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Comparative Analysis of Reagents in Gold and Silver Leaching Process Using the Bottle Roll Test Method

Rizki Ardiansyah[1*], Nabila Putri Wisnu Pratami[2], Zanescaya Wirasangka[3]
[1]Universitas Pembangunan Nasional "Veteran" Yogyakarta, Indonesia
[2]PT Geoservices Ltd., Indonesia
[3]School of Metallurgy and Environment, Central South University, China
*Corresponding author : 116210053@student.upnyk.ac.id

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Abstract

According to Law No. 4 of 2009, mineral processing in Indonesia must be carried out domestically. Cyanide (NaCN) remains the primary reagent in gold extraction due to its efficiency, but its negative environmental impact has driven the development of more environmentally friendly alternative reagents, such as thiosulfate and thiocyanate. This study aims to compare three types of reagents, namely NaCN, EG, and LW, in the gold and silver leaching process using the *Bottle Roll Test* method. The results show that LW achieved the highest gold extraction rate of 94%, followed by NaCN at 92% and EG at 88%. The best reaction kinetics were observed with LW, which reached maximum extraction within 8 hours. In terms of cost, NaCN was the most economical at 11.29 USD/ton of ore, lower than EG at 12.89 USD/ton and LW at 222.82 USD/ton. This study provides guidance for the gold mining industry in selecting the optimal leaching reagent based on extraction efficiency and cost-effectiveness.

Keywords: Reagent, Leaching, Bottle Roll Test, Economic, Extractive Metallurgy

Introduction

Indonesia is ranked as the sixth-largest country with gold reserves in the world in 2023. According to data from the Directorate General of Mineral and Coal, Ministry of Energy and Mineral Resources (ESDM), in 2021, Indonesia produced 118.3 tons of pure gold, making it the ninth-largest gold producer globally. Located in the Pacific Ring of Fire, characterized by volcanic activity due to tectonic plate movement and sub-surface tectonic shifts, Indonesia's mining potential is substantial. The Ministry of Energy and Mineral Resources (ESDM) reports that Indonesia has approximately 1,182,073.53 hectares of gold mining areas spread across 25 provinces (Panjaitan et al., 2023).

As the mining industry grows in Indonesia, the demand for mining companies to add value increases. This is reflected in the 2009 Mineral and Coal Mining Law (Law No. 4/2009), clarified by Government Regulation No. 23/2010 (PP No 23/2010). The regulation requires mining license holders to process and refine minerals domestically, eliminating the export of raw mineral materials (Ruben et al., 2021).

Gold processing technology must consider the characteristics of each type of gold ore in order to achieve optimal results (Lunt et al., 2016). Globally, the majority of Au is extracted from open pit mines, where massive amounts of earth are excavated and processed for targeted metal recovery. It is estimated that to produce a single gold ring, as much as 400 tonnes of rock and soil are mined(Adams et al., 2020). The cyanidation method for gold (Au) and silver (Ag) leaching, which offers high recovery rates, is commonly used (Rapele et al., 2022). Cyanide salts (NaCN/KCN) in the cyanidation process bind gold ions into a gold complex or aurocyanide (AuCN-) (Fahira, 2022). Cyanide salts are the most commonly used reagents due to several advantages, such as high extraction rates, ease of control, low investment costs, simple installation, and proven effectiveness ((Mufakhir et al., 2020). Additionally, cyanide-based ligands exhibit high efficiency in dissolving gold and superior selectivity for gold over other metals (Suratman, 2017).

However, if the cyanidation process is not well-controlled, high pH levels (> pH 12.5) can slow down the reaction kinetics, while low pH levels (< pH 9.4) may result in the loss of cyanide ions (CN-) that can convert into toxic cyanide gas (HCN)(Pitoi, 2015). Cyanide waste is highly toxic, but if managed properly, it can be rendered harmless. The toxic properties of cyanide are temporary and can be easily broken down with the help of additional chemicals in the environment(Sulistiyono et al., 2016). Due to the potential environmental impact, the use of cyanide is highly regulated and requires special permits, leading to increased research on alternative reagents such as thiourea, thiosulfate, iodine, chlorine, thiocyanate, and others.

