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Research Paper

Effect of Sulfur Content in Wetar Pyrite Waste on the Formation Process of Sulfuric Acid Products at AIM Project Morowali

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ABSTRACT: *Pyrite ore is a sulfide mineral commonly found in mining operations, and has the potential to be converted into valuable chemical products. The substantial quantity of high-quality pyrite ore waste generated by the Wetar Copper Mine is non-reusable, potentially causing environmental issues upon exposure to air. To mitigate potential environmental contamination at the Wetar Copper Mine, the AIM Project was initiated to process high-quality pyrite ore waste into sulfuric acid. This study aims to determine the effect of sulfur content, measured as a percentage of sulfur recovery, on the conversion of* SO_2 *gas to* SO_3*. Concentrate and calcine samples with particle size variations of 100, 120, 160 and 200 mesh were analyzed on a Carbon Sulfur Analyzer (CSA) with combustion temperature variations of 1100, 1150, 1200 and 1250˚C to determine sulfur content as a reference for the potential amount of* SO_2 *to* SO_3 *conversion in sulfuric acid formation. The results showed that the conversion of* SO_2 *gas to* SO_3 *increased with increasing particle size, with a particle size of 200 mesh yielding an optimum sulfur gas conversion percentage of 4.12%. At the same conditions, a maximum sulfur recovery rate of 96.66% was obtained from an average particle size of 200 mesh.*

Keywords: *AIM Project; Carbon Sulfur Analyzer; Pyrite Ore; Sulfuric Acid; Sulfur Recovery.*

1. INTRODUCTION

One of the most common sulfide minerals on Earth is pyrite [1]–[3], which is extensively distributed in deposits of precious metals [4] with a high iron content [5] and the mineral coal [6]. The mineral is found in a variety of geological settings, including sulfide deposits of volcanic [7], [8], and hydrothermal deposits [9].

Massive sulfide deposits have very high metal contents, such as Pb, Zn, Cu, Au, Ag, Se, Sn, Mn, Co, Cd, In, Ga, Ge, and Bi. Pyrite formed in hydrothermal deposits is associated with sulfides, oxides, quartz and other minerals [4], [8]. The association of pyrite with ore minerals such as sphalerite, galena or chalcopyrite makes the study of these minerals, in particular their alteration, of great economic and environmental importance.

Pyrite is a solid waste or by-product in the production process of non-ferrous metals (such as copper, lead, zinc and coal processing) which contains Iron (II) Sulfide (FeS_2), so pyrite is utilized as a mineral resource to produce sulfuric acid [9]. However, pyrite concentrate has a very high impurity content and the capacity of sulfuric acid produced is not high, so there is a limit to the sales of sulfuric acid produced [7], [10], [11].

Sulfuric acid can be produced commercially by a contact process where the modern trend towards contact processes using vanadium pentaoxide (V_2O_5) catalysts [12]. More specifically, the contact process combines the three basic operations, each of which is associated with a different chemical reaction. During the contact process, the sulfuric acid produced undergoes a four-stage process that begins with the smelting of elemental sulfur [13]. The process flow diagram in Figure 1 contains parts that follow a hierarchy, starting with SO_2 preparation, purification and oxidation, SO_3 absorption, and oleum dilution ($H_2S_2O_7$) [14].

