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RESEARCH ARTICLE

Process Modeling and Simulation of Ammonia Production from Natural Gas: Control and Response Analysis

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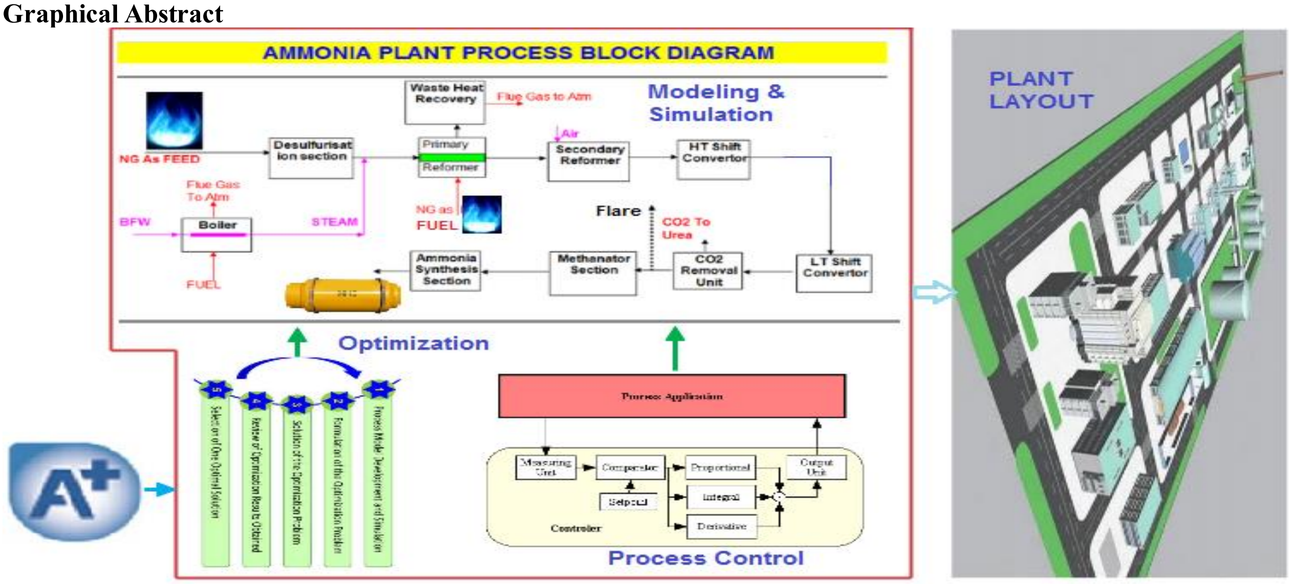
Abstract

Optimal production of ammonia (NH_3) using natural gas is necessary in order to make it available for wide range of applications including the manufacture of fertilizers, fuel for transportation and during synthesis of some chemicals. Achieving this would require strategic implementation of a control scheme to simulated ammonia production, capable of ensuring adequate realization of production targets. The work involves ASPEN Plus modeling, simulation, sensitivity analysis and control of NH_3 production process. Steam/carbon ratio, conversion of CH_4, removal of carbon dioxide (CO_2) and carbon monoxide (CO), hydrogen/nitrogen ratio and heat exchanger and separator temperatures were identified as requiring control in units any of these specifically impacts. As a result, approximately 176 tons of NH_3 was realized daily based on the simulation results and can be scaled-up using a calculated factor equivalent to 1.1375 to 200 tons/day capacity, in this design. Sensitivity analysis resulting in control of certain unit parameters is effective in ensuring process safety, maximum yield of important end-products and reduction in the cost of operation.

Keywords: Ammonia plant; Aspen Plus; Simulation; Process control; Natural gas; Sensitivity analysis; Modeling and optimization; Plant layout

Graphical Abstract



Introduction

Principal source of ammonia (NH_3) manufacture are hydrogen (H_2) and nitrogen (N_2) gas, following a natural or synthetic approach that are described as a complex heterogeneous catalytic or enzymatic process (Rouwenhorst et al., 2021; Tripodi et al., 2018). Roughly, half of the H_2 synthesized in industries (out of 60 million tons) and about 100 MMT of atmospheric N_2 every year, are extracted to produce NH_3 (Baltrusaitis, 2017; Philibert, 2017; Yilmaz & Ozturk, 2022). Liquefaction of NH_3 takes place by cooling the gas to -33°C at 1atm or rising the pressure to ≅10 bar at room temperature (Macfarlane et al., 2020; Stiewe et al., 2022). Major stages of production are steam methane reforming (SMR) of natural gas, water-gas shift reaction, and the Haber-Bosch process or simply grouped into the reforming and synthesis processes (Isah et al., 2019; Liu et al., 2020). While methods of NH_3 production as regards the feedstock used are divided into steam reforming from natural gas or other light hydrocarbons (e.g. naphtha and natural gas liquids), partial oxidation of heavy oil or waste oil, coal gasification and biological means using manure and organic wastes in the presence of microbes (Liu et al., 2020; Morgan, 2013; Partridge, 1976; Tavares et al., 2013; Zamfirescu & Dincer, 2008). Worldwide, 70-80% of NH_3 recovery is from natural gas by steam reforming, 22-30% using coal, 4% fuel oil and 1% naphtha (Bicer, et al., 2017a; Bicer, et al., 2017b; Brohi, 2014; Liu et al., 2020; Tavares et al., 2013). There exists, over 847 billion metric tonnes of proven coal reserve globally, the biggest being in the United States, Russia, China and India (Anantharaman et al., 2012; Burt, 1954; Liu et al., 2020; Pattabathula & Richardson, 2016). Ammonia is a high-purity source of carbon dioxide (CO_2) emissions and ≅ 1% of world's greenhouse gas emissions (Arora et al., 2018; Macfarlane et al., 2020). Morgan (2013) reported that, for 1 metric ton of NH_3 synthesized, 2.7 metric tons and 3.4 metric tons of greenhouse gas CO_2 is released from natural gas and coal respectively.

Areas of application of NH_3 are in explosives, wood and metal surface treatment plants, cosmetic plants, in paper, fertilizer and leather processing industries, as transportation fuel (e.g. during World War II) and in manufacturing plants like electronics, latex, paints, synthetic fibers and rubber, which makes it the second most manufactured chemical globally after sulphuric acid (Brohi, 2014; Ghavam et al., 2021; Liu et al., 2020; Oberauskas et al., 2020). Among the listed, 88% of NH_3 produced world over (of the 170 MMT annual output) goes to nitrogen fertilizer production, serving almost half of the world's food production (Chisalita et al., 2020; Gencer et al., 2022; Heidlage et al., 2017; Klerke et al., 2008; Liu et al., 2020; Macfarlane et al., 2020; Pattabathula & Richardson, 2016; Swearer et al., 2019). Gencer et al. (2022) reported that only 2 fertilizer plants in Nigeria and 1, both in Zimbabwe and Madagascar (making 4), out of a total of 16 in Sub-Saharan Africa (SSA), is in operation at the moment. At the moment, NH_3 utilization as fuel for electricity generation, heavy vehicle transport and energy storage are under development, because the chemical can go into gas turbines and internal combustion engines (Bartels & Pate, 2008; Bicer & Dincer, 2018; Macfarlane et al., 2020; Morlanes et al., 2020). Both NH_3 and H_2 are carbon free fuel alternatives; where Germany (where it began in 2018), Japan, China and Korea are actively planning the production of millions of fuel cell (Bartels & Pate, 2008; Calloway et al., 2019). Moreover, green ammonia is a term for NH_3 produced with zero carbon footprint which can be synthesized by electrolysis (Arora et al., 2018; Murai et al., 2022; Rouwenhorst et al., 2021; Swearer et al., 2019; Ye et al., 2017). According to level of CO_2 emitted, green ammonia production ensures 99.9% pure NH_3, blue ammonia ensures 90% while brown production (or the Haber-Bosch process) ensures that less than 90% of the gas volume is produced (Arrarte, 2022; Del Pozo & Cloete, 2022; Gezerman, 2022; Smart, 2022). Apart from a very high auto-ignition temperature (651 °C), high NOx emission, low flame speed, toxicity, narrow flammability limits (16-25% by volume in air) and high heat of vaporization that characterized NH_3 combustion which can be solved by working with somewhat oxygen (O_2) lean conditions and mixing with methanol, H_2 and gasoline, it is still better compared to H_2 (Brohi, 2014; Morlanes et al., 2020; Murai et al., 2022).

