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Research Paper Check for updates **Optimized Production of High Purity Sulphuric Acid via Contact Process Thlama Mperiju1* , Tome Sylvain2, Moses NyoTonglo Arowo3, Tilak Dhanda4, Abdulhalim Musa Abubakar1,5, Babakaumi Ahmadu Goriya1, Aminullah Zakariyyah Abdul1,6**

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Abstract

Sulphuric acid (H2SO4) is of immense importance to the chemical industry and humanity. The use of Aspen Plus modeling, simulation, and optimization via the contact process has enabled the production of grade sulphuric acid. Notably, the research findings highlight the sensitivity of water flow rate to the maximization of H2SO4 production. Through these methods, a production capacity of around 8 tons per day was achieved, with a purity level of 98.9%. This achievement significantly contributes to meeting the demand for sulphuric acid in various industries. Moreover, exploring alternative sourcing methods, such as utilizing elemental sulphur, offers the potential for further optimizing H2SO4 production. The benefits of improving H2SO4 production extend beyond the chemical industry. Sulphuric acid finds applications in agriculture, petroleum refining, pharmaceuticals, and metal processing. Enhancing the production process ensures a reliable supply for these sectors. In summary, sulphuric acid is indispensable to the chemical industry and humanity at large. Aspen Plus modeling and optimization techniques have successfully improved the production of high-grade sulphuric acid, resulting in increased capacity and purity. Exploring alternative sourcing methods further enhances production possibilities. These advancements have wide-ranging implications, benefiting multiple industries and driving progress in sectors reliant on sulphuric acid.

Keywords *Sulphuric acid; Optimization; Aspen Plus; Oleum; Contact process*

INTRODUCTION

Every year, around 200 million metric tons of sulphuric acid (H_2SO_4) are manufactured across the globe (Davenport et al., 2006; Grzesiak, 2011; SensoTech, 2020). The chemical is corrosive to the nose, mucous membrane, respiratory and gastrointestinal tracts, eyes, skin, and/or any tissue that it comes in contact with (Oberholzer, 2008). Main production routes are via the Contact Process and Lead Chamber process, which is meantfor large manufacturers (Sidana, 2016). The product is widely used in the pharmaceutical (e.g., drugs) and chemical industries (e.g., nitric acid, sulfate salts, and hydrochloric acid) as well as a source for producing phosphate fertilizer (e.g.,

superphosphate of lime), steel, pickling of metals, petrochemical, dyestuff, pigments, detergents and explosives (NPCS, 2012; Varnai et al., 2021). The highly corrosive mineral acid, especially around 200℃, can also be employed to purify gasoline and many other petroleum refinery products (Adeniran et al., 2017; Kemmerich & Storch, 2016). The Contact Process is the most common industrial method for the production of H_2SO_4 . The process involves the oxidation of sulfur dioxide $(SO₂)$ to sulfur trioxide $(SO₃)$, which is then dissolved in water to produce sulfuric acid. There are several optimization problems in H_2SO_4 production via the Contact Process that can impact the efficiency and profitability of the process. Optimization of the Contact Process in sulfuric acid production involves balancing a variety of factors to maximize efficiency, minimize byproduct formation, reduce energy consumption, extend catalyst life, and minimize downtime. Careful monitoring, analysis, and adjustment of process parameters can help achieve these goals, leading to a more efficient, profitable, and environmentally friendly process. Thus, the objectives of the study are to design a plant with the capacity of producing close to 8 tonnes/day of H₂SO₄, which is almost 98.9% pure, model and simulate the contact process method of production and identify unit or stream variables influencing H_2SO_4 recovery through Aspen Plus optimization.

RESEARCH METHOD

Aspen Plus version 8.4 was used to model $H₂SO₄$ production from elemental sulphur. FlowSim, Aspen Hysys, CHEMCAD, and UniSim, used previously in the literature, were studied to model and simulate the process using Aspen Plus (Almirall, 2009; Mounaam et al., 2020; Telang et al., 2001). Other analytical software was used to represent sensitivity plots so as to optimize the process.