According to research by (Badriyah, 2016), leaching with thiourea ($CS(NH_2)_2$) is considered a much safer alternative compared to cyanidation and amalgamation. The addition of Fe oxidizer can enhance the stability of thiourea in gold leaching processes, replacing cyanide, especially in low-sulfide and primary ores. Thiourea is relatively non-toxic and environmentally friendly. Its lower toxicity compared to cyanide makes it safer for use in gold extraction processes (Marsden & House, 2006). The use of thiosulfate reagents in gold extraction processes is currently being developed. This is attributed to thiosulfate's ability to form complex compounds with gold. Under neutral and alkaline conditions, thiosulfate reagents form anionic complexes [Au(S2O3)2] in basic and neutral conditions(Yustanti et al., 2018). The main degradation products of thiourea are sulfur and cyanamide, which can be used as fertilizer, making it more environmentally friendly.

Thiocyanate (SCN) is an analog of the cyanate ion [OCN], where oxygen is replaced by sulfur. It is also known as rhodanide (from the Greek word meaning "rose") due to its red-colored complex with iron. Thiocyanate is produced through the reaction of elemental sulfur or thiosulfate with cyanide (Putri, 2018). In addition to using chemical reagents, leaching can also be carried out using the bioleaching method. Bioleaching is a process of releasing (removing) or extracting metals from minerals or sediments with the help of living organisms, or of converting insoluble sulfide minerals into a soluble form in water by utilizing microorganisms(Muktiana, 2020).

The choice of reagent in the leaching process not only considers the recovery of the process but also its economic feasibility. High recovery is desired, but the reagent should also be economical to use. Therefore, experiments are necessary to compare both recovery and the economic aspects of reagent use in the leaching process.

This study will further explore the use of several types of reagents in the leaching process. The analysis is expected to provide valuable information for the gold mining industry in Indonesia to select the most suitable reagents for efficient and economically viable gold extraction processes.

Research Methods

This study uses the Bottle Roll Test (BRT) method for a duration of 24 hours. In the BRT experiment, the sample is placed in a bottle with holes in the lid to allow air to enter. The bottle is then rotated at a constant speed, and sampling is conducted at intervals of 2, 4, 8, and 24 hours.

The equipment used in this experiment includes a bottle roll, machine/rotor, Erlenmeyer flask, buret, dropper pipette, pH meter (Lutron WA-2017SD), DO meter (Lutron WA-2017SD), urine pot, centrifuge (DM 0636 Multi-Purpose Centrifuge), and falcon tube. The materials used include a -75 µm gold ore sample, NaCN leaching reagent (Reagent A), EG leaching reagent (Reagent B), LW leaching reagent (Reagent C), lime, Rhodanine, silver nitrate ($AgNO_3$), and water.

The research process begins with sample preparation from a mine in West Nusa Tenggara. The sample is first dried using an oven at 60°C for 12 hours, then subjected to crushing and grinding until it reaches a P80 size of 75 µm. The sample is then separated using a filter press and dried again before homogenization using a pulverizer.

Prior to the experiment, head grade analysis is performed to determine the initial gold and silver content in the ore. This analysis is carried out using the Fire Assay method for gold and ICP for silver. The Fire Assay method is chosen for its high accuracy and relatively low cost.

Leaching tests are conducted using BRT at the PT Geoservices Metallurgical Laboratory. A 500-gram sample is placed into the bottle, and water is added to achieve the desired solid percentage. Conditioning is then carried out by checking pH and dissolved oxygen (DO). After raising the pH to 10.2-10.5 using lime, the leaching reagent is added to the bottle. The leaching process is performed by rotating the bottle, and sampling is carried out periodically at hours 2, 4, 6, 8, 24, 36, and 48. During sampling, 2 urine pots of Pregnant Leach Solution are taken, then placed in a centrifuge to separate the solution from the solids. The separated solution is then sampled at 45 ml to check the Au content that has been extracted. Additionally, pH and reagent concentration are checked using argentometric titration. After sampling, lime is added if the pH of the solution is below 10.2, and reagents are added to maintain their concentration, along with water to maintain the solid percentage. The data obtained from each sampling are analyzed to determine the gold (Au) and silver (Ag) concentrations in the solution.

