iJRASET
International Journal For Research in
Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 9 **Issue:** XI **Month of publication:** November 2021

DOI: https://doi.org/10.22214/ijraset.2021.39047

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Chemical Plant Utility – Nitrogen System Design

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Abstract: The study is been conducted to understand the different techniques to separate nitrogen from atmospheric air. Separation of nitrogen takes place by following techniques: Cryogenic air separation, Pressure swing adsorption and Membrane separation technique. Cryogenic air separation operates at a very low temperature, which uses the principle of rectification to separate nitrogen at a very high purity (99.999%). Pressure swing adsorption rely on the fact that higher the pressure, more the gas is adsorbed which results in high purity (95-99.99%) of nitrogen. Membrane separation technology is the process that uses hollow fibre membranes to separate the constituent gases in air, which gives the purity in the range of 93%-99.5%. After the comparative study, it is understood that membrane separation technique is the most efficient technology based on the cost, purity, flexibility in terms of adjusting the purity, maintenance, availability; it operates without heating and therefore uses less energy than conventional thermal separation processes. Different step designs of membrane separation techniques are discussed. A Process Flow Diagram and Piping Instrumentation Diagram is been added for single step membrane separation technique.
Keywords: Atmospheric air, nitrogen, Cryogenic air separation, Pressure swing adsorption, Membrane separation technique.

I. INTRODUCTION

The Scottish scientist, Daniel Rutherford discovered nitrogen in 1772. Nitrogen is the chemical element with the symbol N and atomic number 7. It is used in various applications such as purging out hazardous gases from the system, tire filling systems, pressurised beer kegs, food packaging, in living beings, pharmaceuticals industries, used as a raw material in manufacturing of nitric acid, ammonia etc. Inhalation of nitrogen in excessive amounts can cause dizziness, nausea, vomiting, loss of consciousness, and death.[13]

Atmospheric air contains 78%N_2, 21% O_2 and 1% other gases. In this study, we are focusing on separation of nitrogen from air.

II. VARIOUS TECHNIQUES TO PRODUCE NITROGEN

A. Cryogenic air Separation

Cryogenic air separation is used to gain large-scale production and highest purity (99.999%). Cryogenic air separation based on the principle of rectification. It operates at very low temperatures to separate air components according to the boiling points.[5]

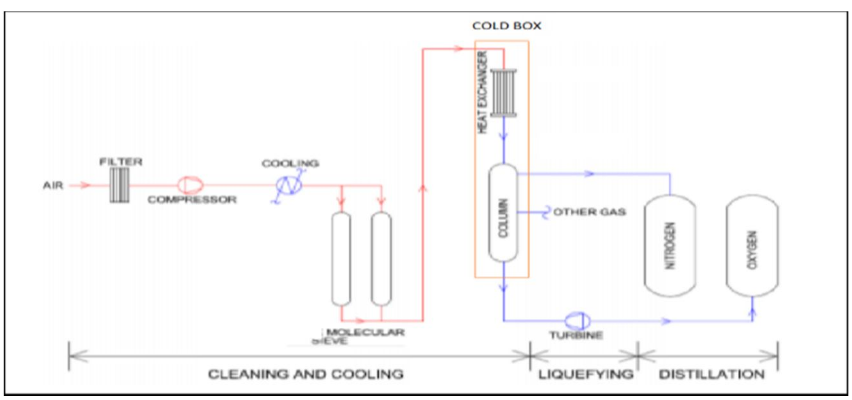

Figure 2.1: Cryogenic air separation

The process begins with the air passing into the filter as a feed where dust particles, impurities and any particulate matter are removed. The cleaned air is then transferred into a compressor, which compresses air to 5 – 8 bar. The clean compressed air is cooled in the pre-cooler (shell and tube heat exchanger used to cool the gas at or nearly the temperature of the cooling water) followed by a spray cooler (Vertical vessel wherein water is sprayed from the top and cool air passed from the bottom, from which all the water droplets are removed).

