DESIGN OF MICROREACTOR FOR AMMONIA SYNTHESIS

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ELECTRICAL & ELECTRONICS ENGINEERING

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By

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Submitted to the Electrical & Electronics Engineering Programme
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CERTIFICATION OF APPROVAL

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TRONOH, PERAK

JUNE 2009

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

IZZAH AMANI BT. TARMIZI

ABSTRACT

The main concern of this project is to come out with a new approach of designing a microreactor which is specialized for ammonia synthesis. In designing this microreactor, it needs to consider several factors such as suitability of materials to fabricate it, basic components inside, relevant temperature and pressure required, stability and so on. As reactor used nowadays in industry in a large size of plant, require high cost in production and maintenance, thus this project major focused on scale it down which will be optimized the cost as well as easy to handle. Designing the microreactor should pass through several stages and needs to make more justifications to choose the best one and economically save. Autocad drawing has been applied in designing this microreactor as a way of engineering perspective to get the exact imaginations in all angles before proceed the actual fabrication. Several testings also being carried out to find the temperature as well as pressure for the gas inside the chamber and in estimating expected value of ammonia production. A new design concept of microreactor within the magnetic field is successfully invented which has been optimized for the cost, reduced maintenance, less complexity as well as easy to handle which is absolutely less time-consuming and also economically save.

ACKNOWLEDGEMENT

I would like to express my gratitude to all who helped me throughout this project. First of all, I would like to thank to my supervisor, Associate Professor Dr. Noorhana bt Yahya, who gives me greatest ideas as well as continuous support in completing my final year project throughout this two final semesters. Another appreciation goes to all the technicians of Universiti Teknologi PETRONAS for their help, contributions in all aspects including ideas, brainstorming as well as much effort in ensuring this project successfully being done by using facilities provided by university. Thank you very much to those who make contributions in this project.

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INTRODUCTION

1.1 Background of Study

This project study is intended to develop microreactor design precisely for ammonia synthesis. Basic concept in designing such a microreactor has been studied and must possess necessary equipments and elements to allow a chemical reaction occurred inside a microreactor in a high-pressurized within specified range of temperature. Apart from that, specification of materials and components also being investigated in depth to choose the best materials could be used in fabricating this microreactor.

Besides, the chemical part; characteristic of both gases and reaction between hydrogen gas and nitrogen gas before forming ammonia also being studied to thoroughly understand the process flow involved in producing the ammonia. The design of this microreactor and materials used must be very comprehensive in order to achieve the quality of efficiency besides enable the chemical reaction successfully occurred inside it.

1.2 Problem Statement

The importance of ammonia cannot be denied any more due to its benefits in many aspects as well as its advantages in oil & gas industry as tabulated in the table below [4]:

Table 1: Ammonia Advantages

Industry	Use
Fertilser	production of: • ammonium sulfate, $(NH_4)_2SO_4$ • ammonium phosphate, $(NH_4)_3PO_4$ • ammonium nitrate, NH_4NO_3 • urea, $(NH_2)_2CO$,also used in the production of barbiturates (sedatives), is made by the reaction of ammonia with carbon dioxide $\underset{\text{carbon dioxide}}{CO_2} + \underset{\text{ammonia}}{2NH_3} \rightleftharpoons \underset{\text{ammonium carbonate}}{H_2NCOONH_4} \xrightarrow{\text{heat, pressure}} \underset{\text{urea}}{(NH_2)_2C}$
Chemicals	synthesis of: • nitric acid, HNO_3, which is used in making explosives such as TNT (2,4,6-trinitrotoluene), nitroglycerine which is also used as a vasodilator (a substance that dilates blood vessels) and PETN (pentaerythritol nitrate). • sodium hydrogen carbonate (sodium bicarbonate), $NaHCO_3$ • sodium carbonate, Na_2CO_3 • hydrogen cyanide (hydrocyanic acid), HCN • hydrazine, N_2H_4 (used in rocket propulsion systems)
Explosives	• ammonium nitrate, NH_4NO_3
Fibres & Plastics	• nylon, $-[(CH_2)_4-CO-NH-(CH_2)_6-NH-CO]-$,and other polyamides
Refrigeration	• used for making ice, large scale refrigeration plants, air-conditioning units in buildings and plants
Pharmaceuticals	• used in the manufacture of drugs such as sulfonamide which inhibit the growth and multiplication of bacteria that require *p*-aminobenzoic acid (PABA) for the biosynthesis of folic acids, anti-malarials and vitamins such as the B vitamins nicotinamide (niacinamide) and thiamine.
Pulp & Paper	• ammonium hydrogen sulfite, NH_4HSO_3, enables some hardwoods to be used
Mining & Metallurgy	• used in nitriding (bright annealing) steel, used in zinc and nickel extraction
Cleaning	• ammonia in solution is used as a cleaning agent such as in 'cloudy ammonia'

Ammonia is a compound with the formula NH_3. It is normally encountered as a gas with a characteristic pungent odor. Yet industry right now used too huge technology system in a large ammonia plant to synthesis the ammonia which requires high cost maintenance and also quite a number of manpower to handle and manage that system. The chemical reaction to produce ammonia is also quite slow and very time-consuming. According to the industrial person outside, the ammonia yields in the plant nowadays is just less than 20%, and normally 17% to 18% of production only. Thus, this project intends to increase the ammonia production by redesign the microreactor to become a comprehensive system which is smaller in size compared to the real microreactor used in industry. Apart from that, synthesis process of ammonia is expected to be increased by the effect of magnetic field as well as by using nano catalyst in size. Several designs have been proposed to justify which one the best as well as able to be fabricated within the time frame fixed (2 semesters). The design of microreactor has been renovated and subject to change from time to time for quality purposes until the actual fabrication.

1.3 Objectives of Project

1. To produce suggested design of microreactor specialized for ammonia synthesis which optimized the cost and much smaller than in industry.
2. To introduce an idea designing microreactor within the specified range of magnetic field.

1.4 Scope of Study

The study on the design concept of microreactor system and implementation is to be completed within two phases. The scope for phase 1 of the project, which is research, collecting data related to this design and making decision on based of several designs or model implemented, are expected to be completed by semester one. The studied has been focused on how to come out with very precise and quality design of microreactor, suitability of tools and materials that are going to be used in this design and chemical reaction theoretical knowledge of ammonia synthesis. Phase 2 which is real implementation and fabrication of microreactor will be started after phase 1 completed.

CHAPTER 2
LITERATURE REVIEW

2.1 Hardware Design & Reliability

Below will be explained some of the design concept as well as tools and equipments needed in designing microreactor.

2.1.1 Microreactor Basic Design Concept

As focused in this project, microreactor which intended to design must be suit within the specified dimension of magnet due to our objectives which is needs to design the microreactor that is fit in a range of magnetic field to see the difference in ammonia production when applying magnetic field effect. The range of magnet measured is about 8.0 cm. Thus, the microreactor designed must be smaller in size than the range of magnet for it to fit in. It must possess several basic tools for the gas reaction to occur while ammonia synthesis is being carried out soon for instance gas tube, air tube, heating element, thermocouple and pressure meter [3].

2.1.2 Gas tube

Gas tube will act as a medium to allow the flow of nitrogen gas and hydrogen gas into the inlet of the chamber [2]. The type of it must be properly chosen to suit with the characteristic of both gases to avoid any leakage or damage that might be happen. It is recommended to be connected to the gas mixer to speed up the gas flow into the chamber regardless of the air resistance [3].

2.1.3 *Air tube*

Air tube required to allow air from outside or cooling system device to prevent over pressurized as well as overheating. One end will be placed outside the chamber and the other end will be connected to the air cooling device to ensure the temperature of the reaction will be maintained as well as to avoid any leakage or equipments damaged that might be happened due to the hot surrounding [2].

2.1.4 *Heating element*

The heating element will be heated to provide the temperature until achieved at about 250°C or above since the reaction of nitrogen gas and hydrogen gas to produce the ammonia just can occur in a high pressurized and temperature condition. In order to meet this requirement, the heating element must possessed melting point more than 250°C and high thermal conductivity that can be bear with high temperature and pressure [2]. Below is shown some types of heating elements that could be used in designing such of this microreactor [10].

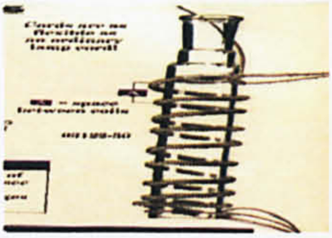


Figure 1: Spiral type

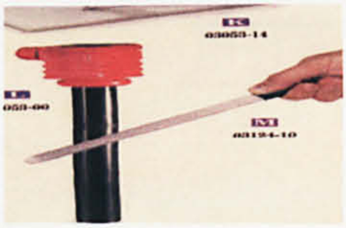


Figure 2: Long type

Figure 1 showed the wrapped heating element around the chamber while figure 2 showed long type of heating element. Both of these heating elements have maximum heat transfer up to about 454°C which is suitable to be used in this synthesis since ammonia synthesis desired temperature need to achieve in this project is only 250°C [10].

2.1.5 Magnet

Magnet might be put at the side of chamber and it is suggested to use here due to its ability in providing magnetic field. As known, magnet consists of two poles; north and south which will attract something that oppose of the poles. Theoretically, magnetic field produced is believed can align the positive and negative charges of hydrogen and nitrogen atoms during the reaction process. This alignment will ease the combination of nitrogen (N_2) and hydrogen (H_2) atom to form ammonia [12], [13].

2.1.6 Thermocouple

Thermocouple is used as a sensor for measuring temperature during the reaction takes place. It consists of two dissimilar metals, joined together at one end. When the junction of the two metals is heated or cooled, a voltage is produced that can be correlated back to the temperature. The thermocouple that is going to be used in this project is type K since type K (chromel-alumel) is the most commonly used and it is a general purpose thermocouple. It is also inexpensive, easy to obtained and available in a wide variety of probes. It can be bear in the −200 °C to +1250 °C temperature range which is meet this temperature requirement [2]. Other than that, there is also a thermocouple which being heated, and able to produce reading via digital sensor, which called temperature device sensor.

2.1.7 Pressure Meter

Pressure meter in microreactor is necessary to record the current pressure while reaction occurred to ensure that it will not goes beyond the limit value. There are pressure meter implemented in digital reading which somehow can record both temperature and pressure of the reaction process. Independent pressure meter which can just only monitor and display the pressure manually of the system also could be used due to the low cost and easy maintenance [10].

2.2 Characteristics Of Materials

A thorough study and research has been done to understand the characteristic of materials in order to find the best and suitable material used to fabricate the microreactor. The choice of materials for high pressure applications depends on a number of different factors. Consideration should be given to the working conditions such as pressure and temperature range, pressure and temperature cycling (fatigue life), corrosion and finally economic factor [6].

Materials for use in high-pressure equipment subjected to tensile stresses should never be brittle. As a general rule, the longitudinal elongation must be between 18% and 20%, whereas the transverse elongation should not be much under 12-15%. Brittle materials should be avoided wherever possible [6].

Since the ammonia synthesis need to be done in a high temperature and pressure, thus the chamber of the reactor at which the reaction takes place must be made of metal which posses higher thermal conductivity and able to bear with temperature at about 450°C - 500°C at 200 atm (after reduced by catalyst) [4].

The metal suitable to build the microreactor is being investigated to ensure that it could be bear with the high presurrized and extremely hot condition. Among of those which possessed this characteristic are aluminum, copper and stainless steel.

Aluminium has a light weight with a density of $2.7x10^3$ kg/m^3 is approximately 35% that of steel. Commercially pure aluminium has a tensile strength of approximately 90MPa and can be improved to around 180MPa by cold working. Aluminium also has a good resistance to corrosion as it possessed an internal element which is thin oxide [6].