After the leaching process is completed, the slurry is separated into filtrate and residue. The filtrate is collected as hazardous waste (B3), while the residue is tested using fire assay to determine the remaining gold and silver content.

The Pregnant Leach Solution (PLS) is analyzed using Atomic Absorption Spectrophotometry (AAS) to determine the Au and Ag content. Atomic absorption spectrometry (AAS) is an analytical technique that measures the concentrations of elements. Atomic absorption is so sensitive that it can measure down to parts per billion of a gram (µg dm–3) in a sample. The technique makes use of the wavelengths of light specifically absorbed by an element(Welz & Sperling, 2008). The tailing residue is also tested using fire assay to determine the remaining precious metal content in the residue. The flowchart of the research can be seen in the following figure.

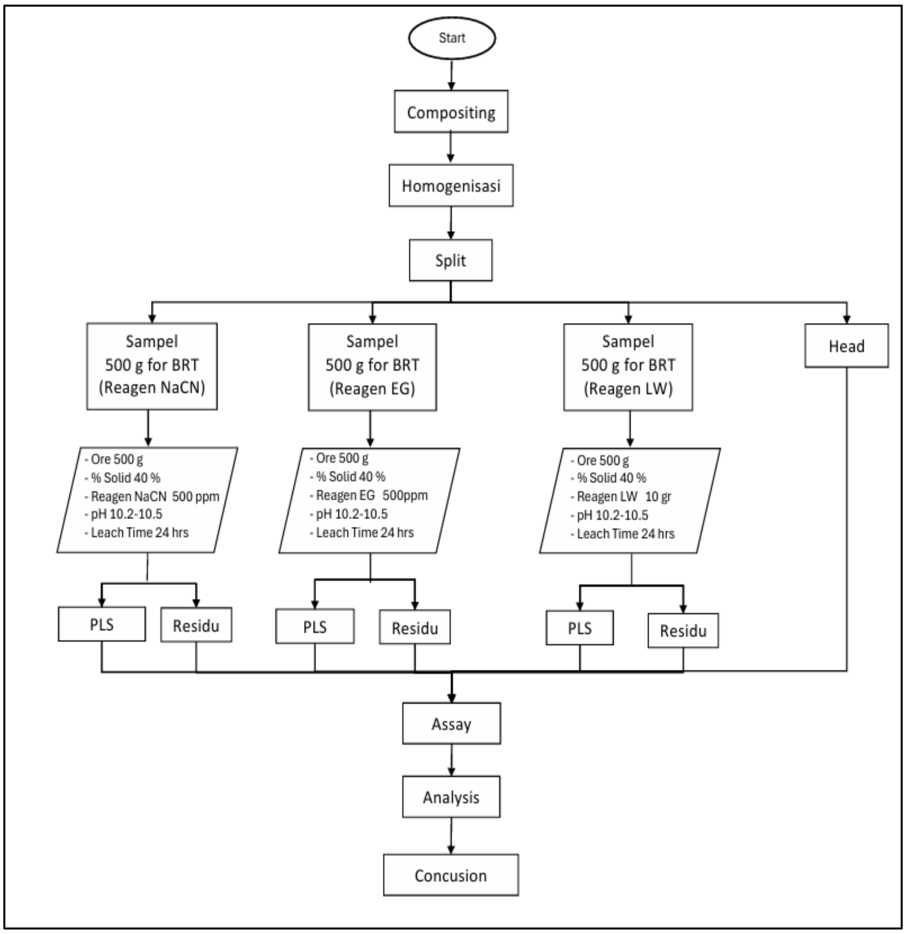

Figure 1 Research flow diagram

Result and Discussion

Results of Research

The sample used in the experiment underwent chemical analysis to determine the type of mineral present. Mineral characterization analysis was performed using XRD (X-ray Diffraction). XRD is a rapid analytical technique commonly used to identify phases within crystalline materials. Additionally, XRD provides information about the dimensions of a crystal unit cell.