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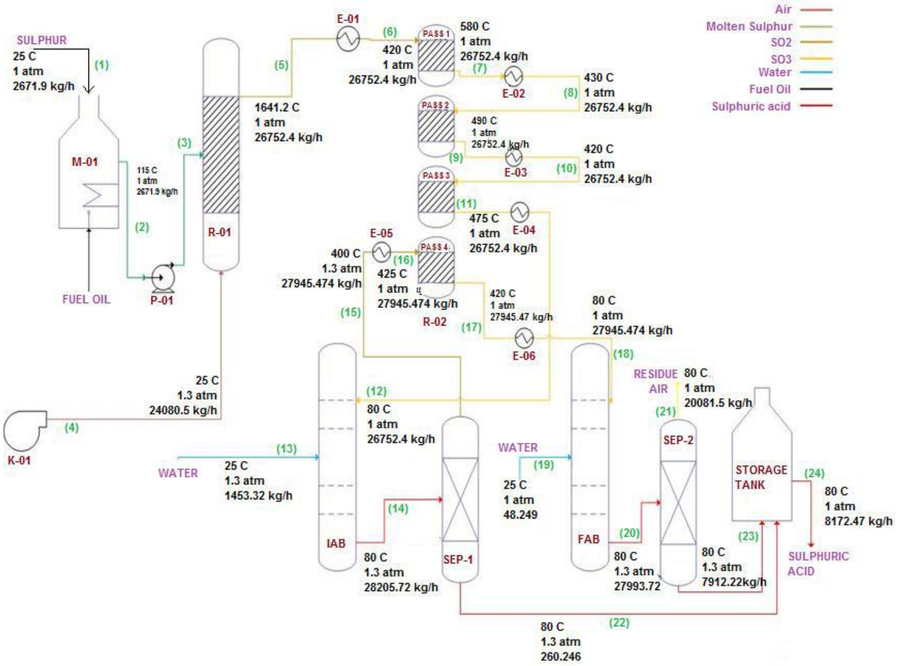


Figure 1. Flowchart of Sulfuric Acid Production Process [15].

First, the raw materials are burned into sulfur dioxide. The resulting sulfur dioxide is fed to a process unit (often referred to as a converter) where it is catalytically oxidized to sulfur trioxide. Typically, 95%-98% (v/v) of sulfur dioxide from the combustion chamber is converted to sulfur trioxide, by a heating process at high temperatures [16], [17]. After being cooled, the converter outlet gas enters the absorption tower. The absorption tower is a column in which acid is sprayed at the top and sulfur trioxide enters from the bottom to achieve the conversion efficiency of SO_2 to SO_3. Finally, sulfur trioxide is absorbed in water:

$$SO_{3(g)} + H_2O_{(l)} \rightarrow H_2SO_{4(aq)}$$

The AIM project is designed to process high-quality waste ore and pyrite ore sourced entirely from the defunct Wetar Copper Mine, which is then further processed into sulfuric acid. One of the factors in sulfuric acid production is the particle size of the pyrite concentrate and the roasting temperature in the roaster area.

The thermal process that takes place during roasting may be made more efficient by using the right particle size and heating temperature. This will ensure full breakdown and sulfur release, as well as boost SO_2 generation through heat distribution during combustion [18]–[20].

This study aims to identify the reference point and optimal point of the product in terms of the effect of sulfur content in Wetar pyrite waste on the conversion of SO_2 gas to SO_3 and sulfur recovery in the sulfuric acid formation process at the AIM Morowali project, in response to increasing market demand for sulfuric acid products.

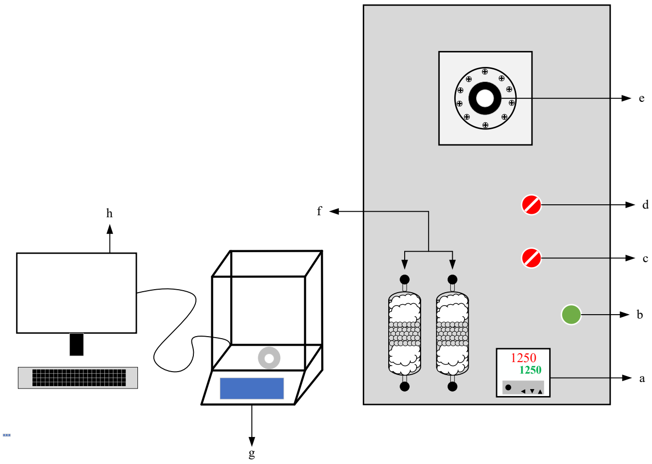
2. RESEARCH METHODOLOGY

2.1 Process Description

This research was conducted at Assay Laboratory, Technical Service Department, PT Merdeka Tsingshan Indonesia, Acid Plant. This research was conducted from June 11 to June 30, 2024. The description of the research process consists of :