For SMR method utilizing natural gas to produce NH_3, if gas prices rise steeply, production cost sharply increase (Kelley et al., 2021; Kermeli et al., 2017; Khan & Kabir, 1995; Schnitkey, 2016). Specifically, natural gas is linked to 70-90% of NH_3 synthesis cost (Zamfirescu, et al., 2017; Huang, 2007). For instance, the world witnessed a decrease in natural gas prices between 2012-2016 due to increase in the gas production in the United States and since September 2021, Europe's natural gas costs are at record-high (Ghavam et al., 2021; Schnitkey, 2011; Stiewe et al., 2022). This would limit the purchasing power of users, especially the United States that accounts for 35-40% of world trade currently, being the largest importer (Zamfirescu, et al., 2017). The end-product or NH_3 can be stored and transported in huge cyrotanks onboard of ships, through mild steel pipelines and/or converting natural gas pipelines to transport it, as it is less costly to transport than H_2 in pipelines (Bartels & Pate, 2008; Zamfirescu, et al., 2017; Stiewe et al., 2022). An NH_3 pipeline built from the Gulf of Mexico to Minnesota which branches to Ohio and Texas is an example (Zamfirescu, et al., 2017). Storage of the gas is influenced by tank size, as 50000 tons NH_3 volume is stored at 33°C and 1 bar in large insulated tanks while amount around 1500 tons is stored under pressure in small stainless steel spherical tanks (Klerke et al., 2008).

However, before storage and transport, NH_3 is manufactured in process industries. In most cases, modeling, simulation, control, optimization, instrumentation and layout are designed (Demirhan et al., 2018; Singh & Saraf, 1981).

Modeling, simulation, optimization and control can be done on particular units or whole NH_3 process units (Araujo & Skogestad, 2008; Mahmoodi & Darvishi, 2017; Reddy & Husain, 1982). Process control had been used to improve plant capacity from 750-850 tpd (Araujo & Skogestad, 2008; Frahm et al., 2001; Mulholland, 1986; Shah, 1967). Commonly, refurbishment, replacement and redesign of an NH_3 plant helps in improving its capacity, efficiency and reliability (Dark & Stallworthy, 1985; Gupta & Borserio, 2004; Sanchez & Martin, 2018). Because energy, process risk, human failure and maintenance cost are two proven symptoms of NH_3 plant shutdown (Delboy et al., 1991; Eng & Gluckie, 2009; Kermeli et al., 2017; Williams, 1978; Williams et al., 1988). It is worthy of note that, implementing control strategies in NH_3 plants could sometimes be challenging (Funk, 1998). Specific objectives of the work are to specify natural gas grade which will go into a designed ASPEN Plus simulation of an NH_3 production plant, identify manipulated variables in need of control to keep prime units or conditions at desired set points by embedding a control scheme to the process design, carryout sensitivity analysis in order to optimize certain operating/unit/stream conditions and to determine the arrangement of different facilities within the processing area that minimizes construction cost and accidents.

Literature Review

Fritz Haber and Carl Bosch developed the Haber-Bosch NH_3 synthesis process in 1913 at Baden Aniline and Soda Factory (BASF) in Oppau, Ludwigshafen (Bartels & Pate, 2008; Brightling, 2018; Rouwenhorst et al., 2021; Smith et al., 2020). The exothermic process is facilitated by iron oxide catalyst at optimal temperature range between 300-600°C to combine N_2 and H_2 in the ratio of 3:1 at a pressure ranging from 100-350 bar (Bicer, et al., 2017b; Brohi, 2014; Flórez-Orrego & Junior, 2017; Macfarlane et al., 2020; Philibert, 2017; Rouwenhorst et al., 2021; Verleysen et al., 2020). Essentially, the H_2 from SMR or coal gasification and N_2 stripped from air is converted to NH_3 in a reactor – while unconverted syngas is recycled (Klerke et al., 2008). Since the inception of the method, there is a sporadic and continuous changes in the design of NH_3 synthesis reactors; yet limitations of the techniques including unfavorable thermodynamic equilibrium for NH_3 synthesis resulting in low yields per pass through the converter, hasn't been solved (Allman et al., 2017; Bartels & Pate, 2008; Jarullah et al., 2013). However, the energy intensive process now accounts to 90% of world's NH_3 manufacturing (Bartels & Pate, 2008; Yilmaz & Ozturk, 2022).

Global NH_3 plant capacity in the 1950s were a few hundred tons/day, which grows to an annual volume of 130 MMT in 2000, 133 MMT in 2008, 205 MMT in 2010, 235 MMT in 2019 and 239 MMT in 2020 (Anantharaman et al., 2012; Ghavam et al., 2021; Gosnell, 2005; Morgan, 2013; Siddiq et al., 2011). With an annual increment ranging from 1.67-2.3% in the last few years, its estimated value is put at 100 billion USD with 226 MMT average annual production between 2010-2020 (Brightling, 2018; Macfarlane et al., 2020; Smith et al., 2020; Zhang et al., 2019). First NH_3 plant was constructed by the German firm, BASF at Oppau, in 1913 (Pattabathula & Richardson, 2016; Rouwenhorst et al., 2022). After then, several other plants of higher capacities (Table 1) were developed, the largest being able to produce 3300 mtpd or 3640 stpd (Brightling, 2018).

Table 1: Capacities of Some Ammonia Plants and their Year of Construction

Ammonia Plant	Capacity at Inception	Current Capacity	Location	Year of Construction	Reference
BASF	30 mtpd	875000 mtpy	Oppau, Germany	1913	(Pattabathula & Richardson, 2016)
Hydro Agri, Sluiskil E-Braun License	1750 mtpd	-	Sluiskil, the Netherlands	1988	(Russo et al., 2010)
BASF- Uhde License	1800 mtpd	2060 mtpd	Antwerp, Belgium	1991	(Larsen & Lippmann, 2002)
Jiujiang Chemical Fertilizer Plant	300,000 mtpy	-	Jiangxi Province, China	1996	(Jiang et al., 2005, 2008)
RCF-Haldor Topsoe (Denmark) & Benfield Corporation (USA)	900 mtpd	2200 mtpd	Rashhtriya Chemicals & Fertilizer (RCF) Ltd, Bombay	1982	(Sharma, 1989)
P.T. Kaltim Pasifik Amoniak- Haldor Topsoe License	2000 mtpd	2700 mtpd	Bontang, Indonesia	2000	(Christensen, 2001)
Profertil S.A. Fertiliser Plant	2050 mtpd	**775000 tons**	Bahia Blanca, Argentina	2000	(Brigden & Stringer, 2000)

Ammonia 3 Plant/Juraj Dimitrov Bratislava Nitrogen Factory Sala-Uhde Technology	1300 mtpd	200000 mtpy	Former Czechoslovak Republic	2003	(Kessler et al., 2006)
Burrup Fertiliser Private Limited (BFPL)-KBR License	2200 mtpd	2200 mtpd	Burrup, Australia	2005	(Jovanovic et al., 2006)
Saudi Arabian Fertilizer Company (SAFCO)- Uhde license	3300 mtpd	3670 mtpd	Al-Jubail, KSA	2006	(Ruther et al., 2005)

It has been speculated that a 4000 mtpd is possible based on Uhde Technology (Ojha & Dhiman, 2010; Ruther et al., 2005). According to Arora et al. (2016), 1000 tpd ammonia plant is capable of meeting the demands of several small countries. However, for 1000 mtpd NH_3 plant, ≅ 3100 mol/hr of gaseous NH_3 is produced in NH_3 converter at about 440°C and 150 atm pressure (Rahman et al., 2014). The pilot scale NH_3 plant at West Central Research and Outreach Center at the University of Minnesota, 11 plants in Canada generating 4-5 mtpy of NH_3 and an ammonia plant at Billingham, UK are few examples of prominent plants in the world (Allman et al., 2017; Zamfirescu, et al., 2017; Brightling, 2018).

Methods of producing NH_3 by reforming methane are steam reforming, partial oxidation, autothermal reforming, dry reforming and the electrolysis of water (Rice & Mann, 2007). SMR of hydrocarbons to carbon monoxide (CO), CO_2 and H_2 for NH_3 production was introduced in 1930 where the choice of feedstock for the reforming process largely depends on its location, availability, and the local energy policy (Bhaumik et al., 2002; Quon, 2012; Ramos & Zeppieri, 2013). Advantages of the method are namely, simplicity, lower cost, its environmentally friendliness, high conversion efficiency, low process operation temperature, high feedstock hydrogen-to-carbon ratio and its widespread utilization (Bhaumik et al., 2002; Vezina, et al., 2017). Industrial scale production of NH_3 using this technique comprises of six main interconnected stages; namely desulphurization, steam reforming of natural gas (methane, CH_4), shift conversion, CO_2 removal (Chaudhary et al., 2017), methanation and NH_3 synthesis. Two reformers called the primary and secondary reformers are employed during NH_3 production and have been used practically in Camargo City Chihuahua State, Mexico and the Billingham NH_3 factory in the 1920-1930s (Brightling, 2018; Flores et al., 1997). In the primary reformer, naphtha is cracked into C and H by heating the fluid over a catalyst loaded tube, given that the reformer makes exhaustive use of heaters (Bhaumik et al., 2002; Flores et al., 1997). To reduce the energy being consumed, a reformer-exchanger system can be used after which its effluent is passed to a secondary reformer, where N_2 for the synthesis is added through a pressurized preheated process air (Cremer, 1980; Flores et al., 1997; Ruddock et al., 2003). Vezina, et al. (2017) compared 15 different methods of generating NH_3 in their work. Several others try to simulate an NH_3 plant of varying capacities using ASPEN Plus, ASPEN Hysys and Industry Design Softwares at steady state mode, making several assumptions (Abdel El Moneim et al., 2018, 2020; Azarhoosh et al., 2016; Chidozie & Koyejo, 2021; Islam et al., 2010; Nwanam et al., 2020; Sulaikha & Soloman, 2021; Tripodi et al., 2018). Previously, a calcium-copper process and process condensate stripper had been integrated into NH_3 plant production (Baboo, 2022a; Martinez et al., 2017). These modifications or innovations are centered towards achieving a state-or-the-art facility with advanced technology (Levy, 1989; Shannahan, 2000). After commissioning, various power source are used during NH_3 plant start-up and operation, including wind, solar and hydrothermal power electricity source (Chun & Barton, 1999; Habermehl & Gill, 2015; Moffatt & Sridharan, 2002; Verleysen et al., 2020; Yuksel et al., 2022). Baboo (2022c) states that, no power plant is 100% efficient in converting fuel chemical energy to electrical energy capable of performing valuable task and that the optimum theoretical efficiency of most fossil fuel power stations is around 64%.