Rietveld refinement is a technique developed by Hugo Rietveld for the characterization of crystalline materials. X-ray diffraction on powdered samples produces patterns characterized by reflections (intensity peaks) at specific positions. The height, width, and position of these reflections can be used to determinaae various aspects of a material's structure. The Rietveld method applies a least-squares approach to refine a theoretical line profile until it matches the measured profile. This technique represents a significant advancement in powder diffraction analysis as it effectively handles heavily overlapping reflections. However, Rietveld refinement does not account for amorphous or less crystalline phases that may be present and do not contribute to diffraction peaks.

In geology and mineralogy, a mineral group is a collection of mineral species that share essentially the same crystal structure and consist of chemically similar minerals. These groupings are based on the chemical and atomic structural classification of minerals. The classification includes Native Elements, Sulfides, Halides, Oxides/Hydroxides, Carbonates/Nitrates, Borates, Sulfates, Phosphates, Silicates, and Organics.

Based on XRD analysis, the results are presented in the following table.

Table 1. Phase Identification

Phase Identification	***Mineral Chemical Formula***	***%***
Albite	$NaAlSi_3O_8$	4,2
Alunite	$Kal_3(SO_4)_2(OH)_6$	1,1
Geothite	$FeO(OH)$	7,2
Illite	$(K,H_3O)(Al,Mg,Fe)_2(Si,Al)_4O_{10}[(OH)_2,(H_2O)]$	20,4
Kaolinite	$Al_2Si_2O_5(OH)_4$	9,4
Orthoclase	$KAlSi_3O_8$	2,7
Pyrite	FeS_2	0,6
Quartz	SiO_2	51,4
Rutile	TiO_2	2,8
	Total	**100%**

After identifying the phases of the sample's minerals, the mineral classification of the sample can be determined, as shown in the following table.

Table 2. Mineral Classification

Mineral Classification	***%***
Silicates	88,2
Sulfates	1,1
Oxides/Hydroxides	10,1
Sulfides	0,6
Total	**100%**

Based on the XRD analysis results above, it can be determined that the sample is composed of silicate minerals, with quartz as the primary mineral. Additionally, clay minerals such as illite and kaolinite are present, along with plagioclase in the form of albite and K-feldspar in the form of orthoclase. Other minerals found in this sample include iron hydroxide such as goethite, titanium oxide like rutile, and sulfate in the form of alunite. Moreover, small amounts of iron sulfide in the form of pyrite are also present. The analysis results indicate that minerals sample does not contain carbon. This is due to the very low mineral carbon content, making it undetectable. The low carbon content can be advantageous during the extraction process, as the small preg-robbing characteristics will not interfere with the extraction. In contrast, a high carbon content in the ore can hinder the gold extraction process (Aminah et al., 2022).

1. Analysis of the Effect of Reagent Variations on Gold and Silver Extraction Percentages

This experiment used different reagents in the leaching process, including NaCN, EG, and LW. Other experimental conditions were kept constant, including the sample weight (500 grams), particle size (P80 75 µm), pH maintenance at 10.5, leaching time of 24 hours, and sampling at 2, 4, 8, and 24-hour intervals. Based on the experimental results, the recovery graphs of Au and Ag for the reagent variations (NaCN, EG, and LW) are shown in the following figure.

Based on the figure, it can be observed that the highest Au extraction percentage was achieved using LW reagent, reaching 94%. This is followed by leaching with NaCN, which resulted in an extraction percentage of 92%, while the lowest was achieved with EG reagent, with an extraction percentage of 88%. The extraction percentage of Ag can be seen in the following figure.

The graph above shows that the highest Ag extraction percentage was achieved through leaching with NaCN and LW, both reaching 92%, while the lowest was obtained using EG, with an extraction percentage of 91%.