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Further, air passes into two molecular sieves – the first molecular sieve adsorbs carbon dioxide, hydrocarbons and water and second molecular sieve is used to regenerate nitrogen from cold box. From the first molecular sieves, air passes into the cold box, which includes Heat exchanger (Temperature of air reduces from ambient to cryogenic) and Distillation Column (Depending upon the boiling point nitrogen is condensed and oxygen is vaporised). Therefore, the products formed are liquid nitrogen, Oxygen and other gases. When gaseous nitrogen is required as a product the liquid nitrogen is heated and vaporized. This vaporized Nitrogen gas is used for further applications. Cryogenic processes do not have economic scale i.e., increase or decrease of plant capacity generally does not necessitate new equipment. It requires compressor and turbine, which increase the capital investment and maintenance costs.[3]

B. Pressure Swing Adsorption

Pressure swing adsorption is a process that separates single gas from a gas mixture. It is mainly used in chemical and petrochemical processes as well as the steel industry, for example to recover hydrogen (H_2) from coking or conversion gases, or to separate oxygen (O_2) and nitrogen (N_2) from air.[8]

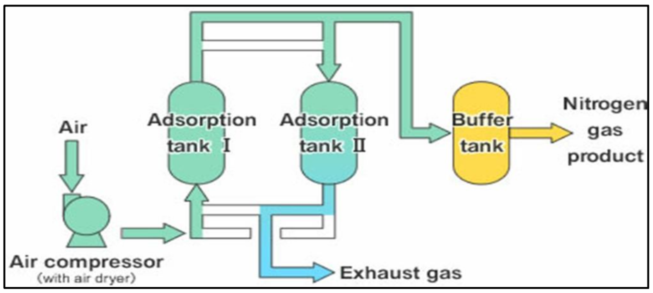

Figure 2.2: Pressure swing adsorption

The adsorption process is based on gas molecules binding to an absorbent material. The adsorbent bed is specially selected depending on the gas to be absorbed. It runs under the fact that higher the pressure, the more gas is adsorbed. When the pressure is reduced, the gas is released, or desorbed. Ideally, only the gas to be separated is adsorbed, while all other gases in the mixture pass through the adsorbent bed. Frequently, absorbents containing carbon (e.g. carbon molecular sieves) and oxide absorbents (e.g. zeolite) are used. If a gas mixture such as air is passed under pressure through a vessel containing an adsorbent bed of carbon molecular sieves that attracts oxygen more strongly than nitrogen part or all of the oxygen will stay in the bed, and the gas exiting the vessel will be richer in nitrogen than the mixture entering. When the bed reaches the end of its capacity to adsorb oxygen, it can be regenerated by reducing the pressure, thus releasing the adsorbed oxygen. It is then ready for another cycle of producing nitrogen-enriched air. The purity of the adsorbed gas not only depends on the adsorbent used, but also the temperature and pressure during the process are important as well. As a result, the control valves used also contribute considerably to the quality of the end product.[9]

C. Membrane Separation Technique

Membrane separation technique uses hollow-fibre membrane technology. There is production of nitrogen from atmospheric air depending on the speeds of the molecules of the constituents. This process requires a conditioning of the feed air due to the clearances in the fibre. The nitrogen cabinet system consists of demister, filter coalescers, immersion heater, carbon vessel, particulate filters & nitrogen membrane. The control system consists of oxygen analysers, purity& product valve.[15]