Methodology

Materials

Aspen Plus version 8.4 was used for the modeling, simulation and control of the process units. Microsoft Visio 2016 Professional (PC) was used to draw the process flow diagram as well as the instrumentation and control diagram. Windows 10 Pro Portable Laptop Computer (DESKTOP-8GVIVAF), with installed RAM of 4GB, 64-bit operating system, and an Intel(R) Celeron(R) CPU processor containing the listed softwares was used throughout the work. Simple basic numerical computations were carried out using Porpo Programmable Calculator. The process layout and other graphical representations were developed using Wondershare EdrawMax version 11.0.

Process Description

NH_3 production process was divided into three major categories: (1) preparation of raw synthesis gas following the steps beginning with desulphurization of natural gas, primary reforming and secondary reforming, (2) raw synthesis gas purification section including high temperature shift (HTS) conversion, low temperature shift (LTS) conversion, CO_2 removal and methanation sections and, (3) synthesis and refrigeration section including NH_3 conversion and NH_3 separation and storage which is described as the heart of the process by Azarhoosh et al. (2016). Main steps followed for NH_3 process simulation using ASPEN Plus is shown in Figure 1 (Islam et al., 2010; Usmonovich & Elmurodugli, 2022).

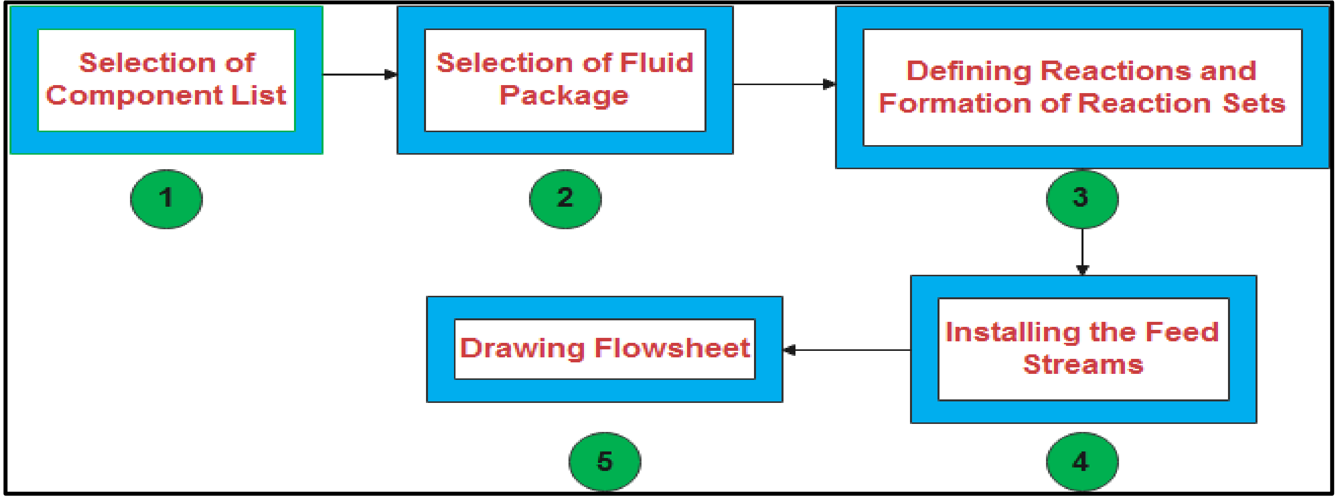


Figure 1: Ammonia Process Simulation Steps Using ASPEN Plus

Input Stream Fractions Specifications

6800 kmol/h of natural gas with component compositions in mole fractions (Table 2) was used in the feed stream. This value was realized based on pre-estimation of the targeted output of NH_3 – put at 200 tonnes/day, even though any amount of the product realized at the end of the process can be scaled-up or scaled-down to pre-set value.

Table 2: Inlet Specification

Components (i)	Molecular Weight (MW)	Mass fraction (x)	Mole fraction (y)
CO_2	44	0.02	0.005
H_2	2	0.1	0.511
N_2	28	0.126	0.047
CH_4	16	0.6	0.3875
Ar	40	0.002	0.0005
S	32	0.153	0.049

The molar specifications in Table 2 wasn't taken from any country's natural gas constituent molar fractions, as different natural gas producing company across the world have specific gas compositions of the natural gas they produce (Denys & Vries, 2013; Kidnay et al., 2015). Together with molar feed flow rates, the compositions were initialized in ASPEN plus. For the feed stream as well as in subsequent calculations for other streams, molar flows of each specie, $\dot{n}_i$ were calculated using Equation (1):

$$y_i = \frac{\dot{n}_i}{\dot{n}_T} \quad \text{OR} \quad \dot{n}_i = y_i \dot{n}_T \qquad (1)$$

Mass flows ($\dot{m}_i$) of all gases was computed using Equation (2) from which the mass fractions (x) were determined by substituting in Equation (3):

$$\dot{m}_i = \frac{\dot{n}_i}{MW_i} \qquad (2)$$

$$x_i = \frac{\dot{m}_i}{\dot{m}_T} \qquad (3)$$

where, subscript i = component gas, $\dot{n}_i$ and $\dot{m}_i$ = molar and mass flows of components i, respectively and $\dot{n}_T = \sum \dot{n}_i$ and $\dot{m}_T = \sum \dot{m}_i$ = total molar and mass flows in the stream.

Natural Gas Desulphurization

Natural gas was preheated in a pre-heater **E-01** to 350-400°C from the well sources. It was then compressed in a compressor **C-04** at a pressure of about 43.5 atm, before it was sent through stream **002** to the desulphurizer, **R-01** for the removal of impurity where sulphur (S) compounds are hydrogenated to hydrogen sulphide (H_2S). Cobalt molybdenum catalyst was used in Reaction 4 in 99% conversion of S.

$$H_2S + ZnO \rightarrow ZnS + H_2 \qquad (4)$$

The mixture was further sent to the separator unit **S-02** were the H_2S is removed (no H_2S in stream **043**). At this point, the S content was removed to < 0.1 ppm in the gas feed and the ZnO remains in the absorption bed.

Primary Reforming

At this stage, process gas from the desulfurizer passes through stream **043** to a mixer **M-01**, which is mixed with incoming steam in stream **004**. Stream **004** is 100% steam (hot H2O) injected at a flow rate of 100 kg/h. The mixture was heated to further 500-600°C in a heater **E-02** before it goes to the primary reformer unit **R-02** containing nickel catalyst. In the primary reformer, the heat supplied was a result of burning natural gas or other gaseous fuels in the fired heater **E-03**. How to select the best fuel that would results in optimal production was previously explained by Nie (1995). Amount of natural gas (fuel) supplied initially per hour was approximately 10 kg. The flue gas leaving the fired heater **E-03** was at a temperature of 900°C, and steam/carbon ratio is 3.0. It is worthy pf note that the stage is highly endothermic. The reformer was operating at a temperature of about 880°C and a pressure of about 25atm. Governing reaction in this unit is given in Reaction (5) and (6), as taken from literature.

$$CH_4 + H_2O \rightleftharpoons CO + 3H_2 \qquad \Delta H°_{298} = 206\ \mathrm{kJ.mol} \qquad (5)$$

$$CO + H_2O \rightleftharpoons CO_2 + H_2 \qquad \Delta H° = -41\ \mathrm{kJ.mol} \qquad (6)$$

Equilibrium constants for steam-methane reforming process for the two reactions as obtained from (Abbas, Dupont, & Mahmud, 2016) are as follows for the above reactions: $k_{eq,4.2} = e^{\frac{-35735.2}{T}+30.114} = \frac{y_{CO} \cdot {y_{H_2}}^3}{y_{CH_4} \cdot y_{H_2O}}$ and $k_{eq,4.3} = e^{\frac{4400}{T}-4.1027} = \frac{y_{CO_2} \cdot y_{H_2}}{y_{CO} \cdot y_{H_2O}}$ where T implies temperature in Kelvin. Majumdar & Mukherjee (1988) wrote on the influence of steam injection in primary reformer to the energy efficiency of the overall plant. Failure analysis of NH_3 plant primary waste heat boiler and energy-intensive design of the SMR furnace were previously carried out by Ardy et al. (2021) and Zecevic (2021) respectively.