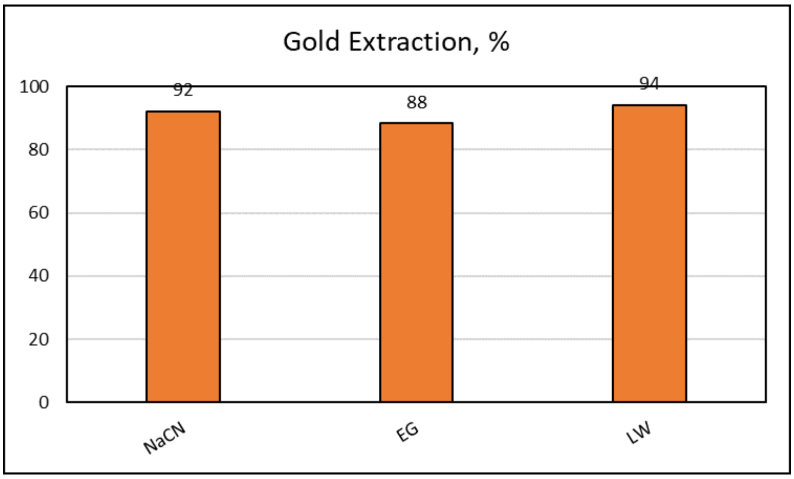

Figure 2. Graph of Au Extraction Percentage

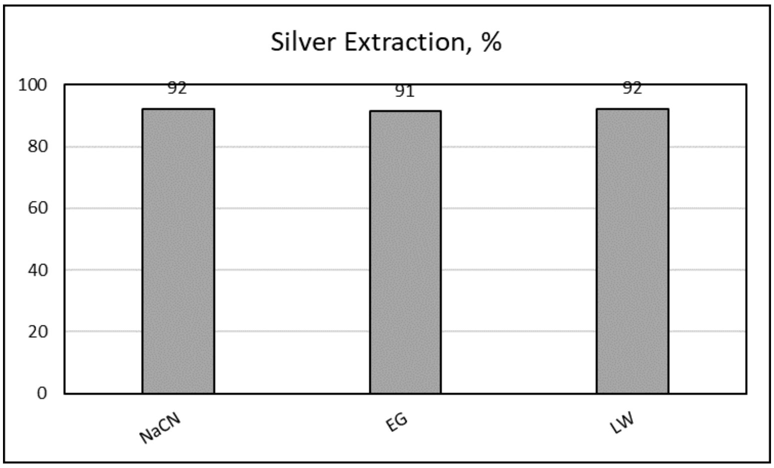

Figure 3. Graph of Ag Extraction Percentage

The NaCN used in this study is technical-grade NaCN, commonly employed in mining companies, whereas EG and LW reagents are produced by two different companies. LW reagent contains 75% cyanide and 25% Leachwell, which acts as a catalyst to accelerate the leaching process. In cyanide-based leaching, the chemical reactions involved can be described as follows (Birich et al., 2019):

$4\ Au + 8\ NaCN + O_2 + 2\ H_2O \rightarrow 4Na[Au(CN)_2] + 4\ NaOH$ ....(1)
$4\ Ag + 8\ NaCN + O_2 + 2\ H_2O \rightarrow 4Na[Ag(CN)_2] + 4\ NaOH$ ....(2)

From the above reactions, it can be seen that CN ions absorb gold and silver to form metal cyanide complexes, with NaOH as a byproduct. In the case of EG reagent, the main component is thiourea, which binds to gold during the leaching process. The leaching reactions using EG reagent can be described as follows (Tache, 2022):

$4\ Au + S_2O_8^{2-} + 4CS(NH_2)_2 \rightarrow 4Au[CS(NH_2)_2]^{2+} + 2SO_4^{2-}$ ....(3)

$4\ Au + 4CS(NH_2)_2 + 2H_2O + O_2 \rightarrow 4Au[CS(NH_2)_2]^{2+} + 4OH^-$ ....(4)

Based on the reactions above, it is evident that the absorbent for Au in this case is thiourea ($CS(NH_2)_2$).

LW reagent on the other hand is a high-cyanide-content reagent, comprising 75% cyanide which is typically used for intensive leaching. Intensive Leach is a specialized method in the leaching process designed to maximize the extraction of precious metals, particularly gold and silver, from concentrates with high efficiency. This method is often applied to concentrates rich in precious metals, such as those obtained from gravity or flotation processes. Unlike conventional leaching, intensive leaching is conducted under more aggressive conditions. One of its distinguishing features is the use of leaching solutions with higher reagent concentrations, such as cyanide or alternative reagents. Additionally, the leaching time in this method is shorter typically lasting only a few hours to 24 hours, compared to conventional leaching, which can take several days.