2.1.1 Preparation of Tools

The instruments used included a Carbon Sulfur Analyzer (CSA, model CS2800G), Analytical Balance, Ceramic Crucibles, Oven, Spatula, Latex and Tray. Hematite type calcine (Fe_2O_3) with brown solid sample specifications obtained from the results of the calcination combustion process in the Roaster Area, and pyrite concentrate (FeS_2) with black solid sample specifications obtained from the flotation process at Pyrite Plant.



Description: a. Temperature Indicator; b. Power On/Off; c. Switch Mode; d. Analysis Mode Running; e. Input/Output Analysis; f. Gas Filter; g.Analytical Balance; h. Computer

Figure 2. Carbon Sulfur Analyzer Tool Set

2.1.2 Preparation of Materials

Raw materials, including pyrite concentrate and hematite-type calcine, were prepared for analysis. Two kilogram samples were separated using a splitter after being crushed in a double crusher. The samples were ground according to their respective types using fineness levels of 100 mesh, 120 mesh, 160 mesh, and 200 mesh to improve the efficiency of the thermal process that occurs during roasting so that the heat distribution during combustion can increase the formation of SO_2. Samples of various sizes were used for testing in the analysis of % sulfur.

2.1.3 Sample Analysis

Pyrite and calcine concentrate samples with predetermined fineness levels were analyzed by the Carbon Sulfur Analyzer (CSA) instrument. Samples of approximately 0.04 gr were weighed and then inserted into the CSA tool and heated with variations in combustion temperature used of 1100˚C, 1150˚C, 1200˚C, and 1250˚C until the sulfur testing analysis was completed. The use of temperatures above 1000˚C can maximize the complete decomposition of pyrite without requiring excessive energy during operation. After that, the % sulfur analysis data obtained is used as an estimate of the sulfur value

that enters the combustion process/roaster area in the calculation of SO_2 conversion to SO_3 and sulfur recovery.

2.1.4 Data Analysis

2.1.4.1 % Sulfur

The calculation of % sulfur is obtained from 4 stages of data processing. The % sulfur of each stage can be calculated with the following equation:

1. % sulfur of pyrite concentrate

The equation for % sulfur in pyrite concentrate is obtained from [21] :

% Sulfur = Pyrite concentrate input (Ton/Hour) x Sulfur Value (%).. (1)

When the mass flow rate of pyrite concentrate, is expressed in units (tons/hour), it is converted to mass units %b/b [22]–[24].

$$\%\ b/b = \left(\frac{\text{Mass flow rate of pyrite concentrate} \left(\frac{\text{tons}}{\text{hour}}\right)}{\text{Total mass flow rate of pyrite concentrate} \left(\frac{\text{tons}}{\text{hour}}\right)} \right) \times 100\% \qquad (2)$$

% Sulfur = %b/b of pyrite concentrate input (%) x Sulfur Value (%) .... (3)

The mass flow rate of pyrite concentrate and the total mass flow rate of pyrite concentrate are obtained based on the amount of pyrite concentrate raw material transferred during production. Meanwhile, the sulfur content is obtained from the readings of pyrite concentrate samples using a Carbon Sulfur Analyzer (CSA) instrument.