Thin oxide film which forms protects the metal from further oxidation, unless exposed to some substance or condition which destroys this protective coating the metal remains protected from corrosion. Other than that, aluminium electrical conductivity grade is approximately 27mΩ/cm and high reflectivity. These reflectivity characteristics lead to its use as an insulating material and in this case, prevented the equipments from damaged due to hot temperature caused by the heat from ammonia reaction. In addition, aluminium also has a quite high melting point which is 660.25 °C (933.40 K) [9].

Copper is a ductile metal with very high thermal and electrical conductivity. Its density is 8.94 $g{\cdot}cm^{-3}$, thermal conductivity at (300 K) 401 $W{\cdot}m^{-1}{\cdot}K^{-1}$and has high melting point which is 1084.62 °C, (1357.77 K). Yet, copper is very expensive in price and quite difficult to have it [8].

Like stainless steel, it does not stain, corrode, or rust as easily as ordinary steel and possessed high melting point, which is 1857°C [8], [14]. Stainless steel differs from carbon steel by the amount of chromium present. Carbon steel rusts when exposed to air and moisture. This iron oxide film is active and accelerates corrosion by forming more iron oxide. Stainless steel has sufficient amount of chromium present so that a passive film of chromium oxide forms which prevents surface corrosion and blocks corrosion from spreading into the metal's internal structure [14].

2.3 Chemical Reaction

In this part, it will touch about catalyst explanation and ammonia process based on Haber experiment.

2.3.1 Catalyst

Catalyst suggested to be used in this ammonia synthesis is nano particle size. It is believed can enhance and speed up the chemical reaction. Besides, it is also made the reaction possible to operate at lower temperature and at the lower pressure as well. There are several types of nano catalyst that could be used to activate NH3 synthesis at atmospheric pressure for instance Ruthenium (Ru), iron (Fe), magnetite (Fe3O4). The most stable and commercially available Ru precursor used for ammonia synthesis is $RuCl_3$ [7], [1], [6]. Yet, since magnetite catalyst is already available, so, this project might proceed with using Magnetite catalyst during the experimental testing soon or with some of other catalysts to see the difference in desired ammonia production in order to find the best one to be constantly used.

2.3.2 Ammonia synthesis

The Haber process is an important process involved for the manufacture of ammonia. It looks at the effect of temperature, pressure and catalyst on the composition of the equilibrium mixture, the rate of the reaction and the economics of the process. Haber process stated that the reaction between one mole nitrogen and three moles hydrogen will produced ammonia gas which is a reversible reaction and the production of ammonia is exothermic, releasing 92.4kJ/mol of energy at 298K (25°C) [4]. In commercial synthesis systems, various ammonia synthesis catalysts are utilized in fixed beds through which the hydrogen and nitrogen reactants are flowered. The unreacted hydrogen and nitrogen are recycled after ammonia is recovered from the product effluent gas, such contaminants and inerts tend to build up in the system or "synthesis loop".

$$\underset{\text{nitrogen}}{N_2(g)} + \underset{\text{hydrogen}}{3H_2(g)} \xrightleftharpoons{\text{heat, pressure, catalyst}} \underset{\text{ammonia}}{2NH_3(g)} \quad \triangle H = -92.4 \text{ kJ mol}^{-1}$$

Or

$$\underset{\text{nitrogen}}{N_2(g)} + \underset{\text{hydrogen}}{3H_2(g)} \xrightleftharpoons{\text{heat, pressure, catalyst}} \underset{\text{ammonia}}{2NH_3(g)} \quad + 92.4 \text{ kJ mol}^{-1}$$

A flow scheme for the Haber process looks like this:

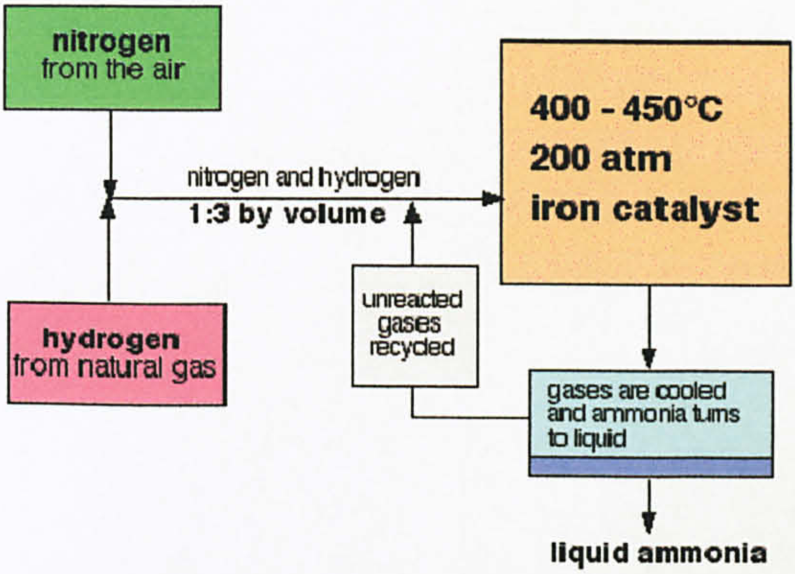


Figure 3: Haber Process [4]

At each pass of the gases through the reactor, only about 15% of the nitrogen and hydrogen converts to ammonia. Under the reaction condition of 400 to 450°C temperature, the ammonia is successfully produced as desired but in a small amount compared to the ammonia production after feedback the unreacted gas. By continual recycling of the unreacted nitrogen and hydrogen, the overall conversion is about 98% based on this Haber's process testing [4].

Contaminants or inert gases in the hydrogen and nitrogen containing feed gas, if present in excessive amounts, may adversely affect catalyst activity or dilute the reaction mass sufficiently to lower the kinetics and yield of product ammonia. Where the unreacted hydrogen and nitrogen are recycled after ammonia is recovered from the effluent gas, such contaminants and inerts tend to build up in the system or synthesis loop. If the mol percent of inerts in the feed is decreased from 0 to 10 percent, with the reaction being carried out at 838°F and 200 atmospheres pressure, the mol percent yield of ammonia is decreased from 27.8 to 22.7 percent.

Another factor is the pressure at which the reaction is carried out, increases in pressure while maintaining reaction temperature constant increase the equilibrium yield of ammonia. It is can be seen that from the research, at a temperature of 838°F, increasing reaction pressure from 100 to 500 atmospheres increases the percent yield of ammonia from 13.6 to 40.3 percent. On the other hand, if pressure is maintained at a uniform value while temperature is increased, the equilibrium yield of ammonia is reduced. For instance, at a pressure of 200 atmospheres, increasing the reaction temperature from 694°F to 968°F decreased the yield of ammonia from 38.7 to 13.2 percent. The decrease of equilibrium yield of ammonia with increasing temperature is exacerbated by the fact that the ammonia synthesis reaction is highly exothermic.

Finally, when the gases leave the reactor, they are hot and at a very high pressure. Ammonia is easily liquefied under pressure as long as it isn't too hot, and so the temperature of the mixture is lowered enough for the ammonia to turn to a liquid. The nitrogen and hydrogen remain as gases even under these high pressures, and can be recycled [4].

2.4 Magnetic Field Effect in Ammonia Synthesis

A new finding is to know the effects of magnetic field in ammonia synthesis and how efficient the production will be by applying this method.

2.4.1 Overview of magnetic field

A magnet produces a vector field, the magnetic field, at all points in the space around it. It can be defined by measuring the force the field exerts on a moving charged particle, such as an electron. The force (F) is equal to the charge (q) times the speed of the particle times the magnitude of the field (B), or F = q*v x B, where the direction of F is at right angles to both v and B as a result of the cross product. This defines the magnetic field's strength and direction at any point. A magnetic field can be created with moving charges, such as a current-carrying wire. A magnetic field can also be created by the spin magnetic dipole moment, and by the orbital magnetic dipole moment of an electron within an atom [9], [7].

2.4.2 Theoretical of magnetic field effects in ammonia synthesis

As being studied, a magnet produces a vector field, the magnetic field, at all points in the space around it. It can be defined by measuring the force the field exerts on a moving charged particle, such as an electron. The force (F) is equal to the charge (q) times the speed of the particle times the magnitude of the field (B), or simply **F=q*v x B**, where the direction of F is at right angles to both v and B as a result of the cross product.

This defines the magnetic field's strength and direction at any point. A magnetic field can be created with moving charges, such as a current-carrying wire. A magnetic field can also be created by the spin magnetic dipole moment, and by the orbital magnetic dipole moment of an electron within an atom.

A magnetic field will be applied to the reaction zone by means of a permanent magnet or preferably an electromagnet placed both at the side, outside of the reaction vessel (chamber) made of non magnetic steel material. The applied magnetic field is stated to help in lifting the fluidized catalyst particles, and to retain the particles in the reaction zone and not allow them to be removed by the flowing gas [7].

CHAPTER 3
METHODOLOGY

This methodology part will be divided into two which is in a flowchart form and followed by explanation in the second part.

3.1 Procedure Identification (Flowchart)

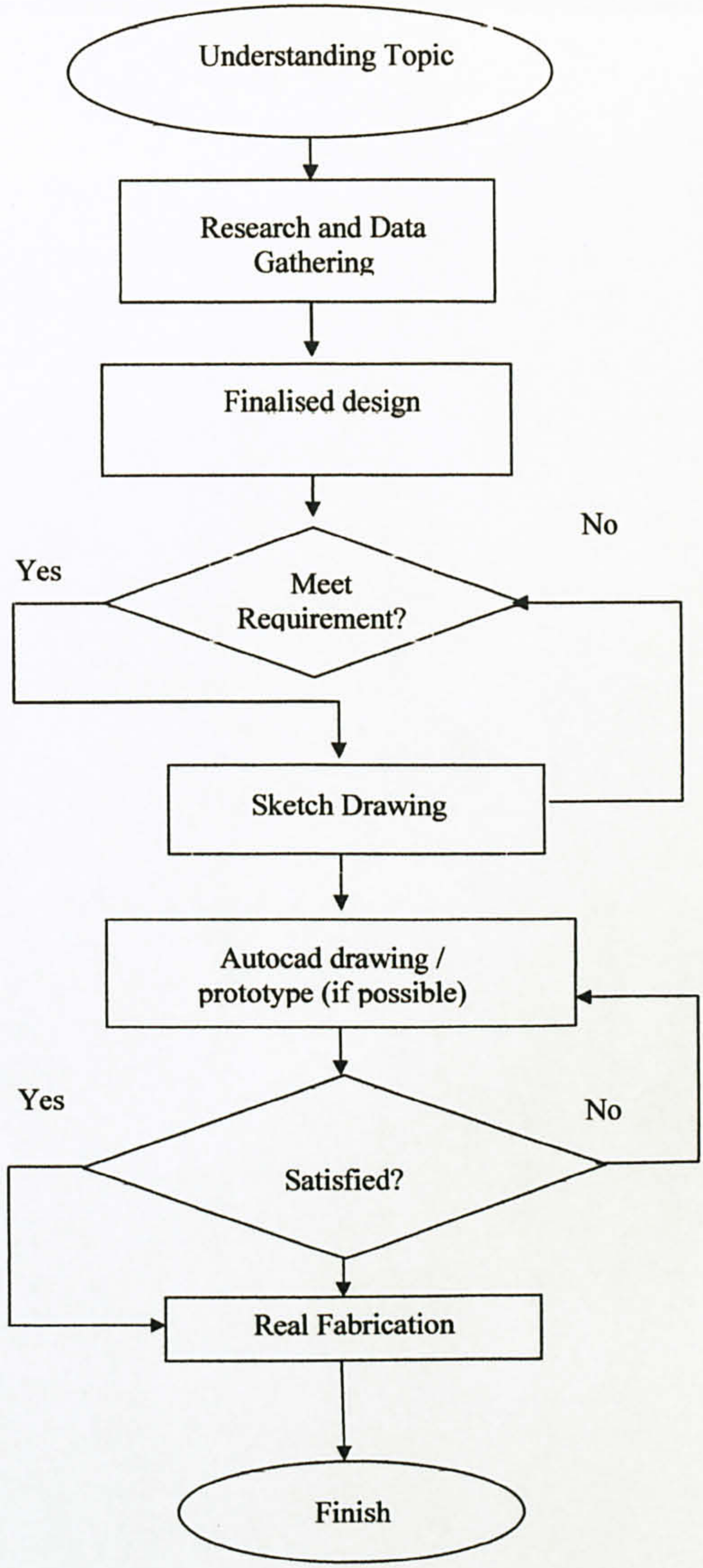


Figure 4: Flowchart

3.2 Procedure Explanation

There are some procedures to be followed in order to carry out and implement the design project. This is to ensure that the project can be accomplished within the given timeframe.