Theoretically, leaching with cyanide is more spontaneous than leaching with thiourea, as illustrated in the following figure.

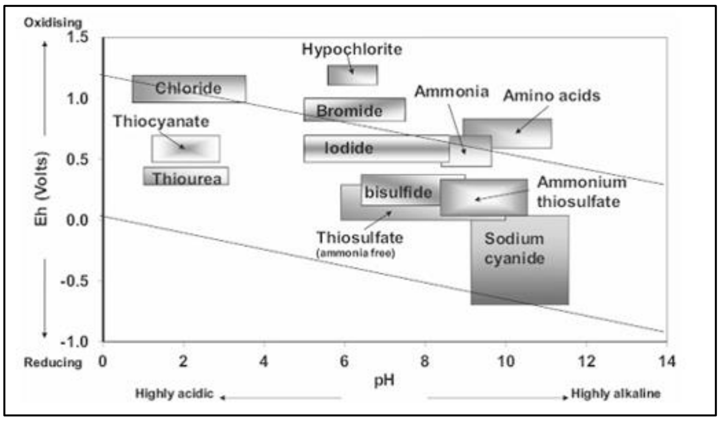

Figure 4. Eh-pH Diagram of Gold Solvent Reagents (Aylmore, 2016)

Based on the diagram above, it can be seen that leaching with cyanide has a more negative potential, making the reaction more spontaneous compared to leaching with thiourea. This aligns with the extraction percentage obtained. The highest extraction percentage was achieved in leaching with LW reagent, which is a high-cyanide reagent. This was followed by leaching with NaCN reagent, and lastly, leaching with EG reagent.

2. Analysis of the Effect of Reagent Variations on Leaching Process Kinetics

This experiment was conducted with a leaching time of 24 hours, with sampling at the 2-hour, 4-hour, 8-hour, and 24-hour intervals. After each sampling, reagents were added to maintain the reagent levels at the initial leaching concentrations. This process aims to observe the kinetics of the leaching reaction using the three different reagents used in the experiment.

Based on the experiments conducted, the kinetics of the leaching process using various reagents can be seen in the following figure.