Entering Components and Fluid Package

Components entered are oxygen (O_2) , nitrogen (N_2) , water (H_2O) , oleum $(H_2S_2O_7)$, sulphur (S), sulphuric acid (H₂SO₄), sulphur dioxide (SO₂), sulphur trioxide (SO₃) and air. NRTL fluid package was specified for the chemical process.

Method of Manufacture

Double contact double absorption (DCDA) and wet sulfuric acid (WSA) process forms the two main H2SO⁴ production methods (Al-Dallal, 2013; Narang, 2018; SensoTech, 2020). The contact process was used based on some factors that include; being more economical, yielding concentrated H2SO4, producing SO_3 and H₂S₂O₇, and a very large amount of H₂SO₄ can be recovered to desired endproduct capacity.

Process Description

A proposed process flow diagram contains sections that follow the hierarchy, beginning with $SO₂$ preparation, purification and oxidation, SO_3 absorption, and lastly, dilution of oleum $(H_2S_2O_7)$ (Sampat, 2022).Units employed as shown in Figure 1 are pump (P01), melter (M-01), reactor (R01), heat exchanger (E01, E02, E03, E04 & E05), catalytic converters (PASS1, 2, 3 & 4) separator (SEP1 & SEP2), tank (TANK), compressor (K01) and absorber (FAB).

Figure 1: Complete Process Flow Diagram of the Sulphuric Acid Production Process

Figure 1 is a slight modification of a simplified H_2SO_4 process flowsheet showing the 3 primary steps, including combustion of S to SO_2 in the presence of dry air, oxidation of SO_2 to SO_3 in a series of adiabatic catalytic packed beds, and absorption of SO_3 in a concentrated H_2SO_4 absorber and conversion to sulphuric acid, earlier illustrated in Zaker et al. (2021). Sulfur is usually obtained from pyrite and natural gas (hydrogen sulfide component) (Al-Dallal, 2013; Tveit, 2003). However, 59% of H_2SO_4 manufacture is from burning elemental sulfur as carried out in this work (Narang, 2018). Sulphur feed basis was specified and fed into a melter (M-01) to be melted. Specifically, at room temperature and atmospheric pressure, fuel oil was used to melt/burn 2671.904 kg of solid S in M-01 without contacting air. Hot, liquid, molten S at 115℃ is then pumped using P-01 to R-01. Air is sprayed in a countercurrent direction as feed to R-01 to oxidize the liquid S at 1641℃ according to reaction 1.

The catalytic converters are the 4-stage packed bed usually placed in the contact process. The reaction in the converter is an exothermic reaction where a temperature rise is expected. However, maintaining a temperature of around 450°C is essential for the maintenance of maximum conversion around the optimum equilibrium condition (other than maintaining pressure around the 1-2 atm). The catalytic action obviously plays a major role in the conversion step in both the lead chamber process and the contact process and its variations. Vanadium oxide (V_2O_5) as a possible candidate for catalysis of H_2SO_4 manufacturing in the oxidation of SO_2 has been under consideration since the beginning of the 20th century. The catalyst has a relatively low tendency of poisoning from contamination, low cost of acquisition and operation, high economic feasibility, and a better option for large-scale production.

The intermediate absorption tower (IAB) is inserted into the selected process to achieve high conversion efficiency in the conversion of SO_2 to SO_3 . Product SO_3 is fed to the main absorption tower (FAB). Sulfuric acid must be fed from storage tanks, and the outlet stream of oleum is sent to

their designated storage tanks. SO_3 reacts with H_2SO_4 and produces oleum. The usual absorption tower in the contact process is to be selected. The exothermic absorption might require a heat exchanger to be installed to maintain the temperature of the absorption process to prevent the excessive formation of sulfuric mist that, once formed, will be carried over by the flue gas. Flue gas from FAB contains oxides of S (and possibly oxides of nitrogen, contained due to high temperature during S melting and burning). SEP-1 scrubber is required to treat flue gas prior to discharge (flue gas desulfurization).

