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Analysis of Iron Content in the Leaching Process of Nickel Slag as Raw Material for Manufacturing Fero Sulfate

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Abstract---*Slag waste is waste generated from the nickel processing and smelting industry. Each process of refining one tonne of nickel products produces 50 times solid waste, equivalent to 50 tons of this problem which causes the waste stockpile to increase. The analysis of the iron content in the nickel slag leaching process as a raw material for the manufacture of ferrous sulfate is quite interesting because nickel slag chemically still contains valuable minerals and metals, and the iron content in nickel slag is the most dominant***.** *The purpose of this study was to determine the effectiveness of dissolving iron content in nickel slag from the leaching process which will later be used as a concentration for the manufacture of ferrous sulfate. This research is an experimental study to determine the dissolved Fe content that will be used to make ferrous sulfate coagulant products with concentrations of 0.2; 0.3; 0; 5 and 1 mol/L. In the results of the research that has been done, the concentration of 0.3 mol/L is the most effective concentration in dissolving iron in slag waste and from this concentration, the coagulant produced from the leaching process can degrade the turbidity of domestic waste by 83%, although its degradation ability is still below that of the PAC coagulant.*
Keywords---*ferrous sulfate, iron content, leaching, nickel slag.*

Introduction

Various kinds of industries have developed quite rapidly. One of the industrial fields that continues to develop is the nickel smelting industry, the impact arising from these industrial activities is the problem of waste. This issue has recently received attention from the government or national and international environmental agencies (Rambu et al. 2021).

To produce nickel in this process, nickel ore is then smelted in an electric furnace at a temperature of 1500-1600ºC to produce ferronickel and nickel slag (Setiawan, 2016). Most of the steel smelting companies carry out stockpiling of slag waste in open fields, which raises concerns about the leachate formed due to exposure to rainwater. This also happened at PT. Growth Java Industry which is stockpiling slag waste in open land. Slag waste can cause environmental pollution, especially the problem of changing the quality of surface water and ground water (Rambu et al., 2021).

Slag has the potential to produce leachate due to accumulation in open land and contains heavy metals in the form of ions which can cause disturbances to soil bio, plants, and reduce water quality. Cases of pollution due to nickel *slag* have occurred in the coastal area of Kolaka Regency in Monowari, the Pomala coastal area of Southeast

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Sulawesi, the pollution produced is heavy metal pollution that exceeds environmental quality standards including Fe, Zn, Cr, Pb, and Ni parameters. As a result of the disposal of *slag waste* which is used as backfill material near river water bodies which has the potential to cause river water pollution (Aprianto & Triastianti, 2018).

The Fe content is the dominant metal element in nickel *slag waste. On the basis of* it, it can be utilized as a coagulant which is made through the nickel *slag leaching* process using sulfuric acid. Therefore, it is necessary to utilize the *slag waste,* one of which is to use it as a raw material for ferrous sulfate heptahydrate (Wang et al., 2010; Wu et al., 2018). The process of making ferrous sulfate from iron waste is as follows:

$$FeO + H2SO4 \rightarrow FeSO4 + H2O \text{ ....(2)}$$

From the above equation, excess H 2 SO 4 was made so that greenish ferrous sulfate crystals were formed. The resulting ferrous sulfate crystals were then purified by recrystallization. Namely the crystals are dissolved with warm aquabides and filtered in a warm state. The filtrate is cooled so that crystals are formed (Jefriyanto, 2018).

The purpose of this study was to determine the effectiveness of dissolving iron content in nickel slag from the leaching process which will later be used as a concentration for the manufacture of ferrous sulfate. This research is an experimental study to determine the dissolved Fe content that will be used to make ferrous sulfate coagulant products with concentrations of 0.2; 0.3; 0; 5 and 1 mol/L.

Research Method

Ingredient

The material used in this research is nickel *slag waste from PT.* Growth Java Industry, 98% sulfuric acid (H2SO4), *Aquadest Water, Ferrous iron reagent,* PAC**,** and PT. Growth Java Industry.