2. % sulfur of calcine

The equation for % sulfur in calcine is obtained from:

$$\% \text{ Sulfur} = \text{Calcine formed} \left(\frac{\text{Ton}}{\text{Hour}}\right) \text{ x Sulfur Value } (\%) \quad (4)$$

The calculation formula is obtained from the amount of sulfur contained in calcine (roasting product) based on the calcine production rate and sulfur content therein. When the mass flow rate of calcine formed, is expressed in units (tons/hour), it is converted to mass units %b/b [22]–[24].

$$\% \text{ b/b} = \left(\frac{\text{Mass flow rate of calcine} \left(\frac{\text{tons}}{\text{hour}}\right)}{\text{Total mass flow rate of calcine} \left(\frac{\text{tons}}{\text{hour}}\right)} \right) \text{ x } 100\% \quad (5)$$

% Sulfur = %b/b of pyrite concentrate input (%) x Sulfur Value (%) .... (6)

The mass flow rate value of calcine and the total mass flow rate value of calcine formed during calcination in the roasting process in the roaster area are obtained based on the amount of hematite (Fe_2O_3) calcine formed during production. Meanwhile, the sulfur value is obtained from the data read from calcine samples in the Carbon Sulfur Analyzer (CSA) instrument.

3. % sulfur of acid production

The equation for % sulfur in acid production is obtained from:

$$\% \text{ Sulfur} = (\text{Sulfuric acid production (Ton/Hour) x Sulfuric Acid Content } (\%)) \text{ x } \frac{32}{98} \quad (7)$$

This calculation is obtained from the principle of mass balance in the sulfuric acid production process [25]. When the mass flow rate of sulfuric acid, is expressed in units (tons/hour), it is converted to mass units %b/b [22]–[24].

$$\% \text{ b/b} = \left(\frac{\text{Mass flow rate of sulfuric acid} \left(\frac{\text{tons}}{\text{hour}}\right)}{\text{Total mass flow rate of sulfuric acid} \left(\frac{\text{tons}}{\text{hour}}\right)} \right) \text{ x } 100\% \quad (8)$$

$$\% \text{ Sulfur} = (\%\text{b/b of Sulfuric acid production } (\%) \text{ x Sulfuric Acid Content } (\%)) \text{ x } \frac{32}{98} \quad (9)$$

Description:

32 = relative mass of sulfur compounds (S)

98 = relative mass of sulfuric acid compound (H_2SO_4)

The mass flow rate of sulfuric acid and the total mass flow rate of sulfuric acid are obtained based on the amount of sulfuric acid produced. While the concentration of sulfuric acid produced ranged from >98.2%.

4. % sulfur of flue gas

The equation for % sulfur in flue gas is obtained from:

$$\% \text{ Sulfur} = (\text{Flue gas (Ton/Hour) x Acid gas waste content } (\%)) \text{ x } \frac{32}{98} \quad (10)$$

This calculation is obtained from the principle of mass balance in the sulfuric acid production process [25]. When the mass flow rate of flue gas, is expressed in units (tons/hour), it is converted to mass units %b/b [22]–[24].

$$\% \text{ b/b} = \left(\frac{\text{Mass flow rate of flue gas} \left(\frac{\text{tons}}{\text{hour}}\right)}{\text{Total mass flow rate of flue gas} \left(\frac{\text{tons}}{\text{hour}}\right)} \right) \text{ x } 100\% \quad (11)$$

$$\% \text{ Sulfur} = (\%\text{b/b of Flue gas } (\%) \text{ x Acid gas waste content } (\%)) \text{ x } \frac{32}{98} \quad (12)$$

Description:

32 = relative mass of sulfur compounds (S)
98 = relative mass of sulfuric acid compound (H_2SO_4)

The mass flow rate of flue gas and the total mass flow rate of flue gas are obtained based on the amount of SO_2 that cannot be converted into SO_3 in the absorber process. The acid gas waste content is obtained based on the amount of SO_2 gas absorbed in the absorber.