Oleum produced in the plant requires a holding tank (PASS 4). The tank can be used as the starting point of distribution of $H_2S_2O_7$ for the air drying unit, while the tank is to be fed from FAB and IAB columns. Oleum is converted to H_2SO_4 in the dilution tank. The flow rate of H_2O is dependent on the required concentration and $H_2S_2O_7$ collecting flow rate. The tank can also be used to equalize the produced final H2SO⁴ concentration. Sulfuric acid streams fed to FAB and IAB towers originate from the tank. The tank should contain agitators to facilitate proper mixing and homogenization. The final product from dilution tanks should be close to 98% sulfuric acid. Heat exchangers may be installed to capture heat emitted during dilution if required.

Reactions Step

Detailed reaction mechanisms of S combustion in H_2SO_4 plants can be assessed by Abumounshar et al. (2021). Here, a step-wise process governed by four reactions was employed in the reactors (Leiva et al., 2020). The guide was sought from Kumareswaran et al. (2013), who, in their work similar to this, described in detail the reaction steps of the contact process involving 4 reactive stages.

To prepare SO₂, elemental sulphur (S) is burned in oxygen (0_2) inside a reactor or burner at 1000°C via reaction (1).

$$
S + O_2 \rightarrow SO_2 \tag{1}
$$

Dust particles, arsenox oxide, vapors, S, and other impurities must be removed so that they don't poison the catalyst or make it lose its efficiency. Oxidizing SO_2 to SO_3 is carried out in a contact tower filled with V_2O_5 by reacting it with air or 02 to convert 98% of the SO_2 , as shown in reaction (2).

$$
SO_2 + \frac{1}{2}O_2 \rightleftharpoons SO_3
$$
 (2)

Normally, sulphuric acid manufacturing plants are the chief source of $SO₂$ release to the atmosphere because oxidation of SO_2 to SO_3 is a reversible reaction, and large quantities of unconverted SO_2 are vented to the atmosphere in the process (Mousavi, 2012). Sultana (2012), in his Master's Thesis, obtained 80% SO₂ emission reduction in the simulation of the sulphuric acid plant using Aspen Hysys. SO_3 is then dissolved to form oleum via the mechanism of reaction (3), which is then diluted with water to form H_2SO_4 through reaction (4).

$$
SO_3 + H_2SO_4 \to H_2S_2O_7
$$
 (3)
\n
$$
H_2S_2O_7 + H_2O \to H_2SO_4
$$
 (4)

Rate models at specific temperature ranges for the reactions and other reactor specifications are obtained by Zaker et al. (2021), Kiss et al. (2010), Nouri & Ouederni (2013), and Shi & Fan (2007). Previously, using UniSim and a double absorption process, Mounaam et al.(2020) obtained a 99.9% conversion to SO_2 and a 99.98% absorption rate of SO_3 . The H_2SO_4 production loop is a 500°C hightemperature manufacturing process (Seo et al., 2009).

FINDINGS AND DISCUSSION

Process Optimization

Components flowrates after running the simulation are shown in the Appendix. In the literature, a prior study of articles written by Tejeda-Iglesias et al. (2018), Kiss et al. (2006), and Sultana (2012) gives first-hand knowledge of process optimization.

Sensitivity analysis on units that are significant to H_2SO_4 yield was carried out. With pressure variation in reactor R-01, it was found that $SO₂$ yield is constant at 5338.20256 kg/h, as indicated in Figure 2.