3.2.1 Data Research and Gathering

Elements of projects involved in this stage including research and study regarding the overview of microreactor, functionality, characteristic and chemical properties of materials, Haber process of ammonia synthesis and type of equipments and tools needed in designing such of this microreactor.

3.2.2 Design Implementation

Several designs have been proposed to justify and select the most comprehensive and efficient design to ensure the reaction is successfully occurred and should be able to produce the desired outcomes. Designs have been justified from various research and reasons after dealing with experts, technicians and other professionals. Besides, this design will be draw by using autocad software to clearly seen the figure from all angles before doing the actual fabrication. Designs of microreactor might be subject to change from time to time for quality and efficiency purposes and at the end, the best design will be finalized and fabricate by using prototype model first to ensure it will be working properly before doing the actual fabrication soon.

3.2.3 Sketch Drawing

Sketch drawing on paper project has been done to get the thorough understanding before proceed to the software based design and further fabrication.

3.2.4 Autocad drawing / prototype

After sketch drawing has been approved, software based approach which is autocad drawing is being done to see the overall figure from all angles and various views. Prototype model is also recommended to be constructed if time permitted to see the system's functionality or any leakage that might happen, so that the system could be rectified before doing the actual fabrication.

3.2.5 Actual Fabrication

Actual fabrication will be carried out basically based on the prototype model or approved autocad design. Actual fabrication for this microreactor has been done in cylindrical shape by using stainless steel material and some by using aluminium metal. Yet, during the actual fabrication, a little bit changes are might be occurred due to the difference between theoretical design and actual situation.

3.2.6 System Testing

Once the real fabrication has been completed, some other maintenance or any changes might have to be done in order to improve microreactor design performance. Final stage will be experimental testing to see to ensure the microreactor system could produce as per desired outcome (ammonia production).

3.3 Tools and Equipments Required

The tools and equipments needed to be used in fabricating the desired microreactor including heating element, thermocouple, pressure meter, temperature monitoring device and air tube which will be connect to the air cooling device.

CHAPTER 4
RESULTS AND DISCUSSION

4.1 Several proposed designs

Several designs of microreactor have been made and been updated from time to time to get some improvement before finalized to a final design.

- First design

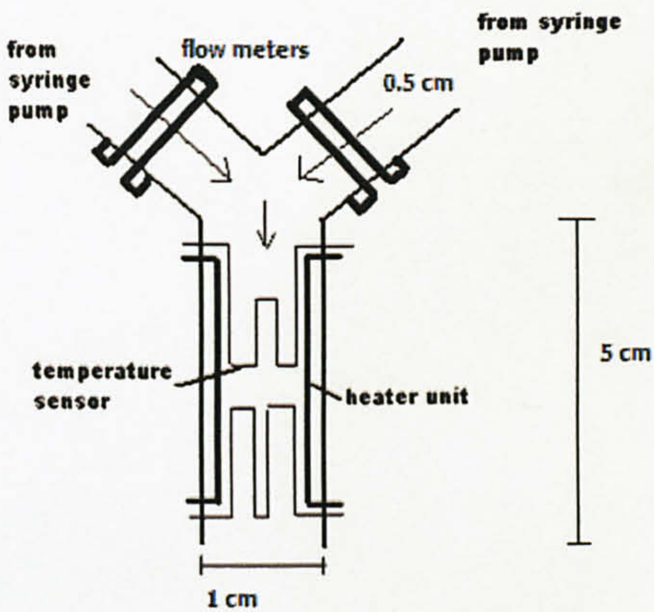


Figure 5: Y - shape

- Second design

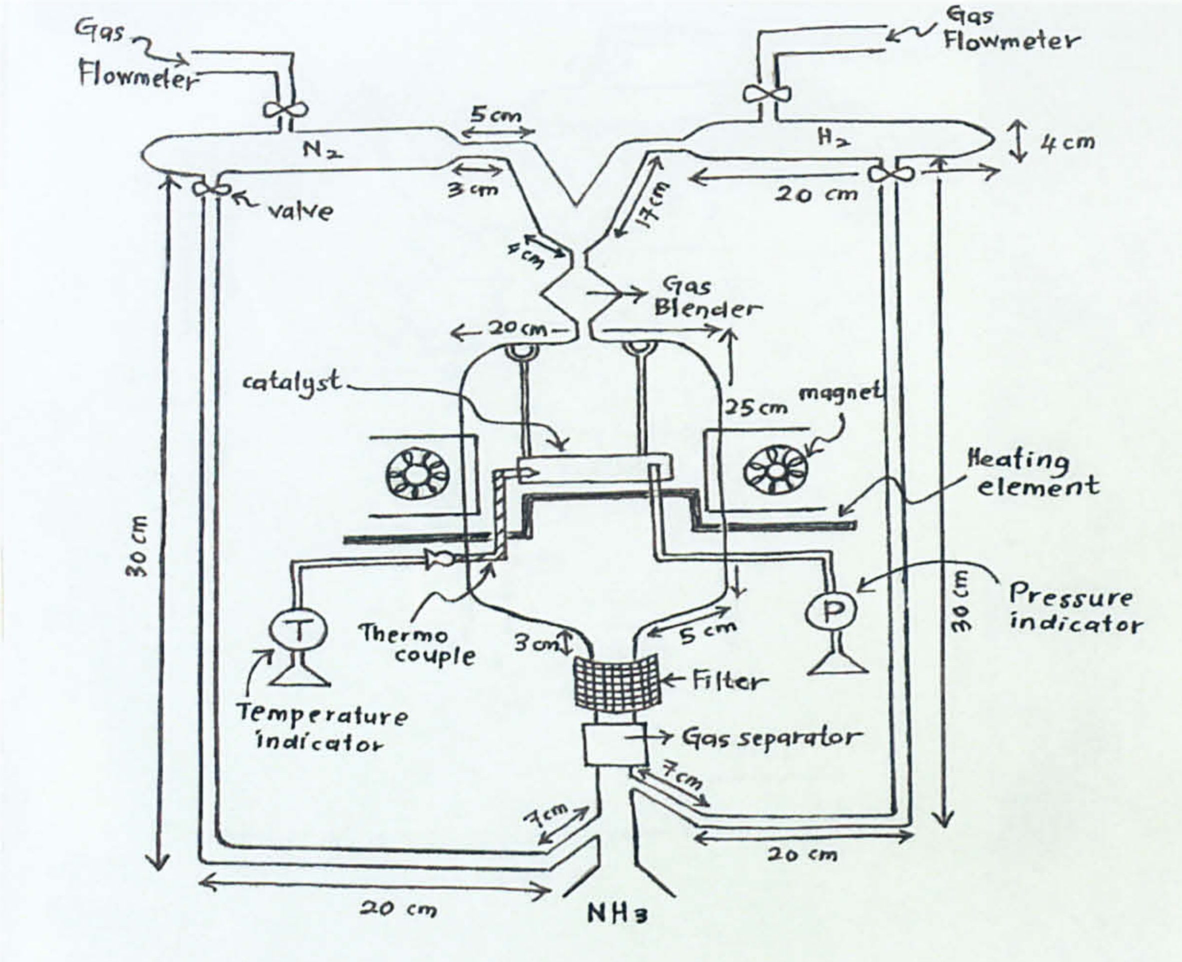


Figure 6: Second Design Approach

Since the second design seemed more complicated and involved several additional elements like gas flowmeter, gas blender and gas separator which are very costly, it is not practical to be fabricated. Modifacation then has been done to get the most comprehensive design.

