, hydrogen peroxide is able to degrade cyanide below the environmental threshold of 0.5 ppm. The effective concentration of copper sulfate is in the addition of a concentration of 0.067 mg / L. The greater the addition of copper sulfate catalyst and the longer the waste treatment time, the greater the cyanide degraded and the variation in hydrogen peroxide dilution does not have a significant effect on the cyanide degradation process.

Acknowledgement

We would like to thank PT. ANTAM for sample support and Politeknik Negeri Sriwiajaya for facilities support.

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