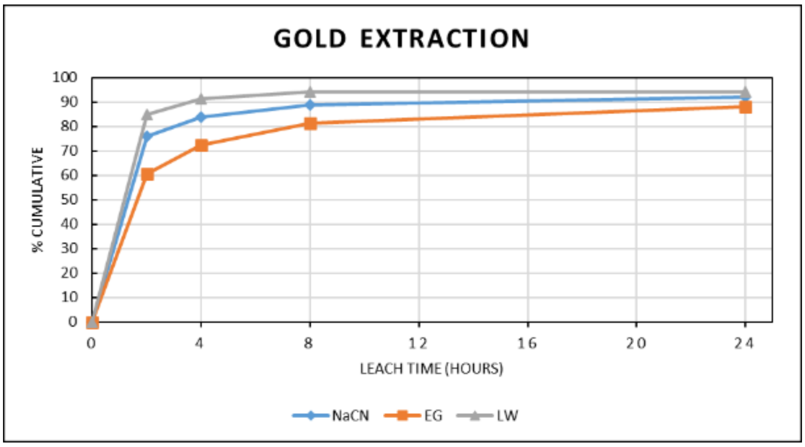

Figure 4. Kinetics of the Leaching Process on Gold Extraction

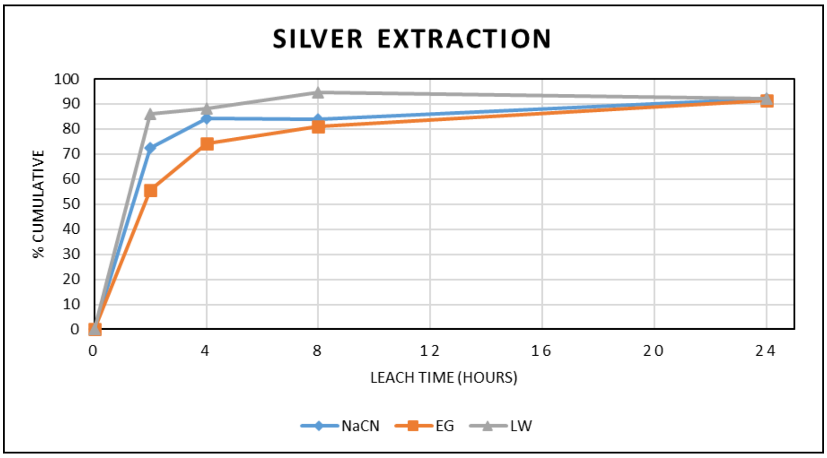

Figure 5. Kinetics of the Leaching Process on Silver Extraction

Based on the two kinetic graphs above, it can be observed that at the 2-hour mark, the highest cumulative percentage of extraction was achieved with leaching using the LW reagent. Meanwhile, leaching with NaCN reagent was still lower than leaching with LW, and the lowest cumulative extraction percentage was observed with leaching using the EG reagent. This trend continued up to the 24-hour mark. At the 8-hour mark, the increase in extraction percentage started to slow down, which is due to reagent saturation, indicating that most of the gold and silver had already been extracted by each reagent.

From the two graphs above, it can also be concluded that leaching with LW shows the best reaction kinetics, as gold extraction reached its maximum in the shortest time, specifically in 8 hours.

3. Analysis of the Effect of Reagent Variation on Reagent and Lime Consumption

In this study, reagent concentrations were monitored, and each sample was titrated to determine free CN levels. Reagents were added as needed to maintain the initial concentrations. Additionally, the pH was maintained within a range of 10.5 to achieve optimal extraction percentages. After the experiment, calculations were made for reagent consumption per ton of ore, as shown in the following graph.

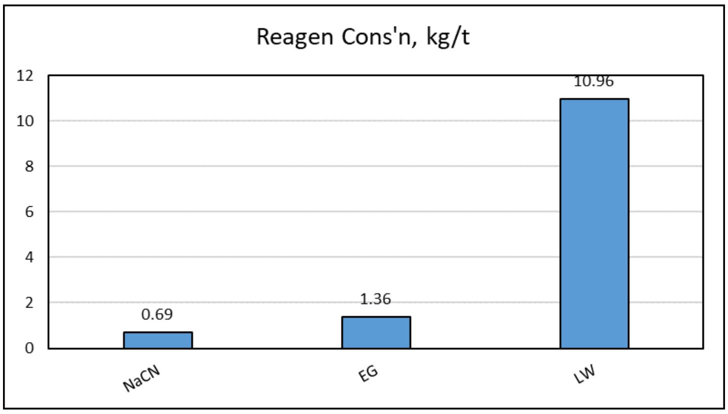

Figure 6. Reagent Consumption in the Leaching Process

Based on the image above, it can be observed that the LW reagent consumes the most, with a consumption of 10.96 kg/ton. In comparison, leaching with the EG reagent consumes 1.36 kg/ton, while leaching with NaCN has the lowest consumption at 0.69 kg/ton.

To maintain pH, lime is added during the leaching process. The amount of lime added is calculated and totaled, resulting in the lime consumption per ton of ore. This can be seen in the following image.

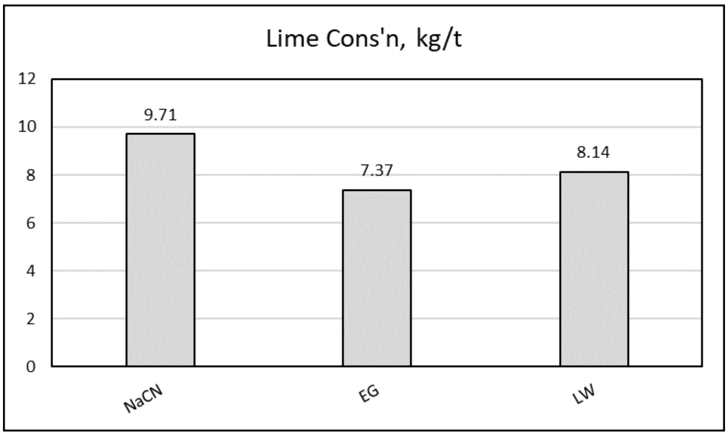

Figure 7. Lime Consumption in the Leaching Process

Based on the graph above, it can be observed that leaching with NaCN consumes the most lime, with a consumption of 9.71 kg/ton. Leaching with EG consumes the least lime, with 7.37 kg/ton, while leaching with LW consumes 8.14 kg/ton.