- Third design

i. Sketch drawing

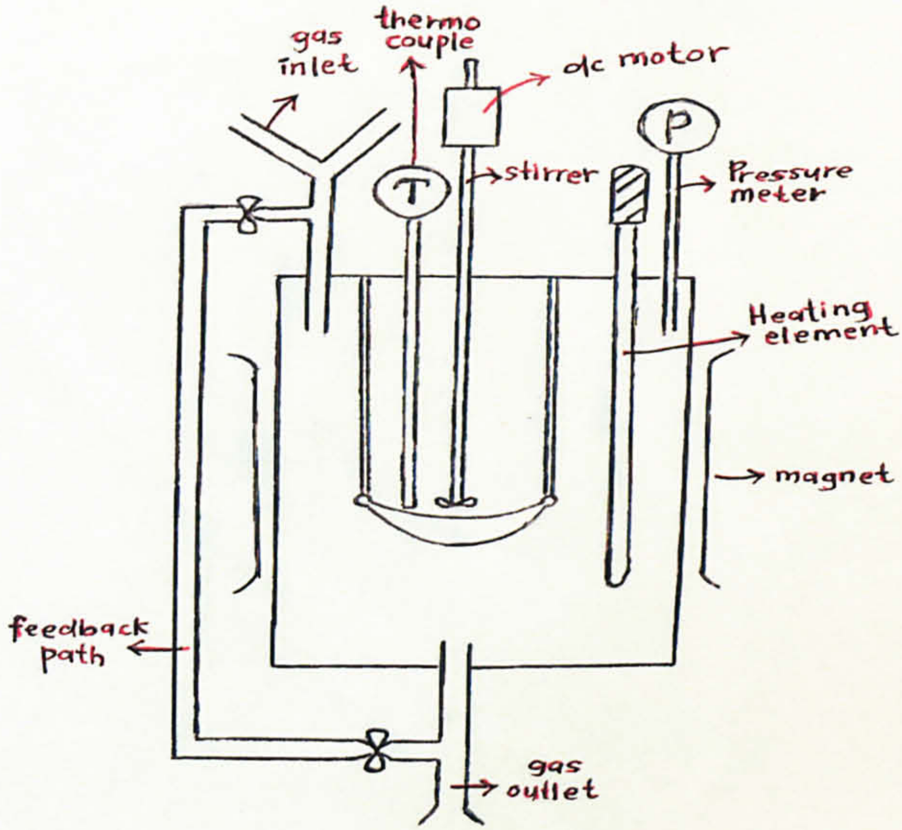


Figure 7: Sketch Drawing

ii. Autocad version

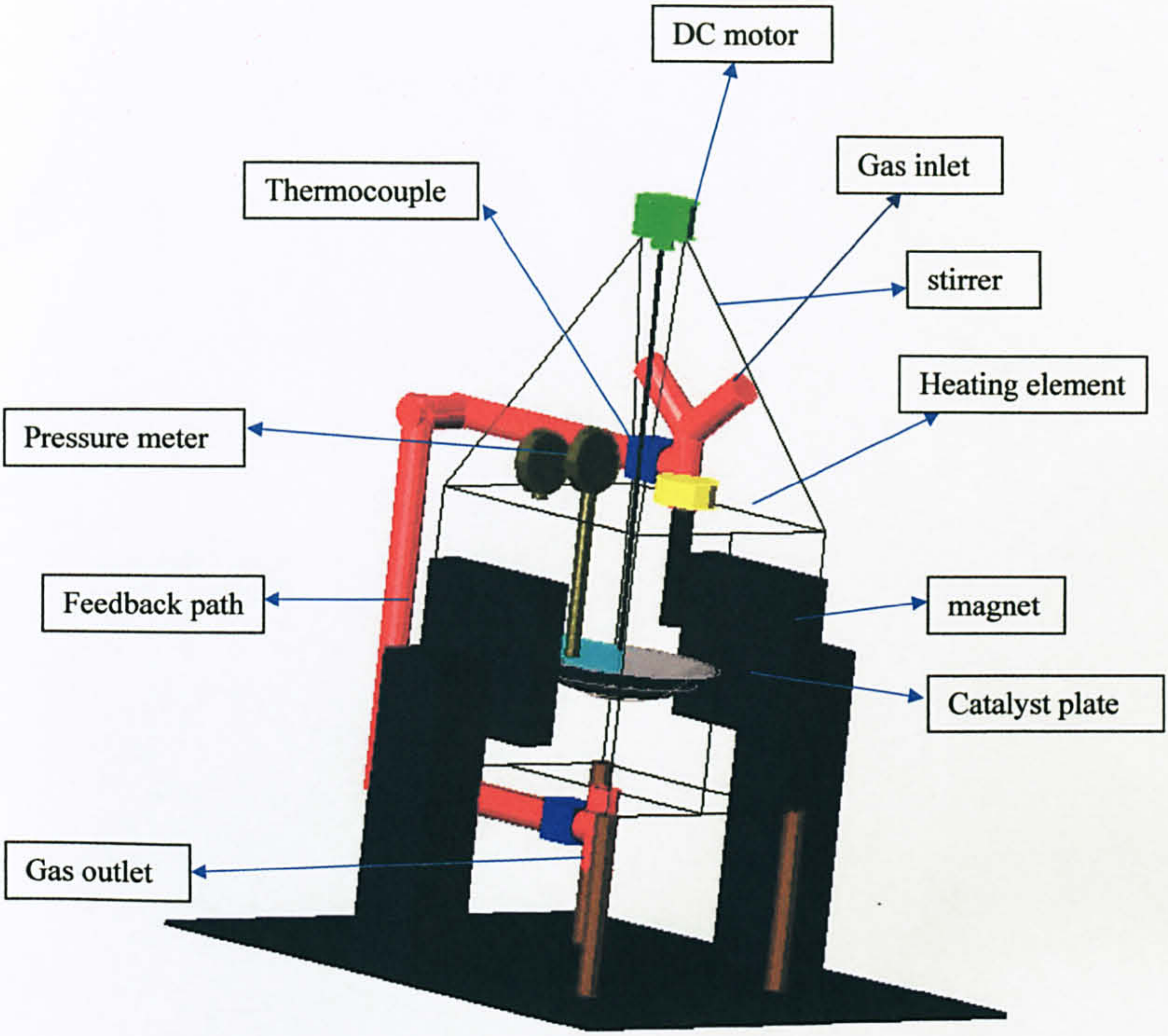


Figure 8: Autocad Version

This design is most preferable to choose due to its simplicity compared to the second design and most comprehensive. The prototype model will be fabricated based on this design. All sides of the square shape design are 7cm, the best measurement that can be suit within the range of magnet, which is 8.5cm.

4.2 Drawing of Microreactor Design in 3-D Views with Different Angles

Below is shown 3-D views with different angles of the latest and selected design of microreactor by using autocad drawing.