Tool

The tool used in this is the Hach DR/900 Spectrophotometer, used as a tool to analyze the content of Fe dissolved in sulfuric acid, pH meter is used as a tool to analyze acidity in the leaching process, Jar Test is used for the stirring process and determining the effectiveness of the solution and a Turbidity Meter for analyzing the level of turbidity in domestic wastewater and other supporting glassware (Olimjonovna & Ibragimovna, 2021).

Work procedures

Solution Making Sulfate

Preparation of solutions with concentrations of 0.2; 0.3; 0; 5 and 1 mol/L. In the manufacture of sulfate solutions using the equation M1.V1 = M2.V2 Where M1 and M2 are the molarity (mol/L) of the solution, V1 and V2 are the volume of the solution in ml.

Leaching Process

In the Leaching Process, the concentration variations used are 0.2; 0.3; 0; 5 and 1 mol/L with 5 days of *leaching process.* The comparison between the extract solution and the extracted material is 400 grams of *slag* and 1 liter of acid solvent sulfate. This experiment was conducted to determine the effect of sulfuric acid concentration and time on the amount of dissolved Fe. So that the effectiveness of the concentration of acid that can dissolve the most iron content can be obtained nickel slag waste (Majalis et al., 2020; Rambu et al., 2021).

Nickel Slag Waste Treatment Becoming Ferrous Sulfate and Concentrating

The process of making ferrous sulfate is carried out after the leaching process for 5 days. After completing the leaching process and knowing the optimal concentration in dissolving, then the waste slag is prepared to be used and a glass beaker is prepared for the concentration stage. Next is the process of concentrating the liquid. Heat the ferrous sulfate liquid and stir with a stirrer at 100ºC. After changing the color to blue-green, remove and cool then do a test to determine the effectiveness of the ferrous sulfate coagulant that has been made with wastewater (Hiroyoshi et al., 1997; Georgiou et al., 2003; Li et al., 2007).

Coagulant comparison

To determine the effectiveness of the coagolan, do the stirring using the Jarr Test to determine the most effective turbidity concentration from the sulfate concentration used and wastewater. After going through stirring, the turbidity level of the domestic waste sample is tested by using a Turbidity Meter (Sunardi, 2016).

Result and Discussion

Potential of Heavy Metal Nickel Slag Leachate through TCLP Test

The TCLP test was carried out to determine the hazard characteristics of B3 waste produced from nickel *slag*. The quality standard in the TCLP test refers to PP No. 101 of 2014. The results of the analysis of metal content in the TCLP test are presented in the table below.

Table 1
Heavy Metal TCLP Test

No.	Parameter	Unit	Research result	Requirements TCLP-A	Requirements TCLP-B
1	Arsenic (As)	mg/L	0.07	3	0.5
2	Cadmium (Cd)	mg/L	0.01	0.9	0.15
3	Chromium (Cr)	mg/L	0.01	15	2.5
4	Copper (Cu)	mg/L	0.01	60	10
5	Lead (Pb)	mg/L	< 0.018	0.3	0.05
6	Nickel (Ni)	mg/L	7.47	21	3.5
7	Zinc (Zn)	mg/L	0.22	300	50

Based on the table above, the heavy metal elements produced from *slag waste* include Ar, Cd, Cr, Cu, Pb, Ni, and Zn. The results of the TCLP test that were carried out were still within the threshold because they did not pass the quality standards for the TCLP-A and TCLP-B categories. this can indicate that the nickel *slag produced from PT.* Growth Java Industry is still safe when used into other products so that it can be useful in reducing the amount of waste heap. Then this *slag waste* can be piled up on the ground directly and can be used as an alternative material.

Content of the Dominant Elements of Nickel Slag

Analysis result The dominant element content in nickel *slag* consists of the following elements : Si, MgO, Fe, Al, Ca, Cr, Mn, Ti, Ni, and Na. From the above element content, Si is the most abundant element in nickel *slag* produced from the test. Furthermore, the test results are classified based on the metal elements produced from the test. The metal content can be presented from the graph below.