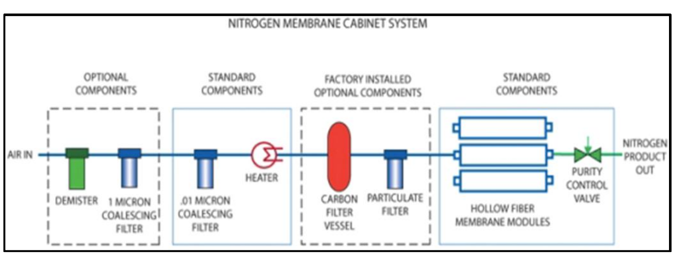

Figure 2.3: Membrane separation technique

The technique implies as follows pulling atmospheric air into the compressor. After compression of air, it is passed through demister to remove dust particles. The first two coalescer filters remove moisture up to 0.01 micron. This air then goes to the heater and is superheated to 10 F to ensure no liquids will enter the nitrogen membrane. Once heated the air will then flow through an activated carbon filter to remove any additional hydrocarbon vapours prior entering the membranes. Further, this air is then passed through the particulate filters to remove any remaining particles down to 0.01 micron. The air has now been conditioned to enter the hollow fibre membrane. The walls of each fibre are permeable to gas molecules, but some gases can pass through more easily than others can. These 'fast' gases, including Oxygen, CO_2, and water vapour, pass through the fibre walls and are exhausted to atmosphere. The 'slow' gas, nitrogen, passes through the fibre wall much more slowly, producing a high purity nitrogen stream at the membrane outlet. There are no moving parts to the membrane, simply controlling the pressure and flow rate of compressed air through the membrane results in high purity nitrogen production. The purity of nitrogen obtained from this membrane separation technique is in the range of (90-99.9%). The advantage of this technique is it is of low cost and the area occupied by the plant is less. The disadvantage is for large capacity of nitrogen to be produced, this method is uneconomic.[9]

Table 2.1: Comparison of production techniques

PARAMETERS	CRYOGENIC AIR SEPARATION	MEMBRANE SEPARATION TECHNIQUE	PRESSURE SWING ADSORPTION
Efficiency	99.999%	93-99.5%	95-99.99%
Start-up Time	12-16 hours	2-3 minutes	5 minutes
Area of the Plant	Large size building and bigger footprint	Compact in size	Comparatively PSA Nitrogen generator is less compact than membrane technique.
Capacity (Nm3/hr)	200- 4lakhs	1-1000	5-5000
Load Range (%)	60-100	30-100	30-100
Operating Temperature (K)	88-98	85-260	283-298
Operating Pressure (bar)	5-8	15-20	4-10
Electricity Consumption to produce 1 Kg of nitrogen (kW)	1976	759	1420
Cost of Nitrogen Production (Rs/Nm^3)	17	2	2

III. SELECTION CRITERIA

Membrane separation technique is an advantageous method based on the following factors:

A. *Simple and Safe Technique*

In membrane separation technique the equipment's used are less in number compared to other methods. Flexible in terms of adjusting the purity. It is easy to operate and install. The area occupied by the plant is less. There is no emission of gases to the environment.

B. *Sustainable and Environment Friendly*

Generating nitrogen gas through membrane separation technique is a sustainable, environmentally friendly and energy-efficient approach to provide pure, clean, dry nitrogen gas. The particular advantage of this method is that it operates without heating and therefore uses less energy than conventional thermal separation processes. Because of the lower energy consumption (up to 62% less) and elimination of the need to transport the gas, there are less greenhouse gases, which are emitted. Thereby helping to protect the environment. Additionally, this technique generates nitrogen at a pressure and flow rate required for the application ensuring that no gas is wasted. It eliminates the dependence on outside vendors as well as the administrative costs are reduced, and the dangers of handling high-pressure cylinders are eliminated. [14]

C. *Purity*

The purity obtained from this technique is in the range of 93-99.5%N_2.The operating conditions for this method are (i) Temperature (85-260K) and (ii)Pressure(15-20 bar). The high capacity that can be used in the range of 1-5000Nm^3/h combined with low air consumption.[11] The increase in flow rate results in low purity & decrease in flow rate results in high purity.

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[11][12]

D. *Economic Technique*

The raw material required for this technique is easily available. In this process, there are no moving parts to the membrane, simply controlling the pressure and flow rate of compressed air through the membrane results in high purity nitrogen production. In this method, there is less usage of equipment's and controls, which results in enormous cost savings in terms of installation, production, running and maintenance compared to conventional gas cylinders.

As we have discussed above, the three techniques of producing Nitrogen; its comparison and taking all the factors and parameters into consideration, membrane separation technique can be considered as the most viable and efficient method.[14]

IV. DIFFERENT STEP DESIGN FOR MEMBRANE SEPARATION TECHNIQUE

A. *One Step Design*

1) The single – step systems used membranes made from Poly (4-methyl-1-pentene) (TPX) with a selectivity of about four.
2) These membranes were incorporated in one – stage designs to produce 95% nitrogen used to render flammable – liquid storage tanks inert.
3) As the membranes improved, more complex process designs, of the type shown in Figure, were used to produce purer gas containing >99% nitrogen.[2]