2.1.4.2 % SO_2 to SO_3 Conversion

The design and operation of a sulfuric acid plant is focused on the following catalytic gas phase chemical equilibrium reactions [26]:

$$2SO_{2(g)} + O_{2(g)} \rightarrow 2SO_{3(g)} \qquad \Delta H = -99\ kJ.mol^{-1}$$

This reaction is characterized by conversion, with the following equation:

$$\%\ \text{Conversion}\ (\%) = SO_{2\ (in)} - SO_{2\ (out)} \quad \text{.....(13)}$$

$SO_{2\ (in)}$ is the sum of the values of % sulfur of pyrite concentrate (%), % sulfur of calcine and % sulfur of acid production (%), while $SO_{2\ (out)}$ is the value of % sulfur of flue gas (%).

2.1.4.3 % Sulfur Recovery

Sulfur recovery is used to recover SO_2 gas that is not converted back to SO_3 in the converter stage, and the most commonly used conversion method is the Claus process. In theory above, it can be calculated with the following equation:

$$\%\ \text{Unconverted Sulfur}\ (\%) = SO_{2\ (in)}\ (\%) - \%\ \text{Conversion}\ (\%) \quad \text{.....(14)}$$

$$\%\ \text{Sulfur Recovery} = \frac{SO_{2\ (in)}\ (\%) - \%\ \text{Unconverted Sulfur}\ (\%)}{SO_{2\ (in)}\ (\%)} \times 100\% \quad \text{.....(15)}$$

$SO_{2\ (in)}$ is the sum of the values of % sulfur of pyrite concentrate (%), % sulfur of calcine and % sulfur of acid production (%).

3. RESULT AND DISCUSSION

3.1 Conversion of SO_2 to SO_3

After the sulfur percentage has been evaluated, the next step is to calculate and evaluate the sulfur conversion as a reference to determine the amount of sulfur dioxide (SO_2) gas oxidized to sulfur trioxide (SO_3) with a vanadium pentoxide catalyst in the converter unit at high temperatures.

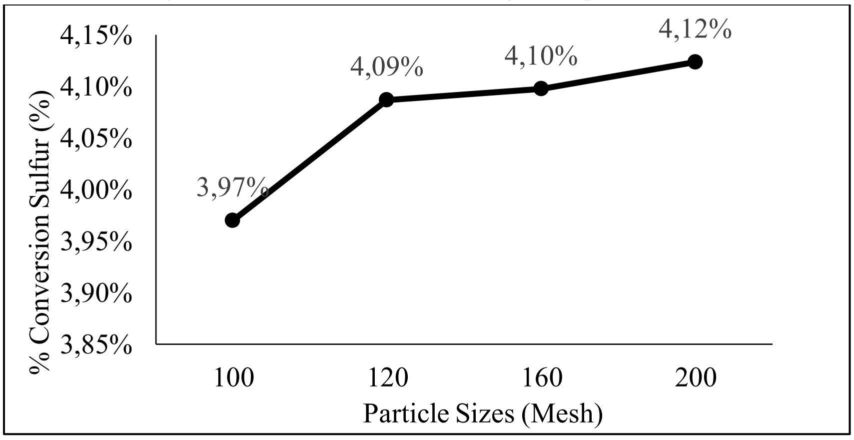


Figure 3. Performance of SO_2 to SO_3 Conversion for particle sizes: (a) 100, (b) 120, (c) 160 and (d) 200 mesh.

The evaluation results showed an increase in the conversion value of SO_2 to SO_3 in the combustion process in the roaster area. With particle sizes of 100, 120, 160, and 200 mesh, respectively, the average conversion of SO_2 gas to SO_3 was 3.97, 4.09, 4.10, and 4.12%. The primary molecule that may react rapidly and exothermically with water to generate sulfuric acid is SO_3, hence the rate at which SO_2 gas is converted to

SO_3 is crucial. As the rate of heat transfer increased, so did the conversion rate of SO_2 gas to SO_3, which increased with each particle size utilized [19]. Consequently, after roasting, the material's porosity increases, influencing heat dispersion and thermal stability in the roaster area [27]–[29]. In this instance, every particle size has the potential to improve the effectiveness of the thermal process that takes place during roasting, increasing the amount of SO_3 produced for every particle size utilized due to the heat distribution during combustion. The quantity of SO_3 gas generated, which will be transformed into sulfuric acid products in the converter unit, might therefore lead to an increase in sulfuric acid output [30].