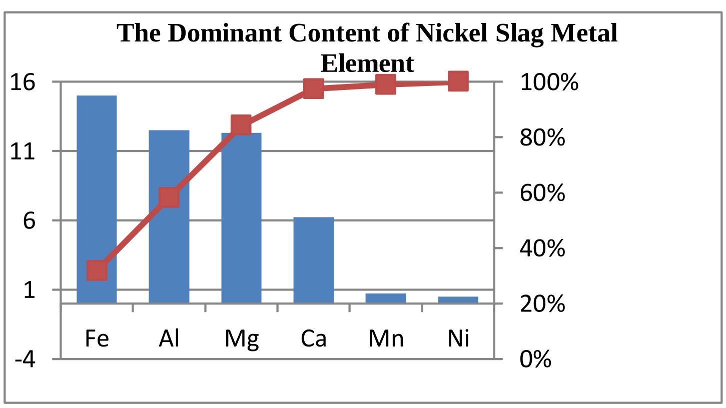

Figure 1. Diagram of the Dominant Content of Nickel Slag Metal Elements

The results of the X-Ray Florance test showed that the dominant element content of the slag waste was iron (Fe). The nickel slag waste is then extracted through a leaching process using sulfuric acid to dissolve the iron content so that it can be used as raw material for the manufacture of ferrous sulfate coagulant (Hsu et al., 2010; Moustafa, 2009; Liu et al., 2019). The Fe content was analyzed using the Hach DR/900 Spectrophotometer. The concentration variations used were 0.2; 0.3; 0; 5 and 1 mol/L with a time of 5 days.

Leaching Process Nickel Slag

Based on the lea ching process that has been carried out at a concentration of 0.3 mol/L sulfuric acid dissolves more Fe(II) and at this concentration is used in the manufacture of ferrous sulfate coagulant. This is because the acid concentration is relatively low but can dissolve relatively high Fe(II) with a dissolving concentration of 767 mg/L on day 5.

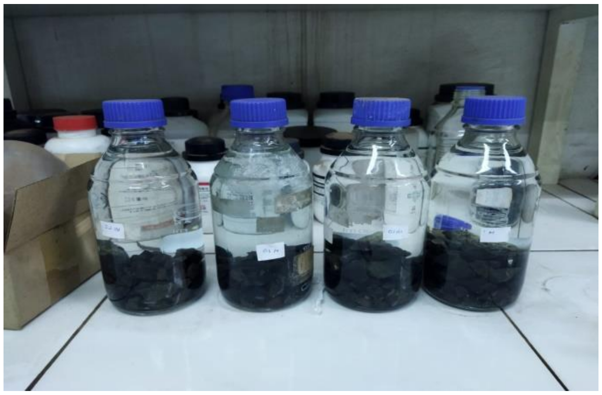

Figure 2. Nickel Slag Leaching Process Using Sulfuric Acid

The low acid concentration also makes the handling process easier if it is associated with safety aspects. The pH resulting from the leaching process tends to increase every day, because the longer the leaching time is carried out, the pH increases to normal pH. The use of sulfuric acid concentration in the range of 0.1 mol/L produces higher acidity because the smaller the pH value, the higher the acidity level produced.

Cu, Pb, Zn, and Fe Test Results in Leaching Solutions

In the process of leaching Fe(II) from nickel *slag* using H_2SO_4 it can dissolve other metals. This condition occurs because nickel slag has quite a lot of mineral and elemental composition. The next objective is to determine that the ferrous sulfate obtained can be used for wastewater treatment processes (Sahuquillo et al., 2003; Burgstaller & Schinner, 1993). For this reason, the extracted solution was analyzed for its metal content. In the *leaching* analysis using a *slag ratio of* 400 grams and a solution of 1000 ml with a sulfuric acid concentration of 0.3 mol. The test results for Cu, Pb, Zn, and Fe are presented in Table 2.

Table 2
Test results for Cu, Pb, Zn and Fe

No.	Parameter	Unit	Test result
1	Dissolved Iron (Fe)*	mg/l	764.07
2	Copper (Cu) Total*	mg/l	0.026
3	Lead (Pb) Total*	mg/l	<0.35
4	Zinc (Zn) Total*	mg/l	01.28

The test results above are taken from a solution of 0.3 mol sulfuric acid concentration. The dissolved Fe content obtained was 764.07 mg/L. this test was carried out using the AAS tool which was carried out by UPTD. Environmental Laboratory of DLHK BANTEN.