Secondary Reforming

This reformer was simulated based on method described by Yu et al. (2006).Synthesized gas from stream **009** was cooled to 735 °C in a cooler **E-04**. It was mixed with compressed air (50 kg) from compressor **C-01** at a pressure of 38 atm in mixer (**M-04**). Normally, air is a mixture of 21% O_2, 78% N_2 and 1% argon, and though not present in stream **010**; amount of air in stream **012** is equal to air in stream **042**. The mixed stream flows down to the secondary reformer **R-03** (Yu, 2002), where as usual the reaction at that stage was considered highly exothermic, operating at 880°C. Ratio of the natural gas (mainly CH_4) to that of steam must be less than 0.5 to prevent coking in the pre-heater. Equilibrium reaction (7) and (8) governing this unit was entered in ASPEN Plus.

$$CO + H_2O \rightleftharpoons CO_2 + H_2 \qquad (7)$$

$$H_2O + CH_4 \rightleftharpoons CO + 3H_2 \qquad (8)$$

Catalyst used at this point was pure iron, even though there are several other catalysts (e.g. nickel oxide) that can be separately evaluated for efficient performance of the secondary reformer (Al-Dhfeery & Jassem, 2012). Water, CO and CO_2 must be removed from the syngas through stream **014** to prevent oxidation of the iron. It is worthy of note that, appropriate design and operating principles must be followed as secondary reformers may sometimes fail, catch fire or explode (Lestari et al., 2019; Mittal & Arvindakshan, 1994; Taghipour et al., 2021).

High Temperature Shift (HTS) Conversion

Synthesis gas from stream **013** usually contains poisonous gas like CO which is poisonous to catalyst. Incoming syngas was cooled to 400°C in a waste steam cooler **E-05**. Process gas from the secondary reformer contains 12-15% of CO, most of which is converted in the high temperature shift reactor **R-04**, consisting of bed of iron/chromium oxide catalyst through which the gas passes through. Reaction occurs at a temperature of 400°C where CO is reduced to 3%. Shift conversion was basically used to condition the syngas to desired ratio and the HTS conversion reaction is given by Reaction (9).

$$CO + H_2O \rightleftharpoons CO_2 + H_2 \qquad (9)$$

Low Temperature Shift (LTS) Conversion

Gas from the HTS converter **R-04** was cooled to 200-220°C, in a cooler **E-06** and then passed through stream **016** to the LTS converter **R-05**. LTS converter operates at a temperature of 200 °C, which was filled with zinc-oxide based catalysts. Optional stream might contain most of S from stream **016** (say 98% of S) together with negligible amount of H_2O (say 0.07%). Water (100kg/h equivalent to 100%) from the exchanger **E-07** was mixed and sent back to the HTS converter **R-04.** In mixer, **M-02**, stream **037** is a recycle stream of solely water (100kg/h = 5.556 kmol/h). Reaction 10 is the LTS conversion reaction as specified in ASPEN Plus.

$$\mathrm{CO} + \mathrm{H_2O} \rightleftharpoons \mathrm{CO_2} + \mathrm{H_2} \qquad (10)$$

Absorption/Regeneration Unit

Synthesis gas from the LTS converter (consisting mainly of H_2, N_2 and CO_2) passes through stream **017** to an exchanger unit **E-07** which cools the gas before sending it to the absorber column **CLM-01** through stream **018**, were the CO_2 is dissolved and sent to the generation column **S-03**. Afterwards, the CO_2 is recovered for other industrial process (including sales). Basic solvent used for CO_2 absorption is the aqueous amine solution, Mono-Ethanolamine (MEA). In the solvent storage tank, **T-01**, MEA absorbing capacity of CO_2 is 4.123 mol% (CO_2 **:** MEA = 0.04123). All CO_2 entering **CLM-01** through stream **018** was absorbed. MEA solution was pumped to the column **CLM-01** through stream **034**. The absorber overhead (stream **022**) was assumed to having no trace of CO_2 while 70% of the solvent (MEA) leaves same stream. Stream **019** is completely MEA and CO_2. A 100% pure CO_2 and MEA-OUT of unit **S-03** was targeted.

Methanation and Ammonia Synthesis

After CO_2 absorption, there exist little amount of CO and CO_2 in the synthesis gas passing through stream **022** from the column absorber **CLM-01** which are also considered to be poisonous for the NH_3 synthesis catalyst, and has to be removed by converting the CO and CO_2 to CH_4. The conversion is done in the methanation reactor **R-06** via Reaction (11) and (12) (Asante et al., 2022).

$$\mathrm{CO} + 3\mathrm{H_2} \rightarrow \mathrm{CH_4} + \mathrm{H_2O} \qquad (11)$$

$$\mathrm{CO_2} + 4\mathrm{H_2} \rightarrow \mathrm{CH_4} + 2\mathrm{H_2O} \qquad (12)$$

Equilibrium constant for reactions 11 and 12 was taken as $k_{eq,4.8} = 0.0007349$ and $k_{eq,4.9} = -0.5855$ respectively. The reactions took place at around 300°C were the reactor **R-06** is filled with nickel catalysts. Gas from the methanator **R-06** was cooled in a cooler **E-08** and then compressed in a single stage compressor **C-03** before channeling to the ammonia converter **R-07**. Syngas from the NH_3 reactor was cooled to 30°C in a cooler **E-09**. Waste gases were purged through a purge stream. Remaining constituents were sent to the separator **S-01** through stream **030** were the unreacted gas are recycled back to mixer **M-03** through stream **031** leaving the NH_3 gas as the output product from stream **033**. It was expected that the recycle stream **031** contains 0.0156% CO_2, 41.68% H_2, 0.174% N_2, 50.69% CH_4, 0.00675% Ar, 0.00127% S, 0.652% H_2O, 6.702% MEA and 0.0331% air in mole basis equal to a mass flow of 161687.942 kg/h in that stream.

Ammonia Synthesis Reaction

Synthesis of NH_3 (in Reaction 13) takes place on an iron catalyst at a pressure usually in the range 100-250 bar and a temperature of 350 °C (El-Gharbawy et al., 2021), even though previously, Liu et al. (2020) developed and applied a new catalyst to synthesize NH_3. Previously, Al-Malah et al. (2018) developed a successful simulation of an NH_3 synthesis process that generates NH_3 at low temperature and pressure using Aspen. Amount of CO_2 and CO coming to stream **028** was reduced to percentages; say 85.11% and 83.9% respectively.

$$\mathrm{N_2} + 3\mathrm{H_2} \rightleftharpoons 2\mathrm{NH_3} \qquad \Delta\mathrm{H} = -46\mathrm{kJ.mol^{-1}} \qquad (13)$$

It was assumed that in the splitter **SPLT**, (1): 2% of CO_2 and CO in stream **030** are purged and (2): 70% of H_2, and NH_3; 58.56% of N_2; 69.81% of CH_4; 24.85% of argon; 69.82% of S and H_2O; 69.81% of MEA and 69.77% of air in stream **030** is contained in stream **041**. Previously, researchers has demonstrated the possibility of removing/recovering/recycling H_2, N_2 and NH_3 from ammonia plant waste stream or purge gases in order to increase the plant's capacity (Rahimpour & Asgari, 2009; Rahimpur & Asgari, 2008; Roos et al., 2001; Safari et al., 2021). Unburnt hydrocarbons, SO_2, C (flyash, particles) in boilers, CO, NOx and CO_2 are the main pollutants (by-products) from NH_3 plants (Baboo, 2022c, 2022b). In separator **S-01**, most of the components are recycled as stream **031** (fresh feed to **M-03**) excluding NH_3.

Summary of the units involved, their code/number and their representative streams is presented in Table 3.