The rate of this reaction can be influenced by several factors, including the flow rate of SO_2 . A higher flow rate of SO_2 can increase the rate of the reaction, leading to greater production of SO_3 and sulfuric acid. However, if the flow rate is too high, it can lead to an increase in pressure within the reaction vessel. The pressure within the reaction vessel is an important consideration in the design and operation of a sulfuric acid production plant. Higher pressure can increase the yield of the desired product, but it can also lead to safety concerns and the risk of equipment failure. The flow rate of SO² must be carefully controlled to maintain the desired pressure within the reaction vessel. If the flow rate of SO_2 is constant while the pressure within the reaction vessel changes during sulfuric acid production (Figure 2), it may indicate a change in the reaction conditions or a problem with the equipment. There are several factors that can cause a change in pressure within the reaction vessel, such as changes in temperature, changes in the flow rate of other reactants or products, or problems with the equipment, such as leaks or blockages. In some cases, a change in pressure may be intentional, such as during start-up or shutdown procedures. However, if the flow rate of $SO₂$ remains constant while the pressure changes, it suggests that the reaction conditions are not being maintained as intended. This can have a number of consequences, including a decrease in the yield of sulfuric acid, a change in the composition of the product, or a decrease in the efficiency of the reaction. For example, if the pressure within the reaction vessel drops below the desired level, it can cause the reaction to slow down or even stop altogether, leading to a decrease in the yield of sulfuric acid. Alternatively, if the pressure increases beyond the desired level, it can lead to safety concerns and equipment failure. Therefore, it is important to monitor both the flow rate of $SO₂$ and the pressure within the reaction vessel during sulfuric acid production to ensure that the reaction conditions are maintained as intended. Any changes in pressure should be investigated promptly to identify the cause and take corrective action as necessary to ensure the safe and efficient operation of the plant.

Temperature and pressure are not sensitive to SO_2 and SO_3 production, respectively, in the catalytic converters since the flowrate (4936.80147 kg/h in PASS1 and 26752.432 kg/h in PASS2) is not varying with temperature or pressure rise, as shown in Figure 3 and 4.

Figure 3: SO² Mass Flow Response to Temperature and Pressure Variation

Typically, the temperature can have an effect on the flow rate of $SO₂$ during sulfuric acid production. The reaction rate of the Contact Process, which is the most common industrial process used for producing sulfuric acid, is highly dependent on temperature. Therefore, changes in temperature can affect the rate of reaction, the yield of H_2SO_4 , and the flow rate of SO_2 . At higher temperatures, the reaction rate of the Contact Process increases, and the equilibrium of the reaction is shifted toward the formation of SO_3 and H_2SO_4 . As a result, the flow rate of SO_2 may need to be increased to maintain a constant rate of production. However, the temperature also affects the solubility of $SO₂$ in the reaction medium, which can have a direct impact on the flow rate of $SO₂$. As the temperature increases, the solubility of $SO₂$ in water decreases, which can lead to a decrease in the flow rate of SO_2 . In addition, at higher temperatures, SO_2 can react with water to form sulfurous acid (H₂SO₃), which can further decrease the amount of SO₂ available for the reaction. Therefore, to maintain a constant flow rate of $SO₂$, it may be necessary to increase the pressure within the reaction vessel or to use alternative methods to deliver $SO₂$ to the reaction.

But, if the flow rate of SO_2 is constant, as shown in Figure 3a, while the temperature changes during H2SO⁴ production, it can have several effects on the process. Firstly, changes in temperature can affect the rate of reaction and the yield of H_2SO_4 . The Contact Process for H_2SO_4 production is exothermic, meaning that it releases heat, and the reaction rate increases with temperature. Therefore, at higher temperatures, the yield of H_2SO_4 can increase, while at lower temperatures, the yield can decrease. If the flow rate of $SO₂$ is kept constant while the temperature changes, it can result in a change in the efficiency of the process and the amount of H_2SO_4 produced. Secondly, changes in temperature can affect the solubility of $SO₂$ in the reaction medium, as I mentioned in the previous answer. If the temperature increases, the solubility of $SO₂$ in water decreases, which can lead to a decrease in the flow rate of SO_2 . If the flow rate of SO_2 is kept constant while the temperature increases, the amount of $SO₂$ available for the reaction can decrease, leading to a decrease in the yield of H_2SO_4 . Lastly, changes in temperature can affect the stability of the equipment and the safety of the process. The reaction vessel and other equipment used in H_2SO_4 production are designed to operate at specific temperature ranges, and changes in temperature can affect their structural integrity and safety. If the temperature exceeds the design limits, it can cause the equipment to fail or create safety hazards.