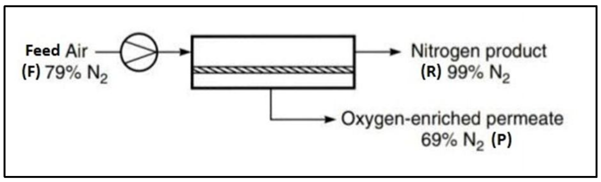

Figure 4.1: One-step design

B. *Two Step Design*

1) The first improvement made, was the two-step process in which concentration of the oxygen gas permeating from the membrane module falls, which is less than the concentration in feed air.
2) Mixing this oxygen-depleted gas permeate with incoming air produces a High quality nitrogen containing less than 1 % oxygen.
3) In this figure 4.2, the second-step permeate gas contains 12.6% oxygen, and recycling this gas to the incoming feed air reduces the membrane area and compressor load by 6%, which relatively saves the capital cost as only few changes in the piping system are been done.[2]

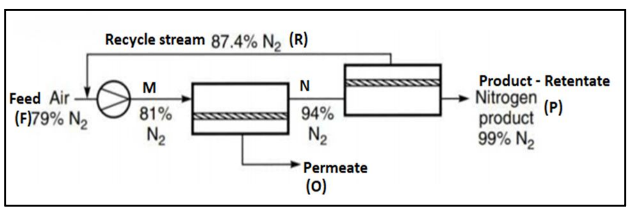

Figure 4.2: Two step design

C. *Three Step Design*

1) The second improvement made, in the three-step process, which was a more efficient design to combine the recycle and feed gas where the feed gas has approximately the same concentration.
2) This design saves a further 2% membrane area and compressor power. As two compressors are needed in the design, saves energy and membrane area but this design brings out extra complexity and higher maintenance cost of second compressor.[2]

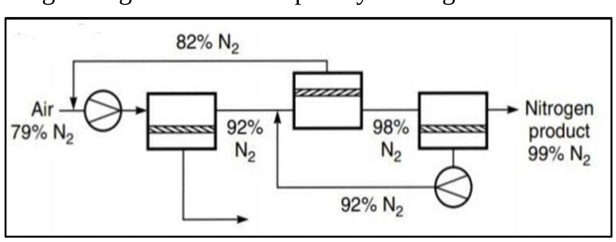

Figure 4.3: Three step design

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Table 4.1: Distinguish between one step and two step designs

Parameters	One step	Two step
Input (moles/hr)	100	100
Retentate (moles/hr)	33.33	41.25
Permeate (moles/hr)	67.67	58.75
Efficiency	41.59%	51.69%
Relative membrane area (m^2)	1.0	0.94
Relative Compressor power (Hp)	1.0	0.94