Table 3: Nomenclature of Units Employed and Their Streams Constituents

S/No.	Units	Operation	Input Stream(s)	Output Stream(s)
1.	E-01	Natural Gas Preheater	Natural Gas	039
2.	C-04	Natural Gas Compressor	039: Preheated NG	002: Compressed NG
3.	R-01	Desulphurizer	002	003: Desulphurised NG
4.	S-02	H_2S Removal	003	043: Sweetened NG H2S: Removed

5.	M-01	Steam & Syngas Mixer	043 004: Steam Addition	005: Mixed Steam + Syngas
6.	E-02	Steam & Syngas Pre-heater	005	006: Preheated 005 Stream
7.	E-03	Fired Heater	Fuel	008
8.	R-02	Primary Reformer	006 008: Combusted NG Fuel	009: Syngas Generated
9.	E-04	Primary Reformer Cooler	009	010: Cooled Syngas
10.	C-01	Air Compressor	Air	012: Compressed Air
11.	M-04	Air & Syngas Mixer	010 012	042: Air & Syngas Mixing
12.	R-03	Secondary Reformer	042	013: More Syngas Generation/CH_4 Reduction
13.	E-05	Waste Steam Boiler	013	014: Syngas Cooling
14.	M-02	Syngas & Water Mixer	014 037: Recycled Water	038: Mixed Syngas & Water
15.	R-04	High Temperature Shift Converter	038	015: Reduced CO
16.	E-06	HTS Cooler	015	016: Cooled 015
17.	R-05	Low Temperature Shift Converter	016	017: Reduced CO
18.	E-07	Syngas Heat Exchanger	017 007: Water Addition	018: Mainly H_2, N_2 and CO_2 Product 037
19.	T-01	MEA Tank	-	034: MEA supply
20.	CLM-01	CO_2 Absorber	018 034	022: Highly H_2 and N_2 Product 019: MEA + CO_2 Stream
21.	C-02	Syngas Compressor 1	022	023: Heated 022
22.	S-03	CO_2 Removal	019	021: CO_2 Removal OPT
23.	R-06	Methanation	023: Highly H_2 and N_2 Stream	024: Highly CH_4, H_2 & N_2 Product
24.	M-03	Natural Gas & Recycled Syngas Mixer	024 031: Unreacted Gas Recycle	040: Mixed 024 & 031
25.	E-08	Syngas Condenser	040	025: Heated Mixture
26.	C-03	Syngas Compressor 3	025	027: Compressed Syngas
27.	R-07	Ammonia Conversion	027	028: Highly NH_3 Output
28.	E-09	Syngas Cooler	028	030: Cooled Product
29.	SPLT	CO_2 & CO Purger	030	Purge: CO_2 and CO 041: Remaining Composition
30.	S-01	Ammonia Separation	041	031 033: NH_3 Product

Material and Energy Balance

Using appropriate energy balance relations (Cremer, 1980; Ghasem & Henda, 2015), the heat flows in and out of all streams was computed. In addition, mass and mole balances as well as heat balances over all units and the entire process was carried out using auxiliary information of the process description, as well as equations from Ghasem & Henda (2015). Important calculations done here also follows a detailed material balance calculations presented for an ammonia plant design by Baniya (2021).

Process Optimization, Instrumentation and Control

Flexible ASPEN Plus features were utilized to optimize the recovery of NH_3 and other intermediate products. Optimization (Anon, 1987) was carried out on four important units which are the **R-01**, **R-03**, **R-04**, **R-06** and **R-07**. Equipment used for the NH_3 production process by steam reforming were heat exchangers, reactors, mixers, separators, storage tank, compressors and absorbers, most of which requires control of their process conditions. Key control parameters identified were temperature, methane slippage, H/N ratio, steam/carbon ratio and pressure. Kinds of controllers suitable for the common variables are PI (for flow and liquid pressure), P (for gas pressure), P or PI (for liquid level), PID (for temperature) and P, PI & PID (for composition), where P, I and D implies proportional, integral and differential controllers.

Plant Layout

A simple layout showing the plant area, utilities, canteen, future expansion and roads and car parking areas was developed.

Results and Discussion

The flow diagram containing results of heat and energy balances (Cremer, 1980; Graeve, 1981) carried out on all units of the plant after interpreting the process described above was produced. Figure 2 was produced using Microsoft Visio. It depicts material and heat flows from every stream as well as their process conditions.

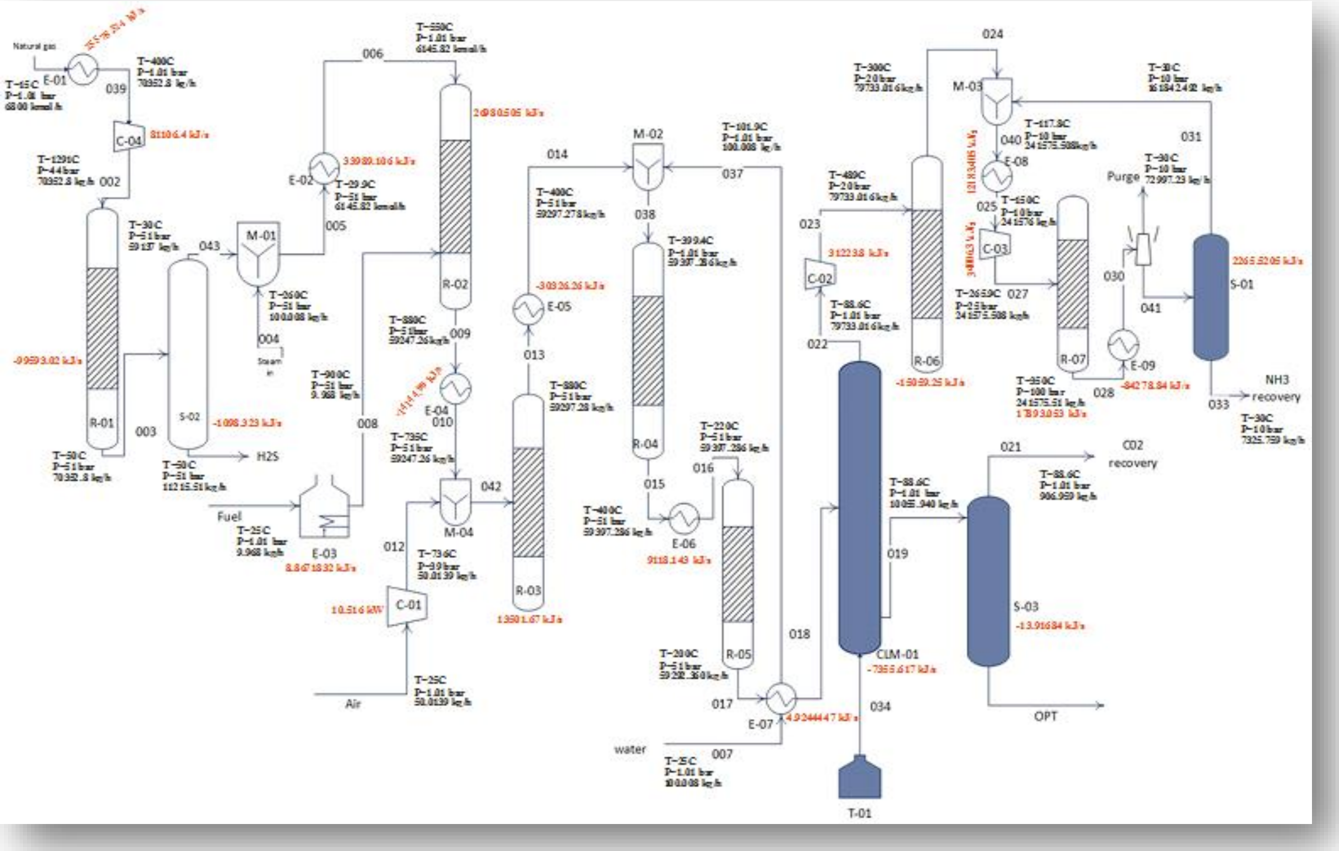


Figure 2: Process Flow Diagram of the Ammonia Plant

It was simulated using ASPEN Plus V.8.4 following some techniques used by Nwanam et al. (2020) during the simulation of the Indorama ammonia plant, which resulted in the output mass and heat flows shown in Figure 2. Output of NH_3 from **S-01** product stream was 7325.759 kg (430.927 kmol) which was then scaled-up to 200 tons/day using a scale-up factor of 1.1375, as small sized plants could be scaled-up to a large size one when necessary (Axelrod, 2006; Ramos & Zeppieri, 2013; Sanchez & Martin, 2018; Vrijenhoef, 2017) reported that there is currently in existence, about 105 large-scale plants ranging from 600-1800tpd in practically every corner of the world, which is apparently close to this capacity. According to Panjeshahi et al. (2008), the recovery of fairly small amount of heat has the ability to accrue into a sizeable energy savings, as NH_3 manufacture is voted as an energy intensive technique. For all heat exchangers present, the second law via Pinch Technology gives the thermal interactions between the chemical process and utility systems that surrounds them to improve utility consumption (Ozturk & Dincer, 2021;

Tavares et al., 2013). Lababidi et al. (2000) and Lundgren (2016) carried out a detailed energy integration study using Pinch Technology on an NH_3 plant configurations prior to final detailed simulation and optimization. To verify that the minimal requirements of utilities, heat transfer area, and total cost are met, a retrofitting analysis of an existing network of chemical plants can be conducted (Chavda, 2019). Hanada et al. (2010) findings show that, if natural gas is used as feedstock to produce NH_3, 28.5 MJ/kg NH_3 (based on lower heating value) would be consumed. How energy in an NH_3 plant can be calculated using designed equations is detailed by Baboo & Manager (2015).