3.2 Sulfur Recovery Efficiency

To determine how much of the total sulfur entering the process unit has been effectively converted into sulfuric acid, the % sulfur recovery rate must be calculated and assessed after the conversion rate of SO_2 to SO_3 has been assessed. This figure shows how well the production unit converts sulfur into products of sulfuric acid.

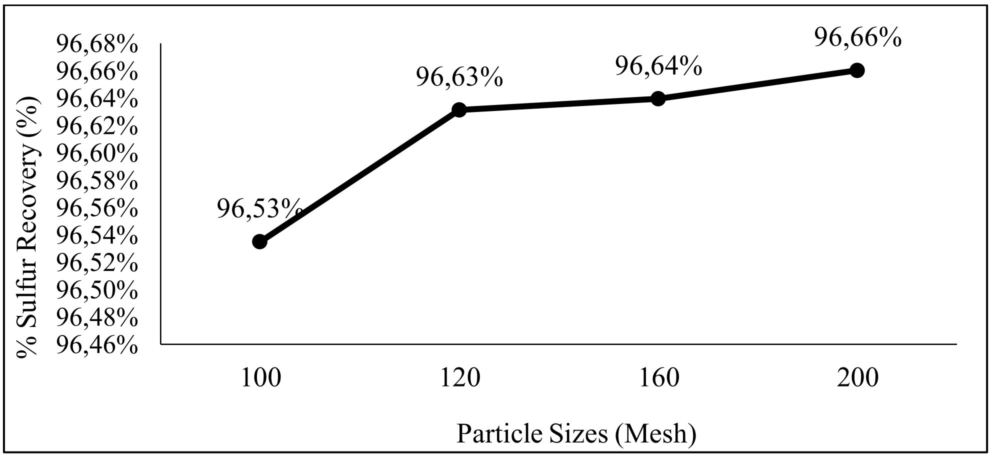


Figure 4. Sulfur Recovery Efficiency for particle sizes: (a) 100, (b) 120, (c) 160 and (d) 200 mesh.

The graph indicates that the following average values were obtained when the sulfur recovery percentage increased for each particle size used: 96.53%, 96.63%, 96.64%, and 96.66%. The highest sulfur recovery rate was obtained at a particle size of 200 mesh, this is because less sulfur dioxide (SO_2) is produced during the combustion process in the roaster area, which leads to less sulfur trioxide (SO_3) being created during the Claus process. Sulfur recovery is used to gauge the output of sulfuric acid production and improve the process to reduce losses. With sulfur recovery rates between 95 - 97 %, the Claus process has a very high efficiency [31], [32].

The highest sulfur recovery rate was achieved at a particle size of 200 mesh. These results are consistent with research [33] stating that finer particle sizes (200 mesh) and effective combustion temperatures (>1000 C) are used in the roasting process to produce optimally formed SO2 in the Roaster Area. This indicates that an optimal temperature of >1000˚C is used to ensure complete decomposition and release of sulfur [33].

4. CONCLUSION

Based on the results and discussion obtained in this study, The conversion of SO_2 gas to SO_3 during the combustion process in the roaster area at a particle size of 200 mesh and an effective combustion temperature of 1250 C acquired a percentage value of 4.12%, according to the study's findings and discussion. This demonstrates how well the production unit converts sulfur gas into products of sulfuric acid. The maximum sulfur recovery rate of 96.66% was obtained from the whole average under the same circumstances at a particle size of 200 mesh. This demonstrates how well the converter unit converts SO_2 gas to SO_3, allowing any unconverted sulfur gas to be collected before being released through the absorber process, thus mitigating the issue of SO_2 gas-induced air pollution.

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