V. PROCESS DESIGN OF MEMBRANE SEPARATION TECHNIQUE

A. Process Flow Diagram

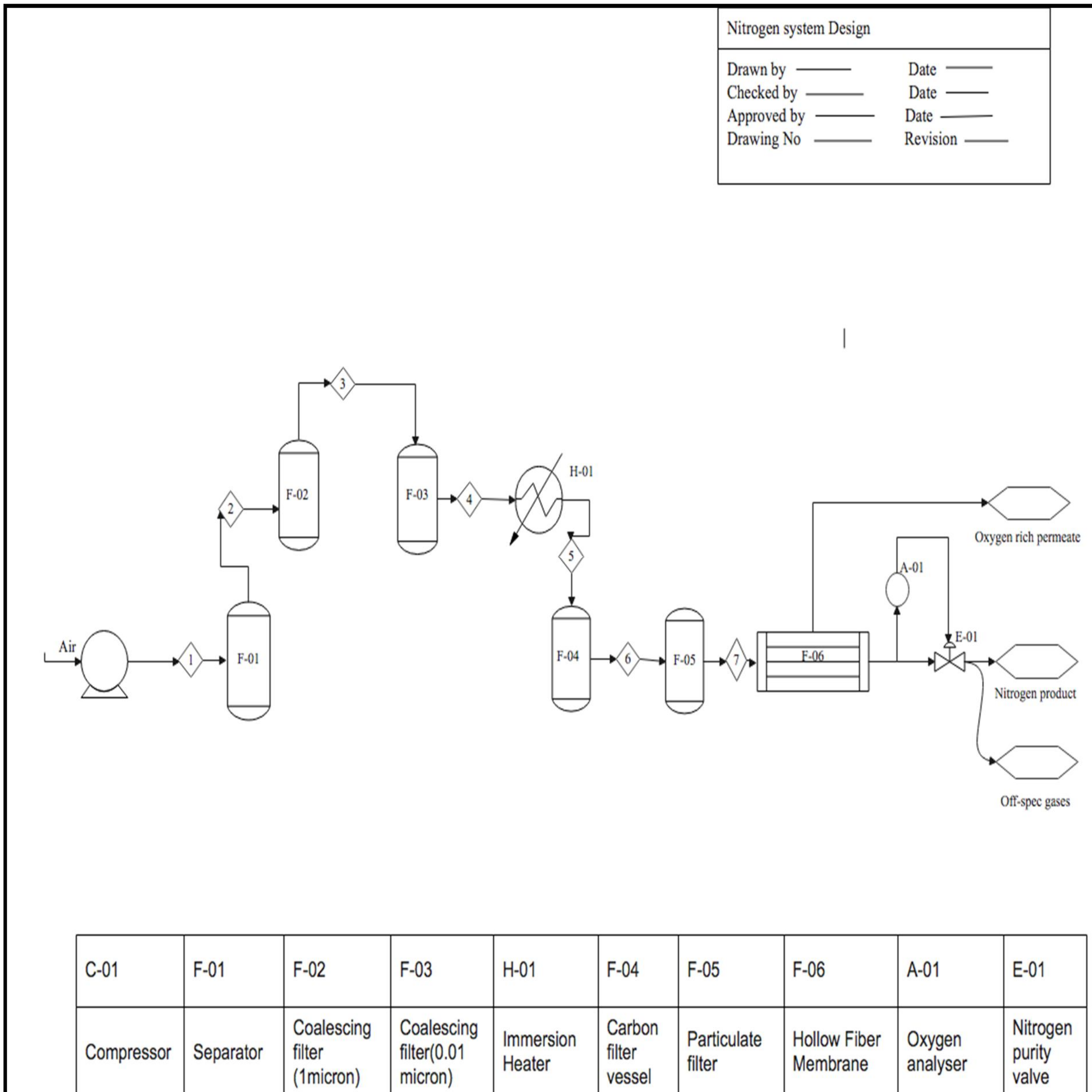

Figure 5.1: Process Flow Diagram of Nitrogen System Design

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ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.429
Volume 9 Issue XI Nov 2021- Available at www.ijraset.com