As for Figure 3b, if the flow rate of SO_2 is constant while the pressure changes during H_2SO_4 production, it can indicate changes in the reaction conditions or problems with the equipment. Changes in pressure can occur due to various reasons, such as changes in temperature, the flow rate of other reactants or products, or equipment issues like leaks or blockages. In some cases, changes in pressure may be intentional, such as during start-up or shutdown procedures. However, if the flow rate of $SO₂$ remains constant while the pressure changes, it indicates that the reaction conditions are not being maintained as intended. If the pressure within the reaction vessel drops

below the desired level, it can cause the reaction rate to slow down or stop altogether, leading to a decrease in the yield of H2SO4. Alternatively, if the pressure increases beyond the desired level, it can create safety hazards and equipment failure. In such cases, it may be necessary to adjust the flow rate of $SO₂$ to maintain the desired pressure within the reaction vessel. Additionally, changes in pressure can affect the rate of reaction and the equilibrium of the reaction, leading to changes in the yield of H2SO4. For example, if the pressure decreases, the equilibrium of the reaction can shift towards the reactant side, leading to a decrease in the yield of H₂SO₄. Similarly, if the pressure increases, it can shift the equilibrium towards the product side, resulting in a higher yield of H_2SO_4 . Therefore, it is essential to carefully control both the flow rate of $SO₂$ and the pressure to maintain the desired reaction conditions and the yield of H_2SO_4 .

The behavior of a catalytic converter during H_2SO_4 production depends on various factors such as temperature, pressure, and the type of catalyst used. However, assuming that the flow rate of $SO₃$ is constant and the pressure inside the converter is also constant, it is likely that the reaction rate of the conversion of SO_3 to H_2SO_4 would be slower than if the pressure was increased. In a catalytic converter, the SO_3 reacts with water to form H_2SO_4 . This reaction is exothermic, which means that it releases heat. Increasing the pressure inside the converter can shift the equilibrium of the reaction to favor the formation of H_2SO_4 , resulting in a higher conversion rate. Therefore, if the pressure is kept constant, the reaction rate may not be as high as desired, leading to lower H_2SO_4 production rates, as shown in Figure 4a. Moreover, if the pressure inside the converter is too low, it can cause the formation of unwanted byproducts, such as sulfur trioxide and sulfur dioxide, which can be harmful to the environment. Therefore, it is crucial to maintain the pressure at an optimal level for efficient and safe H₂SO₄ production.

In Figure 4b, the SO_3 flow rate (\approx 26000 kg/h) being constant with temperature in a catalytic converter during H_2 SO₄ production can have several implications. Firstly, the H_2 SO₄ production process relies heavily on the catalytic converter to convert SO_2 to SO_3 , which is then used to produce $H₂SO₄$. If the SO₃ flow rate is constant, it means that the conversion rate of SO₂ to SO₃ will also be constant, which can affect the overall efficiency of the process. A constant flow rate may not be optimal for the process, and the conversion rate may be too low or too high depending on the operating conditions. Secondly, if the temperature is constant, it can lead to issues with the catalytic converter itself. The catalytic converter relies on a specific temperature range to function optimally, and if the temperature is too high or too low, it can reduce the efficiency of the converter and lead to a decrease in the production of SO_3 . Thirdly, the constant flow rate of SO_3 can also affect the overall quality of the H_2SO_4 produced. If the flow rate is too low, it can result in an insufficient amount of SO_3 , leading to lower H_2SO_4 production. On the other hand, if the flow rate is too high, it can lead to excess SO_3 , which can cause other issues, such as corrosion.

The flow rate of water against the flow rate of $SO₃$ varies. Therefore, it is sensitive. The optimum value for the flow rate of water to absorb the maximum SO_3 to yield an increased flow rate of H₂SO₄ is found to be 1458 kg/h. This sensitivity relationship is illustrated for the intermediate absorber (IAB) in Figure 5.