4. Analysis of the Economic Viability of Reagent Use in the Leaching Process

In mining companies, the main factor considered when choosing reagents for the leaching process, aside from recovery, is economic viability. This factor is crucial in determining the selection of reagents. After conducting the experiments, an economic calculation is made based on the cost of the leaching reagents and the cost of lime usage.

The cost calculation involves multiplying the price of the reagent and lime by the amount of reagent and lime used in the leaching process per ton of ore. The price for NaCN reagent is based on the average market price, while the prices for EG and LW reagents are the official prices provided by the manufacturers, as listed on the official websites of the respective companies.

The prices and usage of reagents in this experiment can be seen in the following table.

Table 3. Comparison of Reagent Prices and Usage

Keterangan	**NaCN**	**EG**	**LW**
Reagen Con'n, kg/t	0.69	1.36	10.96
***Lime* Cons'n, kg/t**	9.71	7.37	8.14
Harga, USD/kg	10	7	20
Reagen, USD/t	6.88	9.55	219.12
***Lime*, USD/t**	4.41	3.35	3.69
Jumlah Cost	11.29	12.89	222.82

The price of lime used is approximately IDR 7,000/kg or around 0.45 USD/kg. Based on the table above, a graph can be created that illustrates the cost of reagent usage in the leaching process per ton of ore, as shown in the following graph.

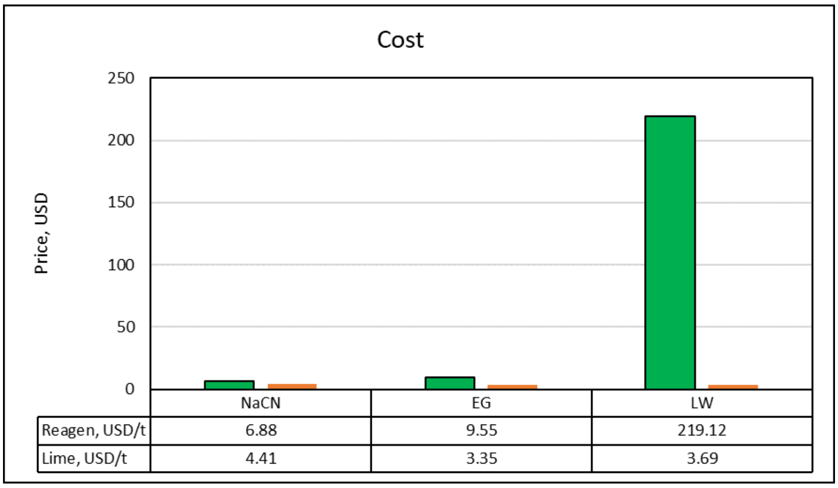

Figure 8. Comparison of Reagent Usage Costs in Leaching

Based on the graph above, it can be observed that leaching with NaCN reagent has the lowest cost, approximately 11.29 USD/ton of ore. Leaching with EG reagent costs around 12.89 USD/ton of ore, while leaching with LW reagent incurs the highest cost, approximately 222.82 USD/ton of ore.

Conclusion

Based on the results of the Internship activities, the following conclusions can be drawn:

1. The highest gold and silver extraction percentage was achieved through leaching with AT, reaching 94%.
2. Leaching with the AT reagent achieved maximum gold extraction in the shortest time, which was 8 hours.
3. In terms of reagent and lime usage, leaching with NaCN is more economical compared to other reagents, with a cost of approximately 11.29 USD/ton of ore and an extraction percentage of 92%.

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