B. Piping and Instrumentation Diagram

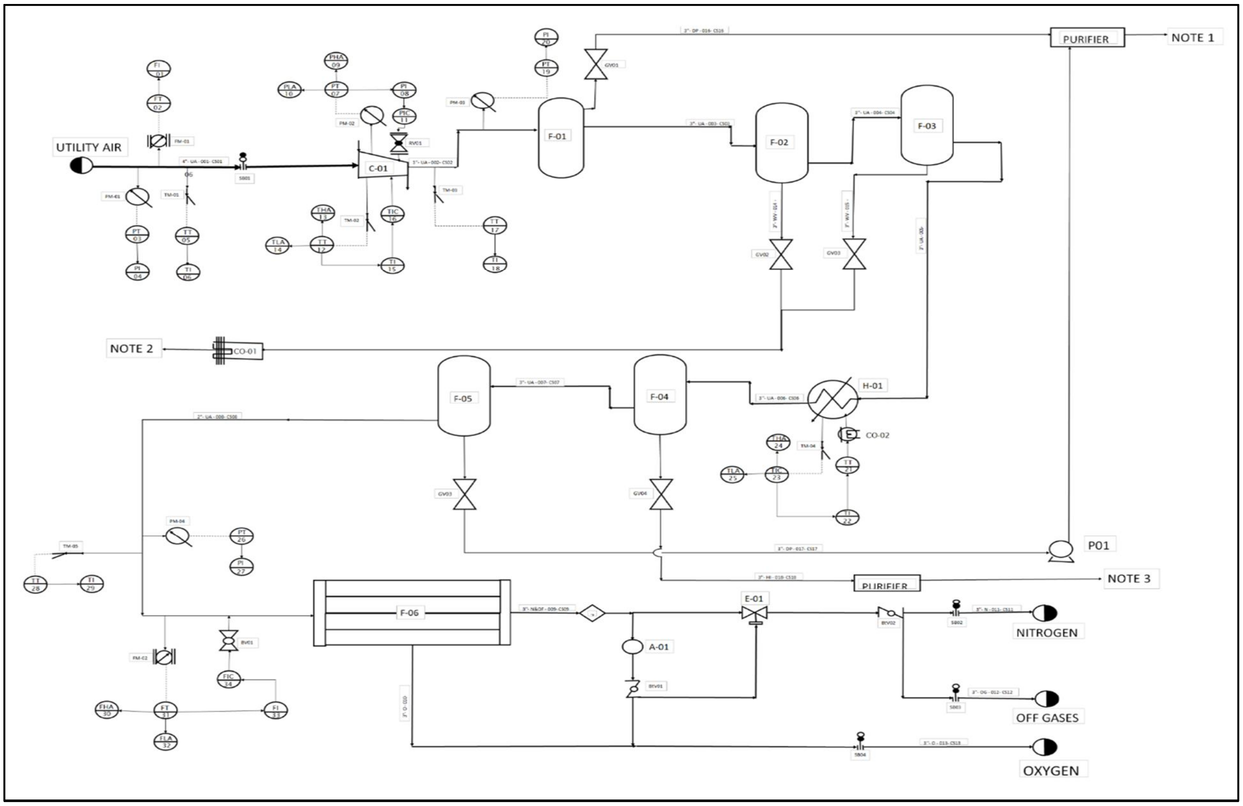

Figure 5.2: Piping and Instrumentation diagram of Membrane separation technique

	Equipment's
C-01	Compressor
F-01	Separator pad
F-02	Filter Coalescers (1 micron)
F-03	Filter Coalescers (0.01 micron)
F-04	Carbon filter vessel
F-05	Particulate Filter
F-06	Hollow fibre membrane
A-01	O_2 Analysers
E-01	Nitrogen Purity Valve
H-01	Immersion Heater
CO-01	Condenser
P-01	Pump

	Sensors and controllers
PT	Pressure Transmitter
PI	Pressure Indicator
PIC	Pressure Indication Controller
TT	Temperature Transmitter
TI	Temperature Indicator
TIC	Temperature Indication Controller
FT	Flowrate Transmitter
FI	Flowrate Indicator
FIC	Flowrate Indication Controller
CO-02	Cooler

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	Valves
RV	Relief Valve
GV	Gate Valve
BV	Ball Valve
BtV	Butterfly Valve
SB	Spectacle Blind

	Abbreviations
UA	Utility Air
WV	Water Vapour
N	Nitrogen
OG	Off Gases
O	Oxygen

Notes	
1	Vent out into the atmosphere
2	Water supplied to various sectors
3	Vent out into the atmosphere

VI. CONCLUSION

Atmospheric air contains 78% nitrogen, 21% oxygen, 1% other gases. The nitrogen is separated from the air using various technologies: 1) Cryogenic Distillation 2) Pressure swing Adsorption 3) Membrane separation technology. After doing the comparative study of these three techniques, we concluded that Membrane separation technique (MST) is the most viable& economic technology based on the following criteria that is cost (3-6 lakhs per unit), availability, simplicity in operation, low maintenance, less area occupied and purity (95-99.5%). We observed a few drawbacks in MST that are concentration polarisation, low membrane lifetime (up to 3 years). However, same needs to be analysed on case-to-case basis and overcome through various arrangements such as multiple membrane modules in series or recycle schemes etc.