Comparison of the Effectiveness of Ferrous Sulfate with PAC

Comparison of effectiveness was carried out to determine the ability of the coagulant tested based on the resulting decrease in turbidity. The sample used in this coagulant comparison is PT. Growth Java Industry with variations in solution concentrations of 3 ml, 6 ml, 9 ml, and 10 ml. The turbidity produced before treatment was 57 FTU.

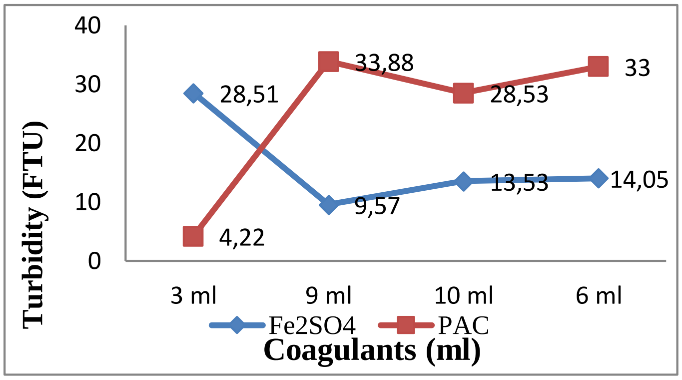

Figure 3. Turbidity Comparison Graph

Hypothesis, Correlation, and Determination Statistical Test

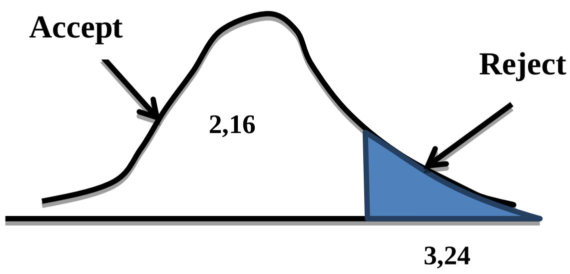

Figure 4. One way ANOVA hypothesis curve

F *count* = 2.162
F *table* = 3.24 (obtained from distribution table F *table* from Walpole book)
F *count* < F *table* then H0 is accepted and H1 is rejected, meaning that there is no significant difference between the dissolution of Fe and the solution concentration of 0.2 (A1); 0.3 (A2); 0.5(A3); and 1 mole (A4) H_2SO_4. k. statistic test results using Spss *One Way Anova* in Dissolving Fe with sulfuric acid for 5 days the hypothesis is that H0 is accepted and H1 is rejected, meaning that there is no significant difference between the dissolving of Fe and the concentration of the solution used . Then for the correlation and determination test, it was obtained from testing the effectiveness of the coagulant between ferrous sulfate and PAC.

From the results of the correlation test, it can be seen that the strong correlation value in these two coagulants is the correlation value of the ferrous sulfate coagulant producing an R of 0.867 or 86.7%. It can be concluded that to reduce turbidity in domestic waste ferrous sulfate requires a higher concentration than PAC coagulant. So that the PAC coagulant is still better in reducing turbidity with a lower concentration.

Conclusion

Based on the leaching process that has been carried out at a concentration of 0.3 mol/L sulfuric acid dissolves more Fe(II) and at this concentration is used in the manufacture of ferrous sulfate coagulant. The dissolution of iron obtained was 767 mg/L on the 5th day.

Result of the comparison of the effectiveness of the Fe_2SO_4 coagulant from waste slag and PAC. The most effective turbidity reduction of the coagulant tested on Fe_2SO_4 was at a concentration of 9 ml of turbidity which was obtained as much as 9.57 FTU. This can reduce the turbidity level of about 83 % while the most effective decrease in turbidity from 1% PAC is at a concentration of 3 ml with a turbidity of 4.22 FTU which can reduce the level of turbidity by about 93%. This study shows that the coagulant produced from slag waste is able to reduce effective turbidity and can compete with PAC coagulant although this coagulant is still in the research stage and needs to be studied more deeply.

In the results of the research that has been done, it can be seen that the coagulants produced from the leaching process can degrade the turbidity of domestic waste, although the degradation ability is still below that of the PAC coagulant. The TCLP parameters produced are still safe to use as building materials and other products so that it is expected to turn waste into high-value products and can be beneficial for environmental sustainability.

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