Optimization Output

In the literature, Flórez-Orrego & Junior (2017) carried out the modelling and optimization of an NH_3 synthesis plant. Using the same idea, 200 tons capacity NH_3 plant was optimized for maximum productivity and efficiency. Firstly, S content in **R-01** was minimized at the outlet stream thereby increasing the yield of H_2S. To ensure that S is minimized, sensitivity analysis was carried to obtain the variables that may likely affect the flowrate of the outlet stream. From Figure 3, it was observed that the output molar flow of H_2S increased with an increase in fractional conversion, obviously asserting the fact that, the yield of H_2S is sensitive to its corresponding fractional conversion, even though there are other variables that may likely have effect on its production. Therefore, the optimum conversion that optimizes the yield of H_2S is obviously 0.999.

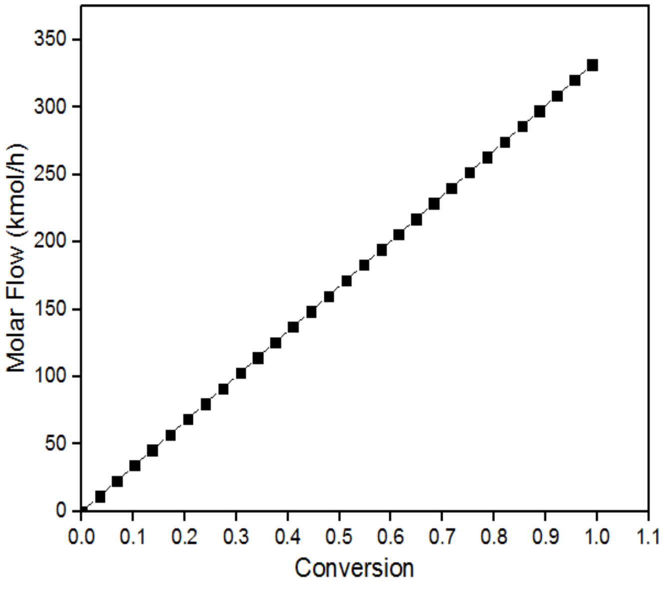


Figure 3: Effect of Conversion on Sulphur Minimization in R-01

Yield of H_2 can be maximize in reactor **R-03**. Sensitivity analysis carried out to identify the process variable capable of affecting the process unit using ASPEN plus software, shows that temperature has drastic effect on the yield of H_2 production. From the analysis, a throughput temperature gave an increment of 3% to 10% of H_2 in the outlet stream, thereby asserting the optimum value for the production of hydrogen to be 1200℃. Figure 4 illustrates this analysis.

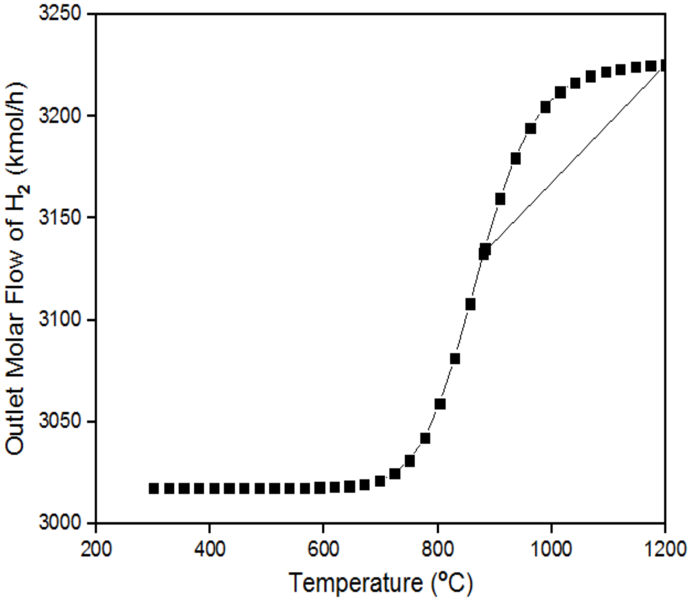


Figure 4: Temperature Effect on Hydrogen Yield

Meanwhile, sensitivity analysis carried on reactor **R-04** shows that for every decrement in the reactor temperature there is an increment in the outlet flowrate of CO_2 (Ali et al., 2010). It proves the fact that, the optimum temperature capable of maximizing the yield of CO_2 is found within temperatures of lower limit (20℃) rather than having a high temperature for the equilibrium reaction taking place in the reactor, as shown in Figure 5.

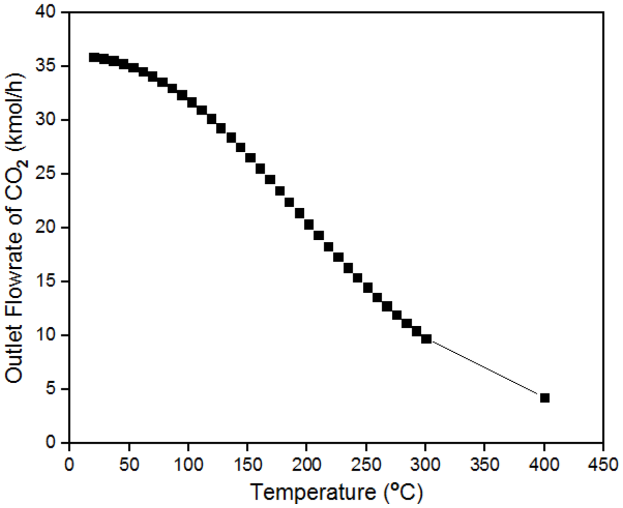


Figure 5: Effect of Temperature on CO_2 Production in R-04

Also, CH_4 can be maximized by minimizing CO in the outlet stream of **R-06**. Sensitivity analysis carried on **R-06** points to an increase in temperature as responsible for a proportional increase in the molar flow rate of CO. Consequently, optimum temperature required to use up CO, lies within the lowest temperature limit (like 250°C); and emphasizes a decrement of about 10-15% of CO in the outlet stream of the reactor, as clearly depicted in Figure 6.

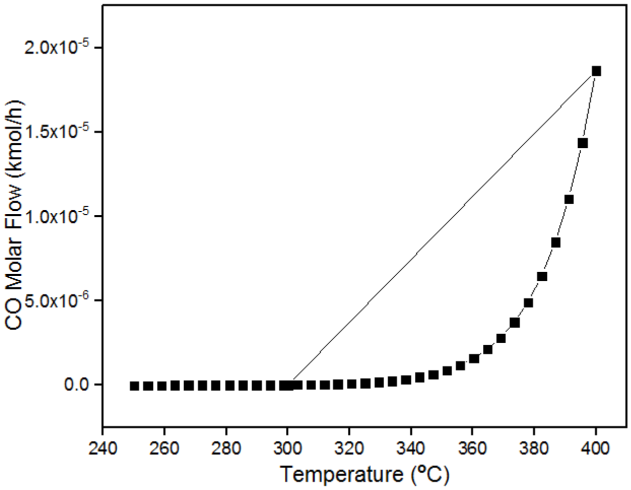


Figure 6: Influence of Temperature on CO Yield

Foremost product of the plant, NH_3, can also be maximized by testing for temperature sensitivity in the reacting system of **R-07**, in accordance with Ivanov (2017) and Bland (2015). From the analysis, there is a 10-12% increment in NH_3 yield with decrease in temperature. Thus, the decision variables are meritoriously the operating temperature of the reactor; and the optimum temperature lies within the possible temperature limit of the reactor (230.589°C), which is very obvious in Figure 7.

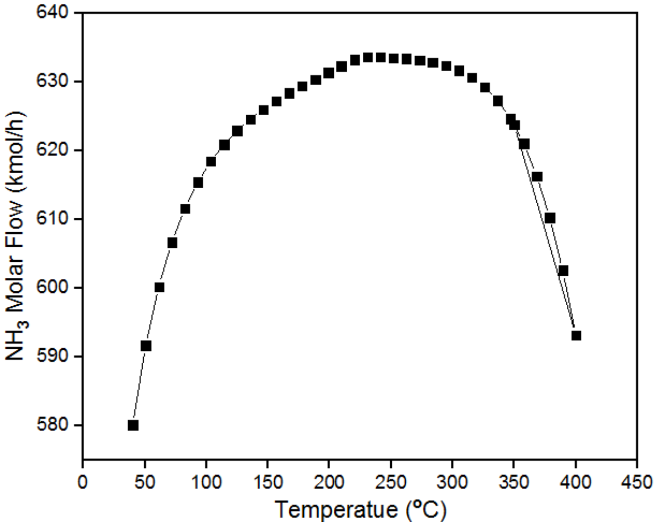


Figure 7: Illustrating the Effect of Temperature on NH_3 End-product

Previously, Tripodi et al. (2018) simulates NH_3 manufacture over Ru/C catalyst, Azarhoosh et al. (2016) uses Genetic Algorithm to simulate a horizontal reactor for NH_3 synthesis, Chehade & Dincer (2021) as well as Lundgren (2016) modeled kinetically, a small modular NH_3 unit, while Chidozie & Koyejo (2021) follows a step-wise approach that involves data collection and ASPEN Hysys simulation of an NH_3 plant front-end waste heat boilers.