VII. FUTURE SCOPE

The global membrane separation technology market is projected to reach USD 28.10 billion by 2022 at a compound annual growth rate (CAGR) of 7.2%.[6] The membrane separation technology market has witnessed significant growth in the recent years, and this growth is projected to persist in the coming years as well. These processes represent more than 80 % of the current gas separation membrane market. All have been used on a large commercial scale for 10 years, and dramatic improvements in membrane selectivity, flux and process designs have been made during that time. For example, today's hollow fine fibre nitrogen production module generates more than 10 times the amount of nitrogen, with better quality and at a lower energy consumption, than the modules produced in the early 1980s. However, the technology has now reached a point at which, barring a completely unexpected breakthrough, further changes in productivity are likely to be the result of a number of small incremental changes.[2]

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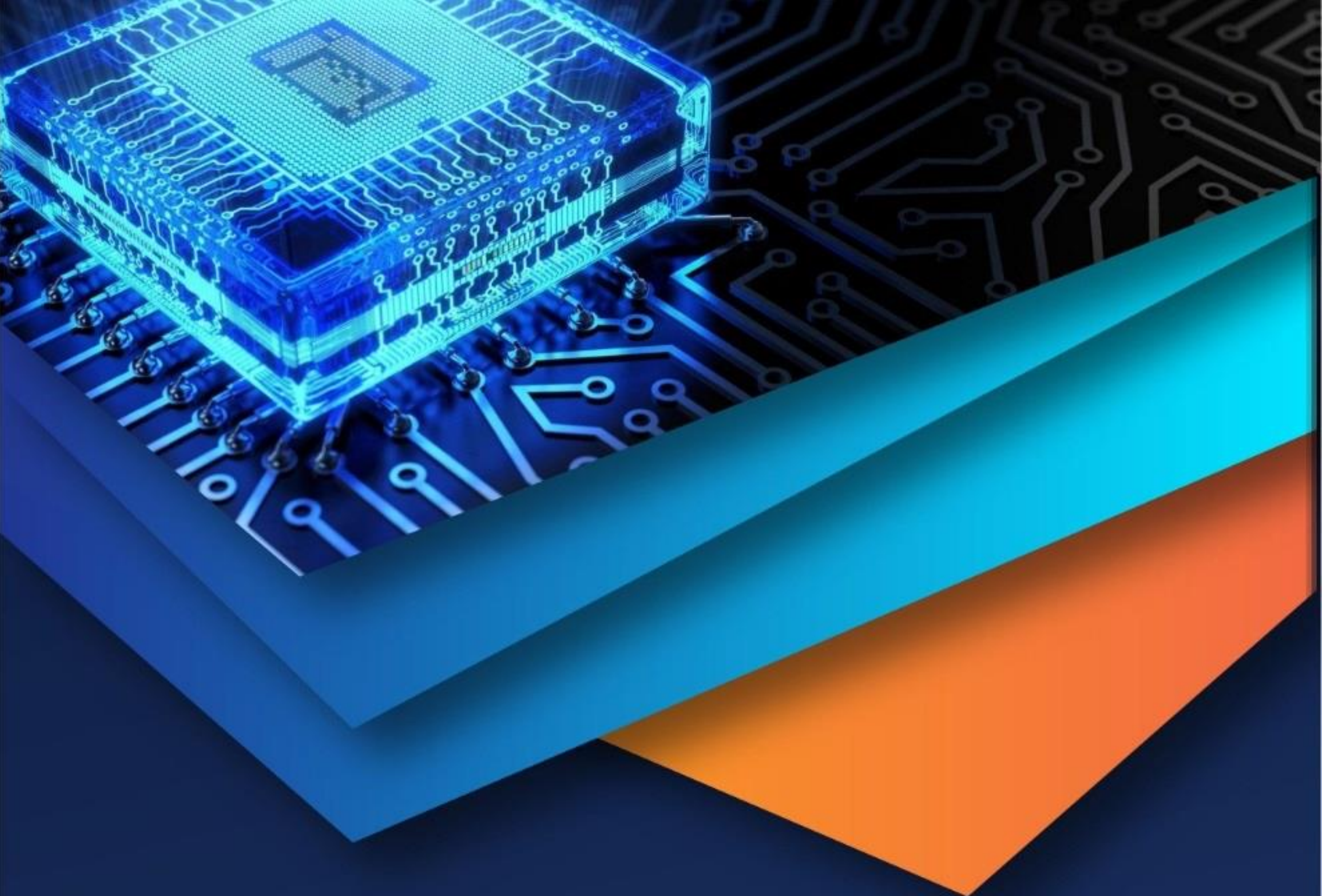

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