- Front view

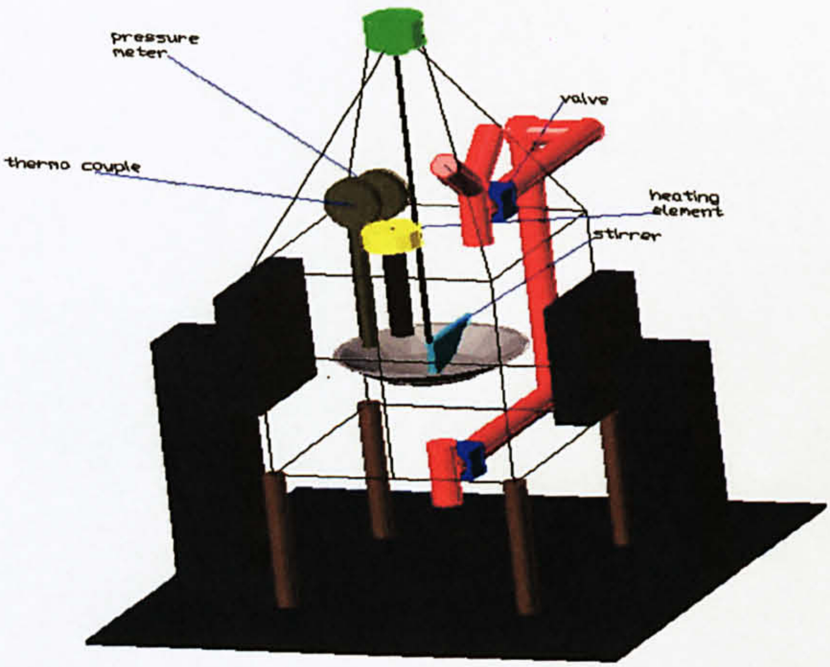


Figure 9: Front View of Autocad Drawing

- Side view

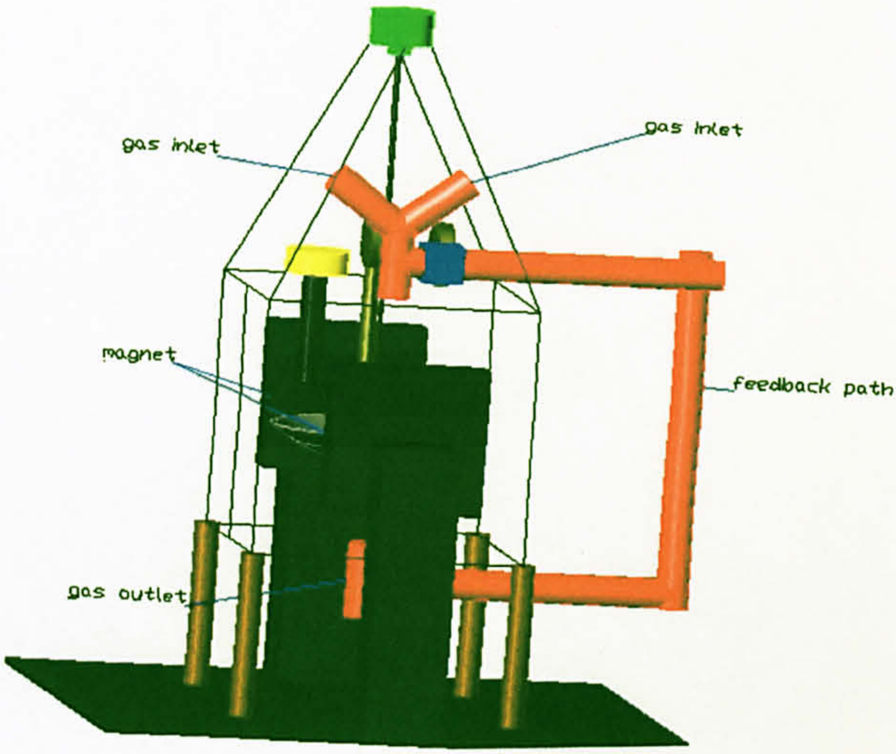


Figure 10: Side View of Autocad Drawing

- Top view

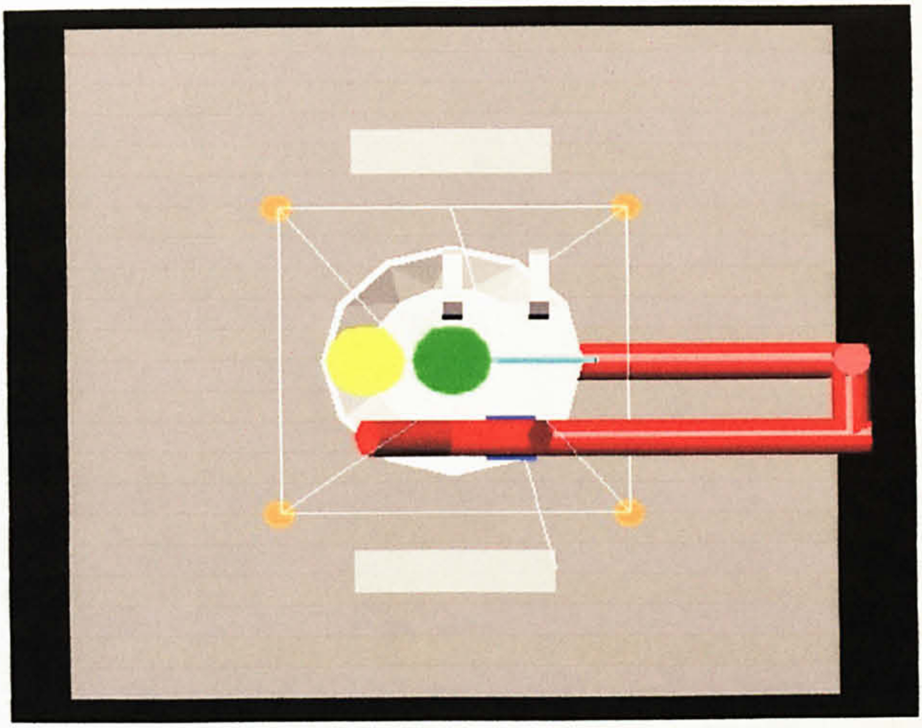


Figure 11: Top View of Autocad Drawing

4.3 Prototype Model

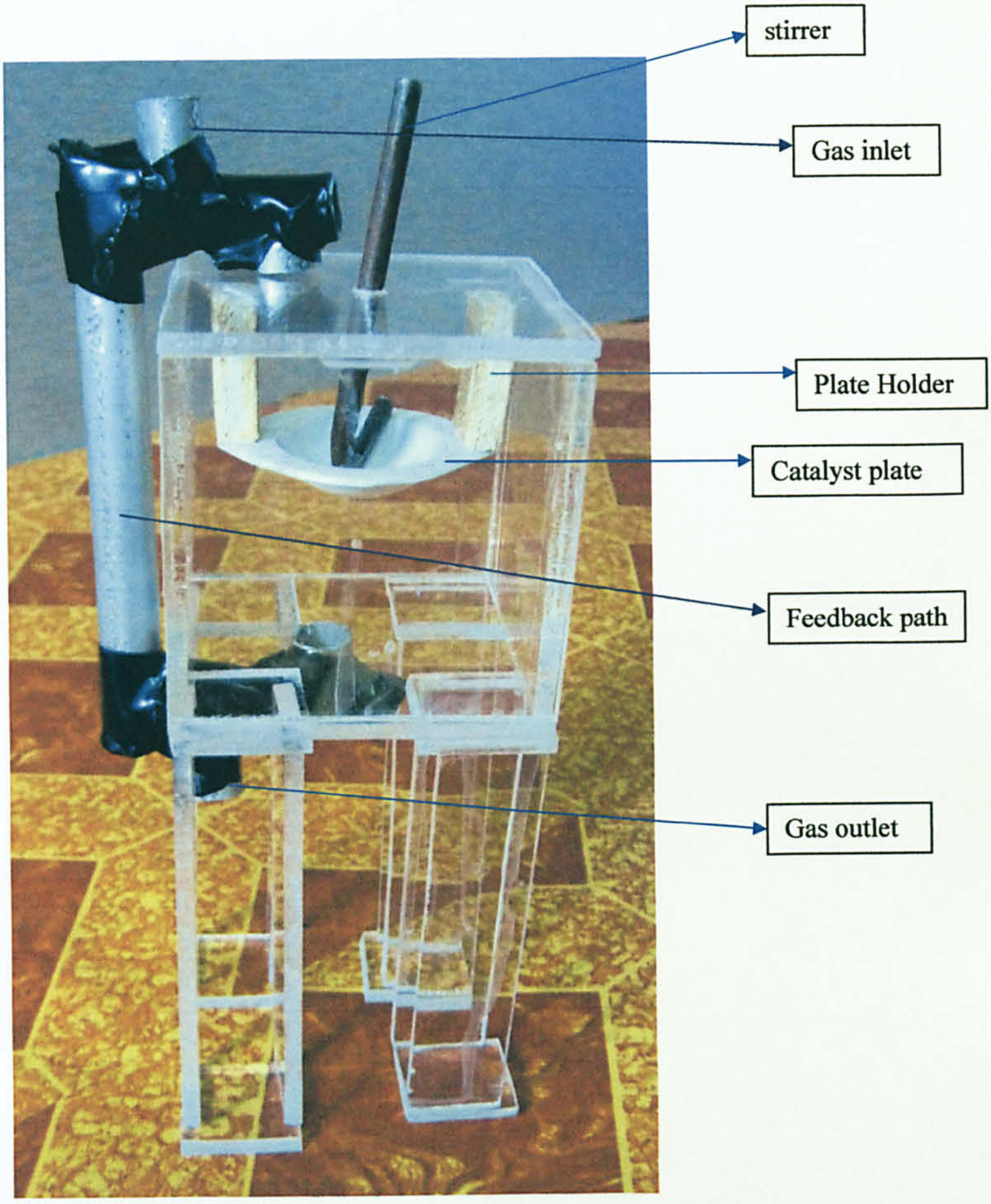


Figure 12: Prototype Model of Microreactor in square shape

4.4 Comparison of Surface Area Between Cylinder and Square Shape:

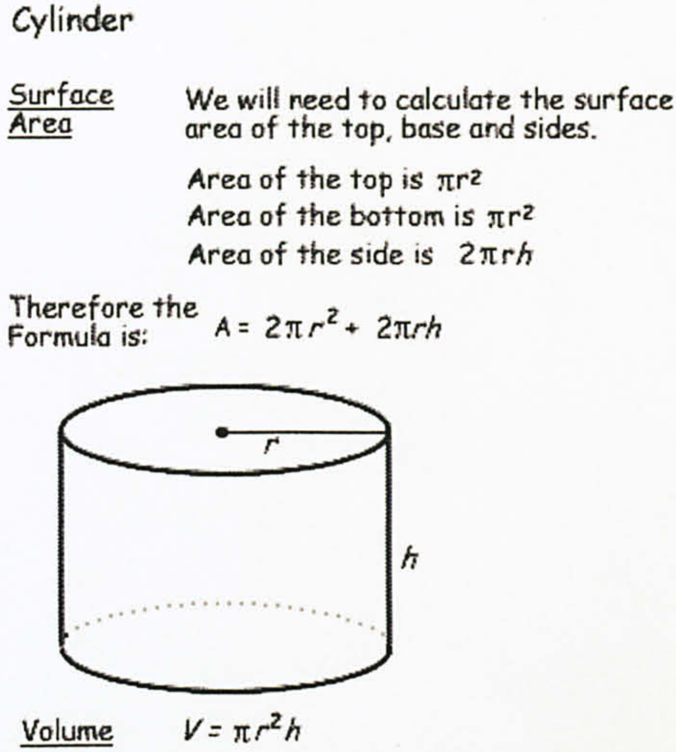


Figure 13: Cylindrical Characteristic of Area and Volume

Calculation:

Given the respective dimensions of the chamber as below:

h = 34cm

r = 5.40cm / 2

Thus, based on the formula stated above, substitute the respective value of designed microreactor,

- Area:

  $A = 2\,(\pi)\,(5.40/2)^2 + 2\,(\pi)\,(5.40/2)\,(34) =$ **<u>622.60 cm²</u>**

- Volume:

  $V = (\pi)\,(5.40/2)^2\,(34) =$ **<u>778.68 cm³</u>**

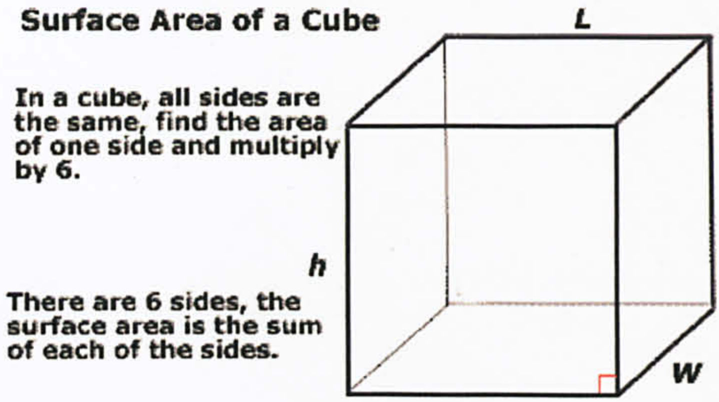


Figure 14: Square Characteristic of Surface Area and Volume

Calculation:

The dimension of the side of square shape is:

L = W = h = 7cm

So,

- Area:

  $A = 6\,(L \times W) = 6\,(7)\,(7) = 6 \times 49cm^2$

  $= \underline{\mathbf{294\ cm^2}}$

Volume of the cube or this square shape can be defined as:

$V = L \times W \times h$

Thus,

- Volume:

  $V = (7)\,(7)\,(7) = \underline{\mathbf{343\ cm^3}}$

From the calculation above, we can conclude that cylindrical shape has maximum surface area as well as shape volume. Due to that, it is the best shape chosen for fabrication of the microreactor chamber.

4.5 Finalised Design

At first, third design has been agreed to be chosen, yet while doing a comparison in terms of surface area and volume, cylindrical shape is seemed more reasonable and meets all the requirements as well as satisfactions. Apart from that, it has been optimized for reliability, safety, more sophisticated and cost minimizing. Below is shown the sketch design as well as an autocad version design, together with the real fabrication model of the smart microreactor.

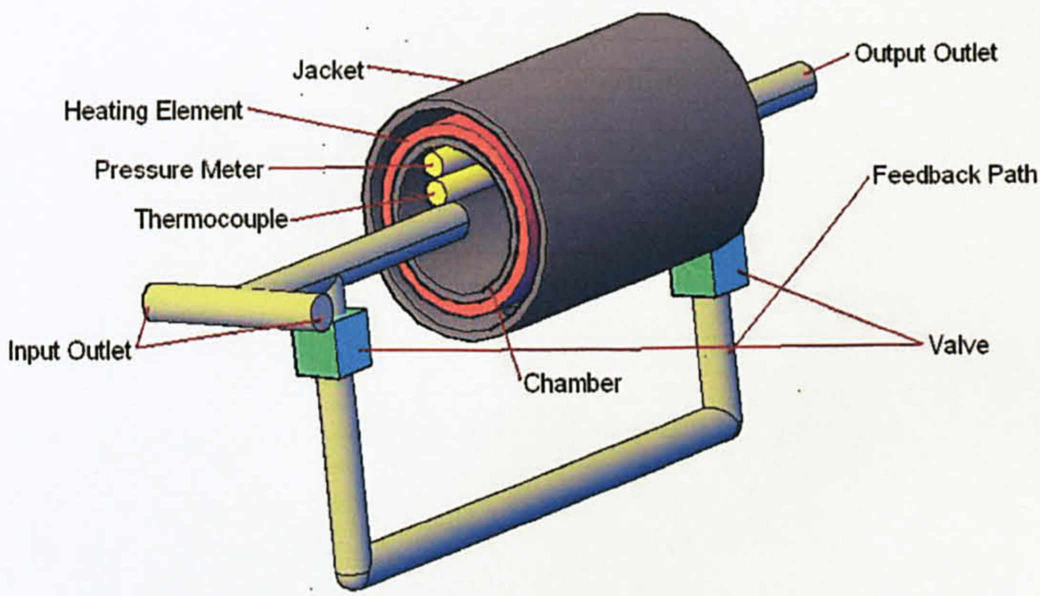


Figure 15: Side view of Cylindrical Microreactor Via Autocad Design

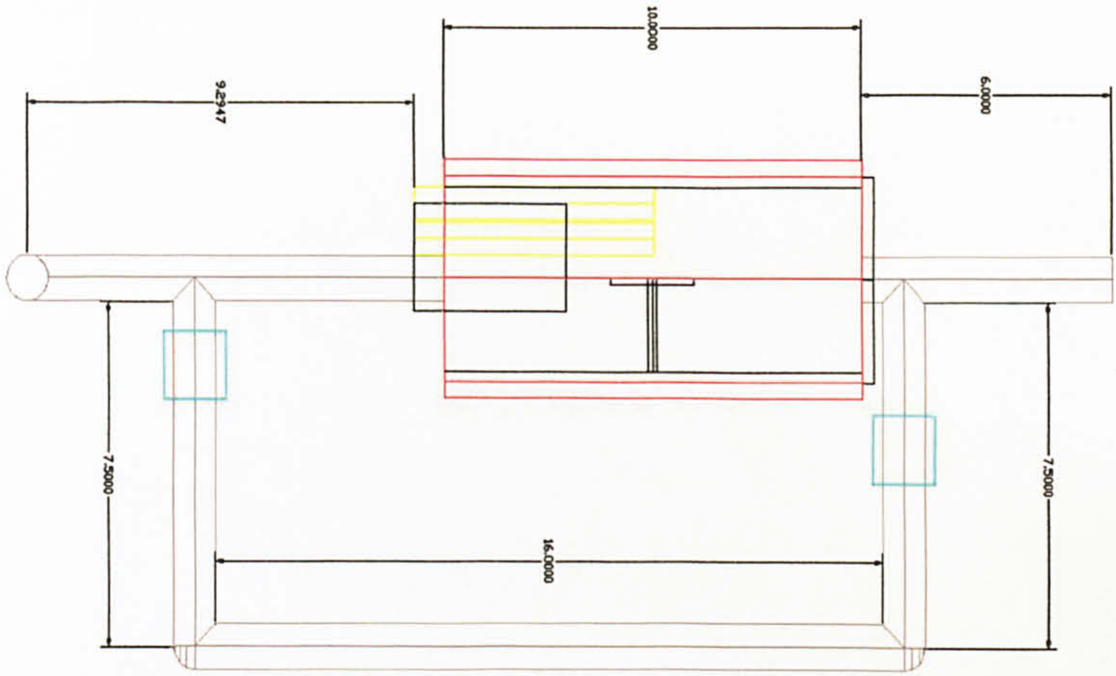


Figure 16: Overall Layout Diagram of Side View With Approximate Dimension

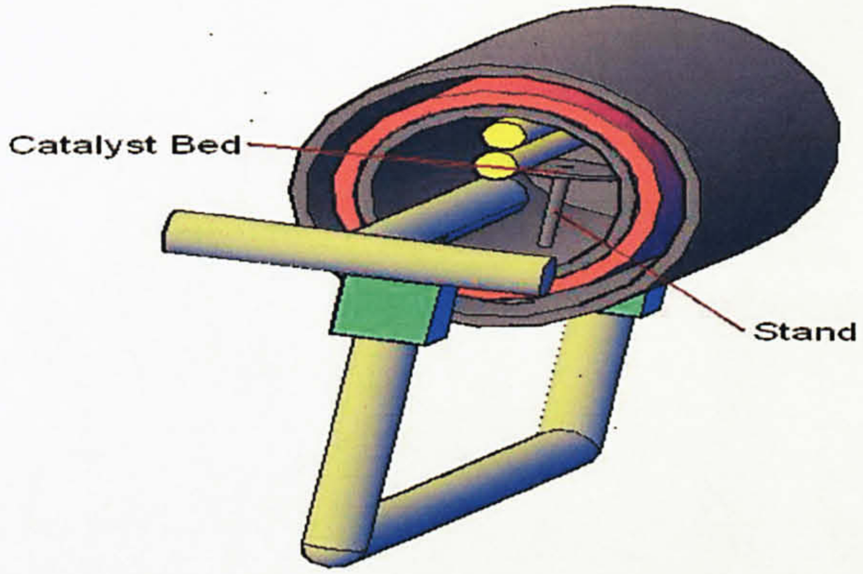


Figure 17: Autocad Drawing To Show The Catalyst Bed Inside The Chamber

4.6 Real Fabrication of Microreactor

Figure 18: Jacket

The figure above showed the jacket of microreactor which made of stainless steel and has specifications as below:

Table 2: Specifications of Jacket

Criterias	Dimensions (cm)
Length	30.0
Diameter	5.70
Width	0.20

The chosen material is stainless steel since it can be bear with a very high temperature as well as pressure. After thorough investigating and research, stainless steel consists of 11% Chromium by mass. Chromium has a melting point up to 1857°C or 2130K. Thus, it is absolutely a very suitable material to be used for microreactor design since ammonia synthesis just required at about 250°C after the temperature is expected to be lowered by nano catalyst activities.

Besides, stainless steel does not stain, corrode, or rust as easily as ordinary steel (it stains less). It is also 100% recyclable. In fact, an average stainless steel object is composed of about 60% recycled material, 25% originating from end of life products and 35% coming from manufacturing processes. Even it is no longer be used, it can be decomposed or recycle back and any waste of materials can be avoided. This jacket will be placed at the outer of the chamber and will be a medium for cooling system purposes.

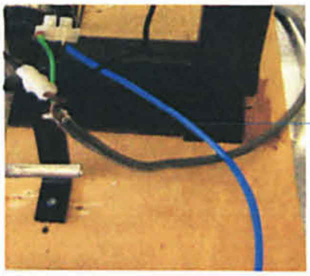


Figure 19: Air Tube

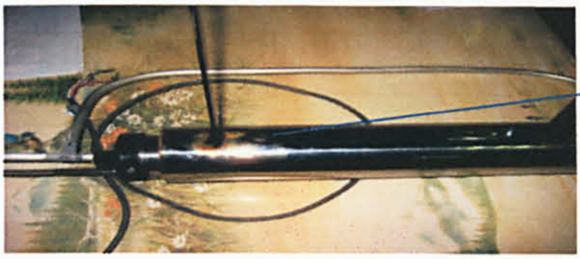


Figure 20: A Hole Drilled At The Bottom of The Jacket

The air tube is made from a rubber which one end will be placed at the bottom of the jacket in the hole drilled and the other end will be connected to the air cooling device. The air tube's measurement is listed in the table below:

Table 3: Specifications of Air Tube

Criterias	Dimensions (cm)
Length	30.0
Diameter	0.70
Thickness	0.15

This air tube is very essential in ensuring the temperature of the chamber is being maintained by allowing the air from air cooling device flowing inside to cool down the temperature of the chamber while reaction is being carried out. This can prevent overheating and any damage that might be happened to the equipments also can be avoided.

Figure 21: Chamber of Microreactor

Above is shown the figure of the chamber which the reaction of ammonia synthesis will take place. The chamber and the cap also made of stainless steel as the jacket is. The cap of the cylinder is painted black in colour to make us easy to see the difference between the chamber and its cap. The corresponding dimension of the chamber is stated as in table below:

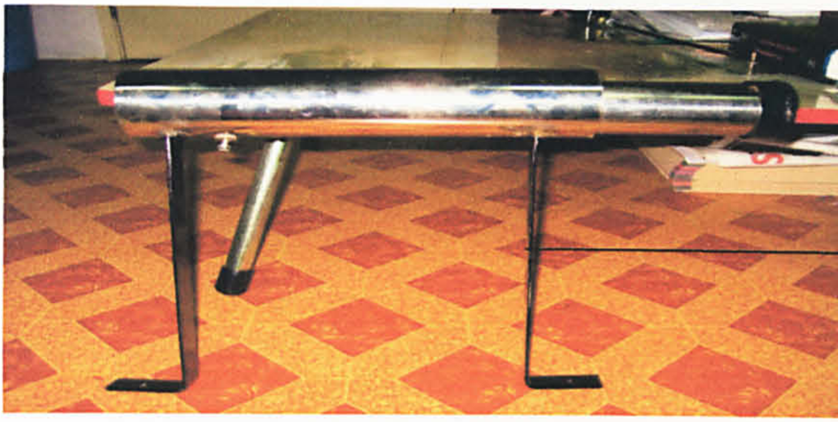
Table 4: Specifications of the Chamber

Criterias	Dimensions (cm)
Length	34.0
Diameter / Width	5.40
Thickness	0.20

The cap is important since it will act as a door to the cylindrical chamber. It will be placed at the both side, in the left and in the right of the chamber and the door will be close while the reaction is being carried out. Once the temperature of the chamber has been cooled down, the door can be open for maintenance purposes or replacing any new equipment inside. The table below listed the specifications of the cap:

Table 5: Specifications of the Cap

Criterias	Dimensions (cm)
Width	2.00
Diameter	5.50
Thickness	0.20



microreactor stand

Figure 22: Microreactor stand

This microreactor stand is made of stainless steel and is painted black in colour same as the cap of the chamber. The function of this stand is to hold the microreactor firmly in an efficient manner as well as to ensure the reaction can be carried out in a good condition. There are two stands used and placed at the bottom of the jacket as shown in the picture above. The stand has specified measurements and has been simplified in the table as below:

Table 6: Specifications of Chamber Stand

Criterias	Dimensions (cm)
Length	20.0
Width	7.00
Thickness	0.10

Figure 23: Pressure Meter

The maximum pressure indicated of the respective pressure meter used in experimental testing of ammonia synthesis is 20bar. The standard atmosphere is a unit of pressure and is defined as being equal to 101.325 kPa. The other unit's equivalent is 760 mmHg (torr), 29.92 in Hg, 14.696 PSI, 1013.25 millibars. The real ammonia synthesis in the plant carried out the reaction process under the pressure of 180bar. Since this ammonia synthesis reaction just require 1 atmosphere pressure (ambient pressure), thus, this pressure meter is suitable to be used due to the ability of this device to bear with the reaction temperature of the chamber as 1 atmosphere (1 atm) is just equal to 1.01325 bar.

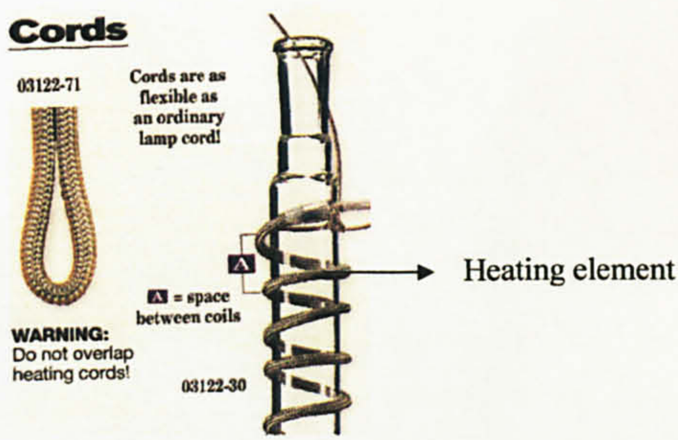


Figure 24: Spiral Type of Heating Element

The heating element that going to be used is suggested to be in spiral type due to the limited space and a small size of microreactor. It will be placed around the chamber as spiral wound heating element packs more heat into each of cord.

Furthermore, it has a heat transfer ability up to 850°F or equivalent to 454°C which is absolutely not exceed than the range of temperature required for ammonia synthesis by using this microreactor, ≈ 250°C and less than 400°C. This heating element requires 120V ac, maximum 10 amperes and maximum power drawn is 1200watts.

$$P = I \times V \longrightarrow (10A)(120V)$$
$$= 1200 \text{ watts.}$$

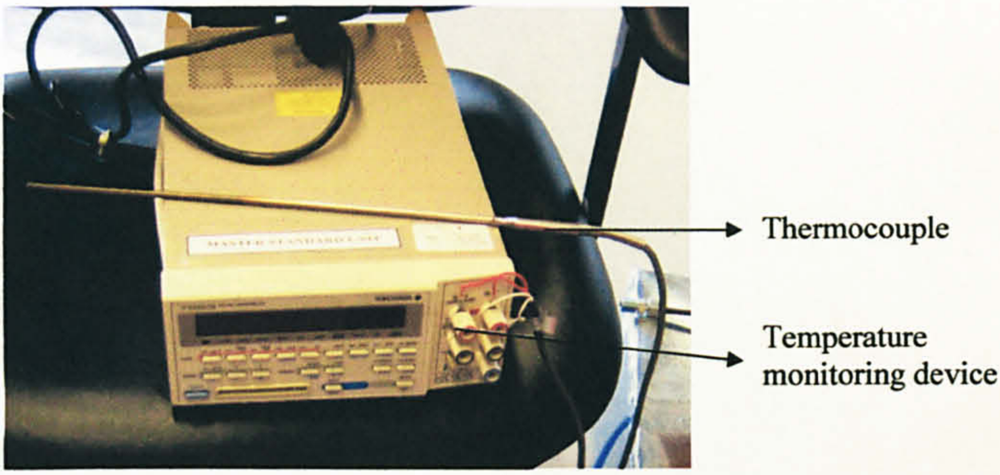


Figure 25: Thermocouple

This thermocouple is used to measure the temperature while the reaction process is being carried out. Maximum point can be measured by this device is until 1000°C. The heating elements installed in the chamber will be heated and soon, the thermocouple will detect the temperature and send the information to the temperature monitoring device to display the reading. This thermocouple is an important element to ensure that the reaction process is being controlled in a well mannered as well as monitoring the temperature to not being exceeded the limit fixed. Otherwise, this will destroy the reaction process causing the equipments become damage too.

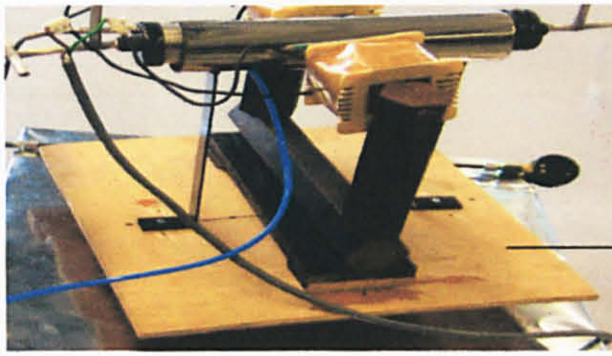


Figure 26: Flexible Supporter

The flexible supporter shown above is made of wood which possessed specifications as listed in this corresponding table:

Table 7: Specifications of Flexible Supporter

Criterias	Dimensions (cm)
Length	40.0
Width	40.0
Thickness	1.00

It is designed in such a way that can be swirl in a freely movement, so that, it is more practical and not time-consuming since ones doesn't have to go to the other side to do any maintenance and rectifications. The magnet and the microreactor which placed in between it are put onto this flexible stand. It has been designed and fabricated firmly to be able to support the total weight of magnet as well as microreactor together with all the equipments.