Process Control

Control in process industries refers to the regulation of all aspects of the process. In real chemical plants, steady-state doesn't exist. Things are always changing. Process control is concerned with making sure that processes perform their tasks in a safe and economical way (Frahm et al., 2001; Grasdal et al., 1998). Process control technology is the tool that enables manufacturers to keep their operations running within specified limits and to set more precise limits to maximize profitability, ensure quality, minimize risk and ensure safety (Novandhini et al., 2020; Ojha & Dhiman, 2010). Control performance depends on the quality of the used Instrumentation and Control (I&C) infrastructure (i.e. sensors, control system hardware and actuators) using the Distributed Control System (DCS) originally designed for process plants (Araujo & Skogestad, 2008; Borikar, 2018; Dziuba et al., 2020). The whole process I&C diagram is as shown in Figure 8.

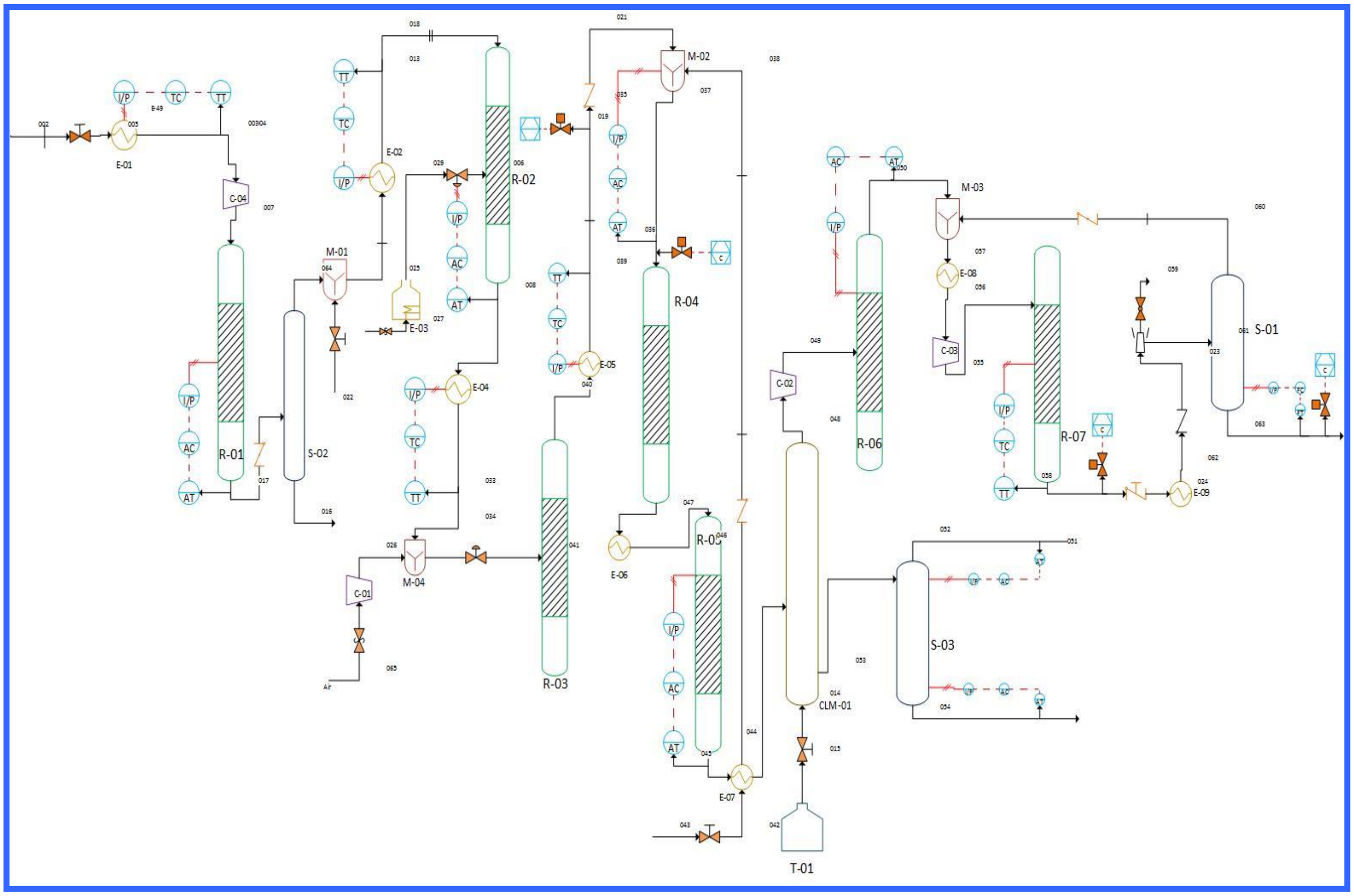


Figure 8: Piping and Instrumentation Diagram

Sulaikha & Soloman (2021) collectively carry out the control of reactor, level, separator and pressure in their work using almost the same approach described in this work. The control objective of **R-02** is to maintain a steam/carbon ratio of 3:1 using compositions of H_2O and CH_4 as control variables. Steam and natural gas reacts in the primary reformer to form H_2 and CO ($CH_4 + H_2O \rightarrow 3H_2 + CO$). Some of the CO will react with the steam to form even more H_2 ($CO + H_2O \rightarrow CO_2 + H_2$). Thus, steam-to-carbon gas ratio requires tight control because energy is wasted when excess steam is produced unnecessarily. In addition, excess CH_4 requires more energy for compression and causes inefficient catalyst activity (Dark & Stallworthy, 1985). A minimum ratio of 3 kmol steam to 1 kmol of carbon, i.e. 3:1 should be maintained. Tight control of the steam to carbon ratio can significantly decrease production costs. The first reaction is endothermic, where an increase in temperature favors formation of the product, which is essential. Decrease in temperature will result in generation of high amount of reactant (steam and CH_4), which is an undesired increase. Malhotra-Kbr et al. (2004) reported an increase in capacity from 1000 mtpd to 1070 mtpd in NH_3 production, despite reduction in natural gas capacity of a primary reformer at Shenzhen Liaohe Tongda Chemicals Company Limited Ammonia Plant, Panjin City, China, when the unit was replaced. Replacement is inevitable in case of reformer tube failure (Bhaumik et al., 2002; Boumaza & El Ketroussi, 1996). The control objective of **R-03** is to increase the conversion of CH_4 by decreasing temperature. It is obvious that only 30-40% of the hydrocarbon is usually converted in the primary reformer. The exhaust is therefore fed into a secondary reformer where the conversion to CO and H_2 continues. It is important to minimize the amount of unreacted CH_4, or methane slippage, from the secondary reformer. If it is not minimized, CH_4 builds up in the NH_3 converter loop which can only be corrected by increasing the conversion of CH_4 to syngas. It is an equilibrium endothermic reaction. Increase in temperature favors the formation of the product (syngas) while a decrease in temperature favors backward reaction (i.e., CH_4 formation). Inlet temperature of 735 ℃ can be reduced to 690 ℃ (setpoint) by adding cooling water.

Control objectives of **R-04** and **R-05** entails the removal of all CO from the process before it enters the NH_3 converter, or catalyst poisoning will occur. This removal takes place by 'shifting' the CO to CO_2 after which the CO_2 is absorbed (Mahmoodi & Darvishi, 2017). This shift occurs in two steps during high and low temperature shifts (Agnesty et al., 2020). If the CO is not properly removed, it can shift back to CH_4, creating a highly exothermic reaction that can damage the next process stage; called the methanator (**R-06**) as clearly exemplified by Alhabdan & Elnashaie (1995).

HTS reaction is exothermic – decrease in temperature will maximize the shifting of CO to CO_2 (according to Le Chatelier Principle) or reduce the amount of CO to a setpoint between 0.1–0.5% concentrations; likewise, in LTS reactor. **R-06** is designed to remove any residual CO and CO_2. Syngas output from **R-06** should be ideally comprised of 75% H_2 and 25% N_2. Production of NH_3 takes place in the converter (**R-07**), vital in the maintenance of the ratio of H_2 and N_2 as close as possible to the stoichiometric ratio of 3:1. **R-07** is governed by the Haber-Bosch reaction 13, where the forward reaction results in a decrease in number of moles, hence decrease in pressure and the backward reaction results in an increase in number of moles hence increase in pressure. According to Le Chatelier's principle, if a high pressure is applied to an equilibrium system, the reaction which involves a reduction in pressure is favored. Conversely, if low pressure is imposed on an equilibrium system, then the reaction which results in an increase in pressure is favored. In the Haber process, forward reaction is exothermic; decreasing the temperature will give a high yield of NH_3. A temperature of 250°C give better yield of NH_3, but is not economically feasible as it takes too much time for the reaction system to attain equilibrium – because the reaction rate decreases as temperature decreases.