The water flow rate increasing with the H_2SO_4 flow rate from 1400-1458 kg/h, as shown in Figure 5, can be explained as follows. In the sulfuric acid production process, an intermediate absorber is used to absorb SO_3 from the gas stream produced by the combustion of SO_2 in the furnace. The absorption process takes place in the presence of water, and H_2SO_4 is formed. If the water flow rate in the intermediate absorber is increased while the H_2SO_4 flow rate is also increased, several effects can occur:

- Dilution of sulfuric acid: When the water flow rate is increased, it can dilute the H_2SO_4 solution formed in the absorber. This can lower the concentration of H_2SO_4 in the solution, which may affect the overall efficiency of the process.
- Reduced SO_3 absorption: As the water flow rate increases, it can reduce the amount of SO_3 absorbed by the intermediate absorber. This is because the increased water flow rate can reduce the contact time between the SO_3 and the water, resulting in less absorption of SO_3 and less $H₂SO₄$ formation.
- Increased load on downstream equipment: When the H_2SO_4 flow rate is increased, it can increase the load on downstream equipment, such as pumps and heat exchangers. This can cause issues such as increased wear and tear, reduced efficiency, and increased operating costs.
- Increased energy consumption: When the H_2SO_4 flow rate is increased, it can require more energy to pump and transport the acid. This can increase the energy consumption of the process and lead to higher operating costs.

Hence, holding the H_2SO_4 flow rate constant (at 7930 kg/h) while increasing the water flow rate in the intermediate absorber (Figure 5) can decrease the efficiency of the H_2SO_4 production process by decreasing the concentration of acid in the solution and decreasing the amount of $SO₃$ absorbed. However, it can also reduce the load on downstream equipment and decrease energy consumption. It is important to carefully monitor the concentration of H_2SO_4 in the absorber solution to ensure

that it does not become too diluted, leading to issues with acid strength downstream in the process. Same way, in the main absorber, the flow rate of water against the flow rate of $SO₃$ varies. Therefore, it is sensitive. The optimum value for the flow rate of water to absorb the maximum $SO₃$ to yield at a maximum density with an increased flow rate of H_2SO_4 is found to be 48.2 kg/h, as shown in Figure 6.

Normally, double-absorption facilities for H_2SO_4 synthesis have capacities of 1000-3000 tons/day (Garcia-Labiano et al., 2016). Hence for large-scale production, outputs from the contact process can be upgraded to achieve more than 8.2 tons/day (8172.47 kg/h) production capacity set in this work (Albrecht, 2016; Paatela, 2003). This can be done by optimizing production, which is not a simple task (Hanekom, 2017). Notwithstanding, Civetta et al. (2014) previously realized 46 tons/day of H2SO⁴ production in their work, Paatela (2003) in 2004 reported the realization of 1650 tons/day of the acid after revamp of the Bandirma Sulphuric Acid Plant in Turkey, while 4077 tons were realized between 2015-2017 in Zambia, in the Kansanshi Sulphuric Acid Plant as Mumba et al. (2017) relates. Satisfactorily, 98.9% of H₂SO₄ was obtained in this work, as other components in the product stream (S24) are negligible, while some appeared in trace amounts. In previous work, 98.5% of H2SO⁴ production was realized (Bhat & Pinjala, 2010; Sultana & Amin, 2011). In chemical process plants, a LiquiSonic analyzer can be used to measure H_2SO_4 concentration (SensoTech, 2020).

CONCLUSION

Process optimization is a means to achieve maximal or minimal output requirements in process units.In this work, appropriate unit selection and combination and feed and unit conditions specification helped in realizing the desired H_2SO_4 production capacity. Control, instrumentation, and plant layout can be implemented in future works. Efficiency in realizing the same concentration output with the double absorption process can be tried.

LIMITATION & FURTHER RESEARCH

In the future, the following should be looked into:

• Geber's method and other methods can be simulated using ASPEN HYSYS or other modeling and simulation tools

- Energy requirement and cost analysis can be carried out together with a layout of the plant's construction.
- Instrumentation and control should be a priority in achieving production set points or targets.

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APPENDIX Streams Component Mass Flows of all Units

Log Op Man Res.