Figure 27: Gas Flow Properties

The figure showed inlet, outlet and feedback path which are made from the same metal, aluminium. Aluminium has been chosen since it is cheaper than stainless steel and easy to find anywhere. Other than that, aluminium is easy to bend for the feedback path. These two inlets will be connected to the Nitrogen and Hydrogen gas tank and the tanks will be located nearby the microreactor.

As known, the ammonia synthesis requirement must possessed three characteristics which are presence of catalyst, Nitrogen gas as well as Hydrogen gas in the relevant temperature and pressure. After reaction of the ammonia synthesis completed in the chamber, the products then will be collected through the outlet. The feedback path is a must for the unused gas to be recycle back to avoid any waste. The dimensions of this aluminium metal have been simplified as below:

Table 8: Specifications of Aluminium

Criterias	Dimensions (cm)
Length	15.0
Diameter	1.00
Thickness	0.15

The overall figure of real microreactor fabrication is:

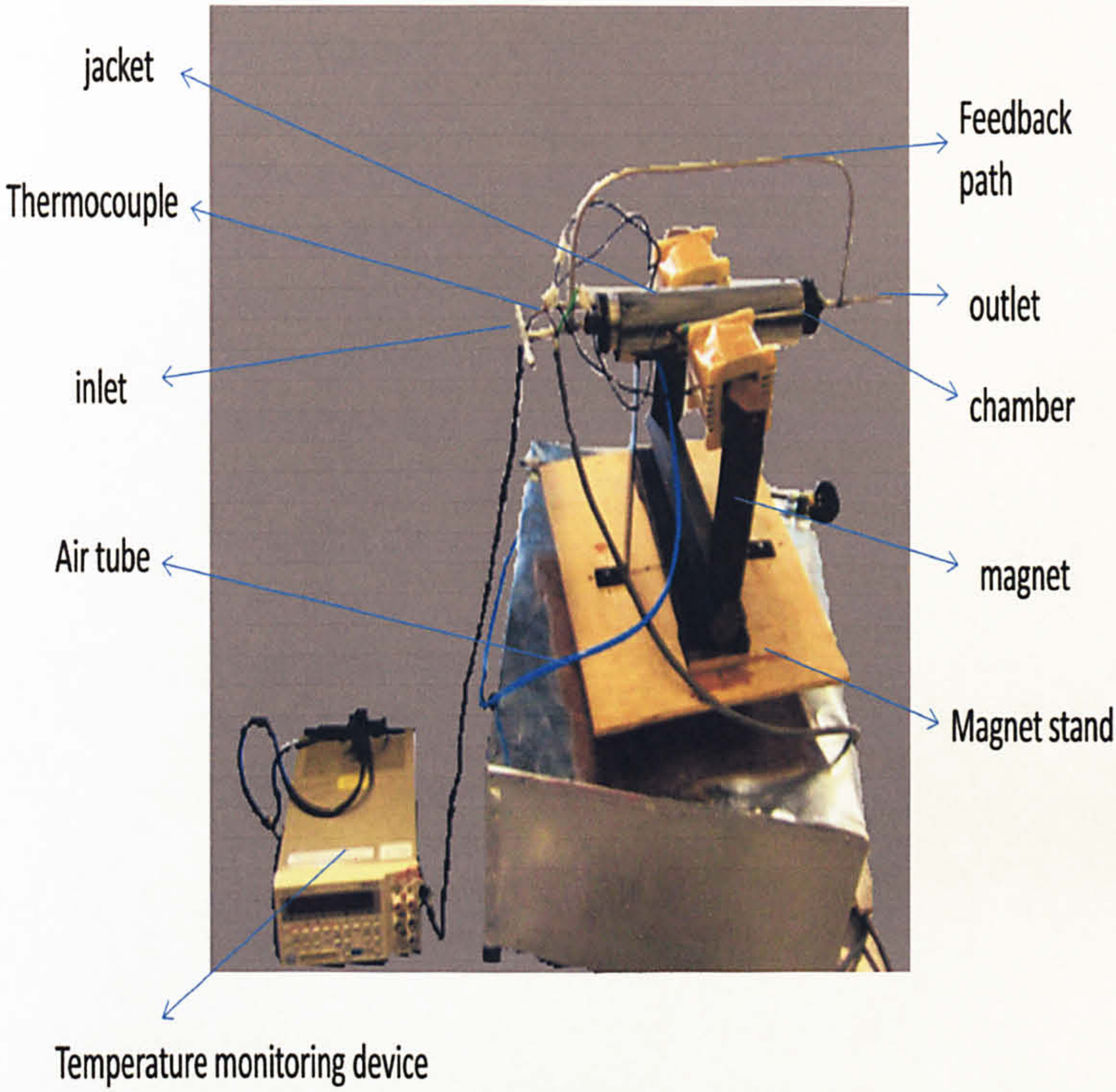


Figure 28: Real model fabrication of microreactor

4.7 Experimental Testing

An experimental testing has been done to the gas inside the chamber to know the relationship between temperature and pressure of nitrogen and hydrogen gases. Assume that the nitrogen and hydrogen gases will follow the properties of the gas inside the chamber, thus the results revealed this pattern based on the experiment conducted:

Table 9: Data of pressure and temperature

Temperature (K)	Pressure (mbar)
303	1013
323	1037
343	1250
363	1099
383	1229
403	1359
423	1468
443	1422
463	1587
483	1650
503	1515
523	1679
543	1993
563	1807
583	1871
603	1996
623	2000
643	1611
663	2328
683	2192

The readings should be started at room temperature as well as room pressure which is extended as below:

Since SI unit of temperature is in Kelvin (K), thus at room temperature, 30°C + 273 = 303K.

Pressure at room temperature is at 1atmosphere (1atm) equivalent to 1013.25mbar or 1.01325bar.

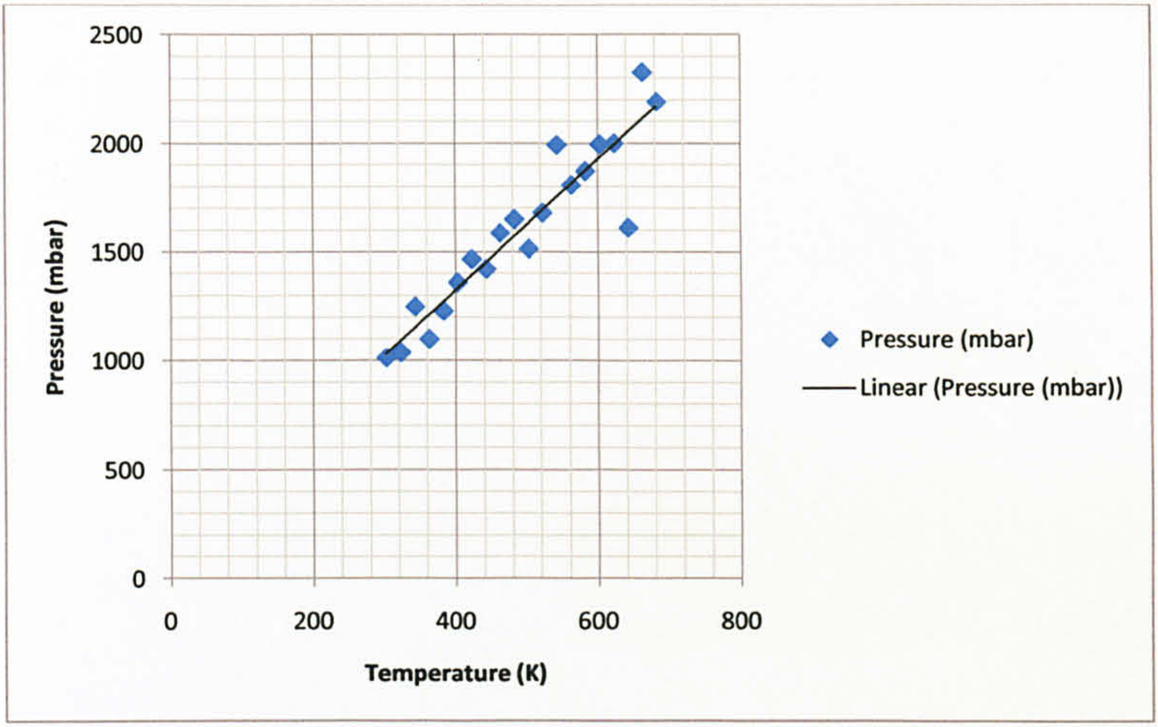


Figure 29: Graph temperature vs pressure of the gas inside the chamber

From the Ideal Gas Law, it stated that:

$$\frac{P_1V_1}{T_1} = \frac{P_2V_2}{T_2} = k$$

where P = pressure of the gas inside the chamber
T = temperature of the gas inside the chamber

k = constant
V = volume of the chamber

Volume has been fixed and is a constant which is 779cm^3. Volume of the gas will fulfill the whole volume of the chamber since the behavior of the gas is fulfilling the space. Thus,

$$\frac{P_1}{T_1} = \frac{P_2}{T_2} = k \qquad \text{or we can simply write as} \qquad \frac{P}{T} = k$$

So, from the graph, the slope of the graph is simply P / T which is represented the value of constant k that is needed to be defined.

Pick two points that are located almost exactly on the line, which have been identified as point sixth with coordinate (403, 1359) and point fourteenth with coordinate (563, 1807). From that,

$$\mathbf{k} = \frac{(1807 - 1359)}{(563 - 403)} = \frac{448}{160} = \mathbf{2.8}$$

As known, $\frac{P}{T} = \frac{k}{V}$

Thus, $P = T \times \frac{k}{V} = T\frac{(2.8)}{(779)} = 0.00359T$

So, it can be concluded that based on the testing carried out, the relationship between pressure and temperature of the gas inside the chamber is given as $P = 0.00359T$.

The work is then proceeded to estimate the expected ammonia production after the practical works have been carried out by. From the general equation of ammonia which is

$$3H_2 + N_2 \leftrightarrow 2NH_3$$

Let take for example $389cm^3$ in estimation of hydrogen gas is allowed to flow into the chamber, thus:

$$\rho = \frac{m}{V}$$

where ρ = density of hydrogen
m = mass of hydrogen
V = volume of hydrogen

So, to find mass of the hydrogen is simply $m = \rho V$ whereby density of hydrogen is a constant value given as $0.000084\ g/cm^3$. Substituted the value into the equation results

$$m = \rho V = (0.000084)(389) = 0.032676\ g$$

So,

$$\text{mole} = \frac{\text{mass}}{\text{molecular mass}} = \frac{0.032676\ g}{(1.0079 \times 2)} = 0.01621\ \text{mole}$$

Thus, estimated:

$0.01621\ H_2 + 0.0054\ N_2 \leftrightarrow 0.01081\ NH_3$ by taking ratio from the general equation which is $3H_2 + N_2 \leftrightarrow 2NH_3$.

So, estimated mole of NH_3 produced is 0.0108. Molecular mass of ammonia is 17.031 g/mol.