Measured variable for a heat exchanger is the temperature of the outlet stream. So, the set points for all eight heat exchangers contained in this design are 400°C **E-01**, 550°C **E-02**, 735°C **E-04**, 400°C **E-05**, 220°C **E-06**, 199.8°C **E-07**, 150 °C **E-08** and 350 °C **E-09**. Even at desired operating conditions, corrosion effects of carbon steels in NH_3 plants heat exchangers must be checked regularly, as they may be caused by the operating temperature, mechanical factors and the corrosive media (Nikitasari et al., 2020; Prifiharni et al., 2020). A separator works on the principle of 'different component having different boiling point'. The most volatile component is collected at the top with low boiling points. Higher boiling point fluids are collected at the bottoms. This control setup aims at making the top and bottom outlet temperatures of the separator be at a set point. Hence, the control variable is temperature of the outlet streams. At the top, the outlet temperature is measured by the temperature sensor transmitter, it sends an electronic signal to the controller which has a setpoint of T_{set} = 88.6°C for separator **S-03**; 30 °C for separator **S-01** and **S-02**. Temperature controller (TC) then, calculates the error signal and sends an output called controller output to current-to-pressure (I/P) transducer to be implemented by the control valve. The same happens at the bottom of the column where their respective setpoints are 30°C for **S-01**, 50°C for **S-02** and 88.6°C for **S-03**.

In all, the controller compares the measured value to the desired value (set point) and calculates an appropriate output signal that is sent to an I/P transducer where it is converted to an equivalent pneumatic (air) signal that is compatible with the control value. As for the mixers, the proportional-integral-derivative (PID) controller is chosen because of its robustness and simplicity in tuning parameters. The process variable or measured variable (MV) is the mixture quality (q), measured by the composition analyzer-transmitter (AT). The mixture quality is defined as the average output concentration of the mixture. The set point is SP as specified by the control engineer. The composition controller (AC) sends a signal, p(t) of the manipulated variable known as controller output to the mixer tank. Disturbance input is the input mass flowrate fluctuations. Absorption is simply the transfer of material from a gas (absorbate) to a liquid (absorbent). Also known as "scrubbing" or "washing". Transfer is based on the preferential solubility of a gaseous component in the liquid. The manipulated, measured, control variables and set points are column inlet flowrate, 450 kmol/h, flowrate and 499.9 kmol/h. In the control loop, FT implies flow transmitter. It senses the flow into the column and measures it. FC is short for feed rate controller. It receives signal from FT, compares it with the given set point and make appropriate corrections to it after which it sends a signal to I/P. I/P then responds by sending pneumatic signal to the final control element for appropriate action.

Plant Layout

Normally, producing a design of any process plant is a highly intricate and demanding task (Peters et al., 2003; Srisukh, 1976; Talarico & Scotto, 2012). Constituting the process layout is crucial part of plant design and one of the most central tasks prior to the plant construction as described by Rahman et al. (2014). Figure 9 depicts the location of the plant area, security units, utilities and other essential units of the designed NH_3 plant.

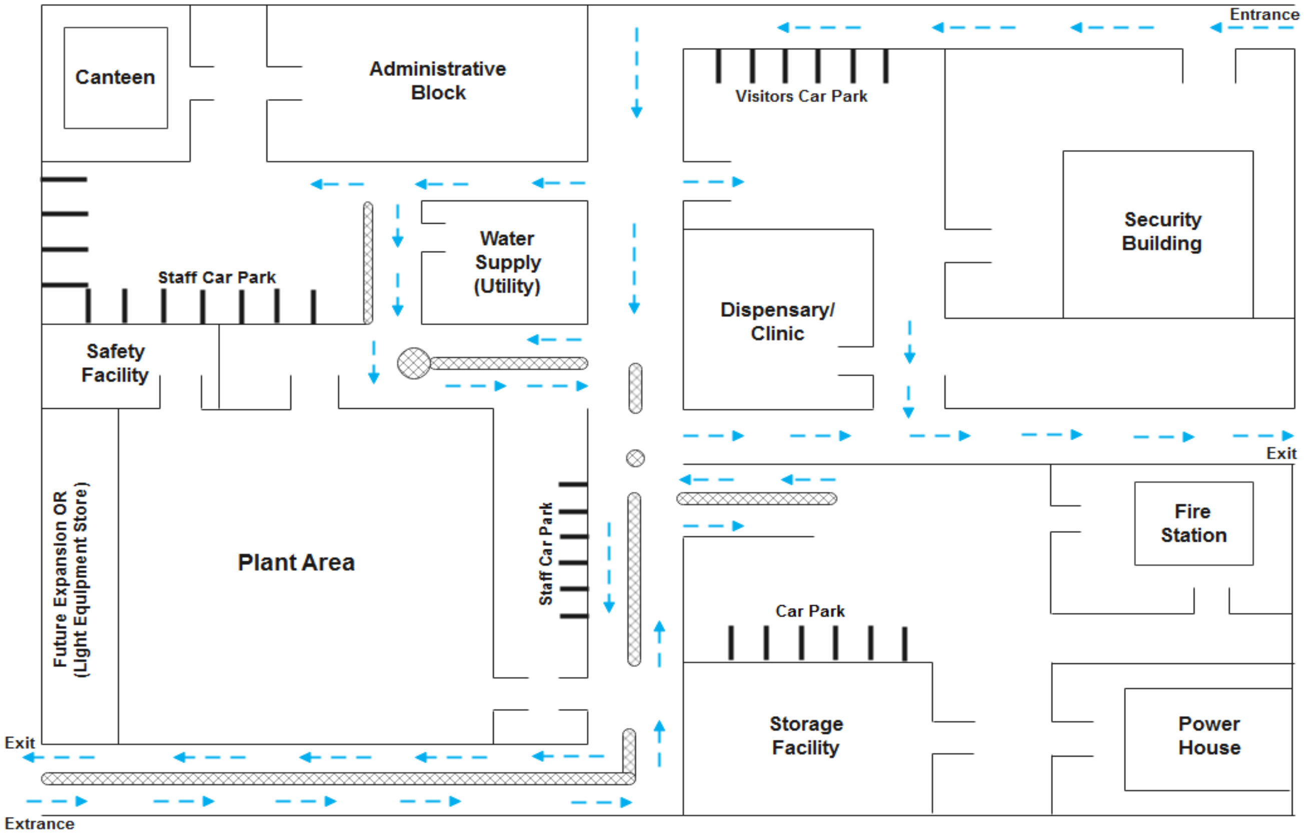


Figure 9: Proposed Layout for the 200 TPD Ammonia Plant

For example, Rahman et al. (2014) produces the layout of an existing NH_3 plant of Karnaphuli Fertilizer Company Ltd (KAFCO) at Rangadia, Karnaphuli, Chittagong-4000, Bangladesh, occupying a total processing area of 123m×106m = 13,038 m^2. Also, a plant layout by Baniya (2021) for an ammonia plant considers siting the pipe bridges, loading and off-loading facilities, plant roads, future expansion, store, workshops, fire and safety department, plant utilities, emergency water, process plant, laboratory, stray yard, canteen, packaging plant and the administrative block at strategic points suitable for personnel and materials transition in and out of the plant. Looking at various structures involved, plant layout design is a multidisciplinary task that needs partnership among different experts (Lira-Flores et al., 2019; Rahman et al., 2014). And according to Syeda et al. (2017), every failure has certain repercussions and effects on the surrounding region. Understanding the potential effects of these failures on the nearby facilities and their occupants is essential to designing a layout with the least amount of risk.

Conclusion

It is clear that the aforementioned objectives have been successfully achieved. Units **R-01**, **R-03**, **R-04**, **R-05** and **R-07** that are sensitive to some process variables were identified to find the optimum by analyzing the variable over an inequality constraint. Adequate controls of process variable have been carried out. Most important for an NH_3 plant are methane slippage, steam-to-carbon ratio, temperature, hydrogen-to-nitrogen ratio and S composition. All these variables were seriously emphasized in the process units and specified set points adequate for optimum operation of the units. With safety in every engineer's mind, this work encourages the adherence to safety measures that would prevent leakages as well as ensure safety of personnel and environment. Based on several available feedstock and energy sources present, different energy sources can be explored to find out the most convenient and efficient for NH_3 production, even though natural gas is one of the best alternatives for the supply of energy, currently. Ammonia can be made from many different energy sources, which could help stabilize the NH_3 price by allowing multiple technologies to compete for the lowest cost form of NH_3 production. Further research should be carried out on other NH_3 production techniques in order to reduce their cost.

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