To find expected mass of ammonia,

$$\begin{aligned} \text{mass} &= \text{mole x molecular mass} \\ &= 0.0108 \times 17.031 \\ &= 0.18411\ \text{g} \end{aligned}$$

Then, density of NH_3 at (1.013 bar at boiling point) = 0.86 kg/m^3. So,

$$V = \frac{m}{\rho} = \frac{0.18411 \times 10^{-3}\ \text{kg}}{0.86\ \text{kg/m}^3} = 2.14 \times 10^{-4}\ \text{m}^3 \longrightarrow \quad \text{need to change in cm}^3$$

Thus, $2.14 \times 10^{-4} \times 10^{6} \approx$ **214 cm^3** of expected NH_3 will be produced.

Normally in real industry, ammonia produced after the overall synthesis process just at about 17% or less than 20%. So, expected ammonia yields based on this volume is

$$\frac{17}{100} \times 214\ \text{cm}^3 = \mathbf{36.39\ cm^3} \text{ without feedback path}$$

By using a feedback path, based on the theoretical and research that have been done, it will cause approximately 5% increment in ammonia production. [4]. Thus,

$$\frac{22}{100} \times 214\ \text{cm}^3 = \mathbf{47.08\ cm^3} \text{ with feedback path.}$$

So, it can be concluded that expected ammonia increment is at (47.08 cm^3 – 36.39 cm^3) = **10.69 cm^3** when applied feedback path in the design.

4.8 Scale factor and estimation of power consumed

In industry:

Normal Pressure = 194 Bar = 191.46 atm

Volume reactor = A x h

$= \pi r^2 \times h$

$= \pi \times (1.250\ m)^2 \times 21.210\ m$

$= \mathbf{104.11\ m^3} \longrightarrow \mathbf{10.411 \times 10^7\ cm^3}$

If the pressure for reactor consider for 1 atm, the volume will be:

$104.11\ m^3 / 191.46\ atm = 0.544\ m^3 = 5.44 \times 10^{-1}\ m^3 \longrightarrow 544{,}000 cm^3$

The volume of microreactor according to the design is:

$\pi \times (0.054/2\ m)^2 \times 0.34\ m = 7.787 \times 10^{-4}\ m^3 = \mathbf{778.675\ cm^3 \approx 779\ cm^3}$

Scale factor = Vr / Vm = $5.44 \times 10^5\ cm^3 / 779\ cm^3 = 698$

Scale factor = **1 : 698**

Now, the calculation has been proceeded to estimate the power consumed of micoreactor by this design. From the research, it is known that power used by microreactor in ammonia plant is about 308 kW with ammonia produced normally at $1000 cm^3$. By scaling factor, estimated power consumed is 308 kW/698 = 0.44 kW with expected ammonia production as calculated above is $47.08\ cm^3$.

Table 10: Comparison of microreactor in industry and designed project

Subject	Microreactor in industry	Design project microreactor
Estimated power consumed (kW)	308	4.4×10^{-1}
Expected ammonia production (cm^3)	1000	47.08

If this design used the same power as in the plant, whereby 308 kW, thus the expected ammonia production is $47.08\ cm^3 \times 698$ (scale factor) $\approx$ **32 862 cm^3** which is much higher than the current production in industry. Thus, it can be concluded that via this design of microreactor, it is expected could increase the ammonia production theoretically.

CHAPTER 5
CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

It can be concluded that the new design of microreactor for ammonia synthesis has been successfully invented together with the idea of introducing a magnetic field in this design. This magnetic field effect theoretically is believed to quicken the production of ammonia. This microreactor design has been scaled down in size compared to the large scale used in industry. As a result of that, it is less in materials costs and maintenance, easy to handle and also not time-consuming.

5.2 RECOMMENDATION

There are some possible recommendations could be done to improve the microreactor design as stated below:

5.2.1 Tools and Equipments

Some of the tools and important equipments for real microreactor fabrication for instance heating element might cannot be installed yet due to the long time needed for delivery system ordered from overseas and difficult to find exact specifications required by this type of microreactor. Like pressure meter, it cannot be installed yet too in this microreactor fabricated due to the valves and fittings problems. Thus, it is recommended to use remote monitoring of temperature and pressure by giving commands in software based.

5.2.2 Filter

This filter must be a type of chemical filter and has its own specifications to suit with the ammonia synthesis. It can be introduced at the bottom part of microreactor to remove any unwanted chemicals and to get pure ammonia.

5.2.3 System Analyser

Another recommendation is putting the ammonia system analyser in the outlet path to analyse or calculate the ammonia production.

5.2.4 Software Simulation

After fabrication phase is being completed, it is much better if it is being tested by engineering software simulation to ensure its efficiency before experimental testing being done.

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APPENDIX A

Table

Z	Name	Symbol	Melting Point in °C (Kelvin)
2	Helium	He	(Does not solidify at normal pressure, even near absolute zero, but might at extreme conditions, at 0.0213K)
1	Hydrogen	H	−258.975 °C (14.2 K)
10	Neon	Ne	−248.447 °C (24.7 K)
8	Oxygen	O	−222.65 °C (50.5 K)
9	Fluorine	F	−219.52 °C (53.6 K)
7	Nitrogen	N	−209.86 °C (63.3 K)
18	Argon	Ar	−189.19 °C (84.0 K)
36	Krypton	Kr	−157.22 °C (116 K)
54	Xenon	Xe	−111.7 °C (161 K)
17	Chlorine	Cl	−100.84 °C (172 K)
86	Radon	Rn	−71 °C (202.1 K)
80	Mercury	Hg	−38.72 °C (234 K)
35	Bromine	Br	−7.1 °C (266 K)
87	Francium	Fr	27 °C (300 K)
55	Cesium	Cs	28.55 °C (301.70 K)
31	Gallium	Ga	29.76 °C (302.91 K)
37	Rubidium	Rb	39.64 °C (312.79 K)
15	Phosphorus (white)	P	44.1 °C (317.3 K)
19	Potassium	K	63.35 °C (336.50 K)
11	Sodium	Na	98 °C (371 K)
53	Iodine	I	113.5 °C (386.7 K)
16	Sulphur	S	115.36 °C (388.51 K)
49	Indium	In	156.76 °C (429.91 K)
3	Lithium	Li	180.7 °C (453.9 K)
34	Selenium	Se	221 °C (494 K)
50	Tin	Sn	232.06 °C (505.21 K)
84	Polonium	Po	254 °C (527 K)
83	Bismuth	Bi	271.52 °C (544.67 K)
85	Astatine	At	302 °C (575 K)
81	Thallium	Tl	304 °C (577 K)
48	Cadmium	Cd	321.18 °C (594.33 K)

82	Lead	Pb	327.6 °C (600.8 K)
30	Zinc	Zn	419.73 °C (692.88 K)
52	Tellurium	Te	449.65 °C (722.80 K)
51	Antimony	Sb	630.9 °C (904.1 K)
93	Neptunium	Np	640 °C (913 K)
94	Plutonium	Pu	640 °C (913 K)
12	Magnesium	Mg	650 °C (923 K)
13	Aluminium	Al	660.25 °C (933.40 K)
88	Radium	Ra	700 °C (973 K)
56	Barium	Ba	729 °C (1,002 K)
38	Strontium	Sr	769 °C (1,042 K)
58	Cerium	Ce	798 °C (1,071 K)
33	Arsenic	As	817 °C (1,090 K)
63	Europium	Eu	822 °C (1,095 K)
70	Ytterbium	Yb	824 °C (1,097 K)
20	Calcium	Ca	839 °C (1,112 K)
99	Einsteinium	Es	860 °C (1,130 K)
57	Lanthanum	La	920 °C (1,190 K)
59	Praseodymium	Pr	931 °C (1,204 K)
61	Promethium	Pm	931 °C (1,204 K)
32	Germanium	Ge	938.3 °C (1,211.4 K)
47	Silver	Ag	961 °C (1,234 K)
97	Berkelium	Bk	986 °C (1,259 K)
95	Americium	Am	994 °C (1,267 K)
60	Neodymium	Nd	1,016 °C (1,289 K)
89	Actinium	Ac	1,050 °C (1,320 K)
79	Gold	Au	1,064.58 °C (1,337.73 K)
96	Curium	Cm	1,067 °C (1,340 K)
62	Samarium	Sm	1,072 °C (1,345 K)
29	Copper	Cu	1,084.6 °C (1,357.8 K)
92	Uranium	U	1,132 °C (1,405 K)
25	Manganese	Mn	1,246 °C (1,519 K)
4	Beryllium	Be	1,278 °C (1,551 K)
64	Gadolinium	Gd	1,312 °C (1,585 K)
65	Terbium	Tb	1,357 °C (1,630 K)
66	Dysprosium	Dy	1,407 °C (1,680 K)
14	Silicon	Si	1,410 °C (1,680 K)
28	Nickel	Ni	1,453 °C (1,726 K)

67	Holmium	Ho	1,470 °C (1,740 K)
27	Cobalt	Co	1,495 °C (1,768 K)
101	Mendelevium	Md	1,521 °C (1,794 K)
102	Nobelium	No	1,521 °C (1,794 K)
68	Erbium	Er	1,522 °C (1,795 K)
39	Yttrium	Y	1,526 °C (1,799 K)
26	Iron	Fe	1,535 °C (1,808 K)
21	Scandium	Sc	1,539 °C (1,812 K)
69	Thulium	Tm	1,545 °C (1,818 K)
46	Palladium	Pd	1,552 °C (1,825 K)
91	Protactinium	Pa	1,600 °C (1,870 K)
98	Californium	Cf	1,652 °C (1,925 K)
22	Titanium	Ti	1,660 °C (1,930 K)
71	Lutetium	Lu	1,663 °C (1,936 K)
90	Thorium	Th	1,755 °C (2,028 K)
78	Platinum	Pt	1,772 °C (2,045 K)
40	Zirconium	Zr	1,852 °C (2,125 K)
24	Chromium	Cr	1,857 °C (2,130 K)
23	Vanadium	V	1,902 °C (2,175 K)
45	Rhodium	Rh	1,966 °C (2,239 K)
43	Technetium	Tc	2,200 °C (2,470 K)
72	Hafnium	Hf	2,227 °C (2,500 K)
44	Ruthenium	Ru	2,250 °C (2,520 K)
5	Boron	B	2,300 °C (2,570 K)
77	Iridium	Ir	2,443 °C (2,716 K)
41	Niobium	Nb	2,468 °C (2,741 K)
42	Molybdenum	Mo	2,617 °C (2,890 K)
100	Fermium	Fm	2,781 °C (3,054 K)
103	Lawrencium	Lr	2,961 °C (3,234 K)
73	Tantalum	Ta	2,996 °C (3,269 K)
76	Osmium	Os	3,027 °C (3,300 K)
75	Rhenium	Re	3,180 °C (3,450 K)
74	Tungsten	W	3,407 °C (3,680 K)
6	Carbon (diamond)	C	3,550 °C (3,820 K)
6	Carbon (graphite)	C	3,675 °C (3,948 K)
6	Carbon (amorphous)	C	3,675 °C (3,948 K)

APPENDIX B

Gantt Chart

Semester 1

No.	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Research Work															
2	Research Work Continue															
3	Gathering Related Data															
4	Implement Design															
5	Continue Design Implementation															
6	Finalised Design															
7	Sketch / Autocad Drawing															
8	Fabricate Prototype															
9	Fabricate Prototype															
10	Testing Prototype Model															

Suggested milestone

Process

Semester 2

No.	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Prototype Enhancement															
2	Experimental Work															
3	Experimental Work															
4	Experimental Work															
5	Result Analysis															
6	Rectification Process															
7	Actual Fabrication															
8	Actual fabrication															
9	System Testing															
10	Evaluation Day															

Suggested milestone

Process