



ARENHA

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1. EXECUTIVE SUMMARY

1.1. Description of the deliverable content and purpose

The aim of this deliverable is to gather the technical requirements for the novel ammonia-based storage energy supply system. The proposed system includes a hydrogen production unit through a SOEC electrolyser, an ammonia production unit and an ammonia decomposition unit together with an ammonia storage unit. The industrial partners have looked up and collected the information available on state-ofthe-art systems and identified all the characteristics process parameters, such as general process layout, raw material specifications, process control and flexibility. The objective is to identify current technical limitations of conventional systems and tie them to economic constraints when applicable. This would strengthen the position of the proposed novel system assessed within the frame of ARENHA project as a viable solution to enable ammonia as an efficient energy carrier.

1.2. Brief description of the state of the art and the innovation brought

The ARENHA concept aims at integrating different innovative technology bricks to create an optimal solution for renewable energy storage and recovery through ammonia.

In power-to-ammonia phase, the renewable electricity is stored into ammonia by converting electricity into green hydrogen by electrolysis and then converting hydrogen into ammonia catalytically converted at high pressure and temperature into ammonia. The coupling of a high temperature electrolyser with a conventional ammonia synthesis loop should allow to increase the overall electrical efficiency up to 15% compared to the conventional fossil fuel-based synthesis. This significant energy demand reduction can be further improved through dedicated development of the ammonia synthesis loop with two possible approaches when targeting large or small scale production: A) Replacing the condenser of the H-B system by a solid-state ammonia absorbent, allowing an operation at much lower pressure. In combination with the development of complex-forming transition metals to catalyse the synthesis at lower temperature. Overall leading to significant reduction in CAPEX and OPEX. B) NH₃ electrosynthesis, from N₂ and water, would also lead to significant CAPEX and OPEX reduction while offering flexibility for scaling down and adaptation to fluctuating electricity production from renewable sources.

In addition, ARENHA aims to develop solid-state ammonia absorbent to provide a suitable solution for safe and cheap stationary storage of ammonia under solid form. With this solution, the operating costs will be reduced compared to state-of-the-art storage solutions. Partially due to the elimination of the compression and condensation stages and to heat integration possibilities from the exothermic absorption reaction.

In ammonia-to-power phase, although NH₃ presents compelling advantages for its penetration as energy storage, there is still a clear lack of technologies for its final utilization. In this context, the integration of advanced membranes into an intensified reactor not only provides a quantum leap in process intensification, integrating reaction and separation into one single unit, but also promotes the full conversion of ammonia at temperatures of at least 100 °C lower compared to conventional reformers, reducing the capital investment and the operating costs of the energy recovery process.



2. CONVENTIONAL SYNTHESIS OF AMMONIA

Ammonia is conventionally synthesized through the chemical reaction of hydrogen and nitrogen. At standard temperature and pressure, this reaction is not spontaneous. More than a century ago, Fritz Haber and Carl Bosch demonstrated that the hydrogenation of nitrogen can be efficiently produced at industrial scale using a metal catalyst under high temperature and pressure conditions. Thermodynamically, the reaction is exothermic favouring high pressures and low temperatures. Although high temperatures are applied in the real process to accelerate the rate of ammonia formation at the price of lower single-pass equilibrium concentration.

 $N_2 + 3H_2 \rightarrow 2NH_3 \qquad \Delta H^\circ = -91.8 \text{ kJ/mol}$

For this process to be economically feasible, it needs to operate under specific requirements which include:

- Operating at high pressures to increase ammonia content (above 200 atm)
- Operate at high temperature to increase the reaction rate (between 500 and 600°C)
- Include a high pressure recycle loop to achieve near complete conversion
- Include heat management strategies to increase overall efficiency

Currently, ammonia syngas is produced using two main routes: steam methane reforming if natural gas is used as feedstock or partial oxidation if fossil fuel or coal feedstocks are used. Regardless of the chosen route, the synthesis process consists of several basic steps including feedstock purification, syngas production, syngas compression and ammonia synthesis loop.



Figure 1. Input & output diagram of conventional ammonia process.



The Haber-Bosch process consumes a colossal >1% of the global fossil fuel production, due to its use of methane derived H_2 as a feedstock. For the 21st century, we need a more sustainable solution. Ideally, a sustainable N_2 reduction process would take place at low pressures and temperatures, allowing for ammonia to be produced locally at the point-of-consumption. The reaction would rely exclusively on renewable electricity to produce hydrogen and nitrogen, eliminating the need for fossil fuels. Besides, sustainably produced ammonia is also a highly attractive energy vector that benefits from solid infrastructure and worldwide developed transport network, which allows it to be stored safely and reversibly at high density under liquid form.

2.1. Conventional steam reforming

There are several methods of obtaining the required syngas. The syngas must be a pure mixture of hydrogen and nitrogen in the stoichiometric ratio of approximately 3:1 regardless of the combination of methods used in the syngas production. Nearly all industrial ammonia plants in operation today use water, air and fossil fuels as the raw materials for the syngas.

The steam reforming process uses a nickel catalyst that is sensitive to sulphur poisoning, therefore sulphur compounds present in the feedstock must be eliminated prior to reforming. This is achieved using hydrodesulfurization with a zinc oxide absorbent. The initial step is to react hydrogen – which usually comes from the synthesis loop of the plant – with any sulphur compounds present in the feed gas to produce hydrogen sulphide. The second step uses a bed of zinc oxide particles for hydrogen sulphide removal, producing zinc sulphide in the process.

The primary reforming process uses a furnace containing a myriad of tubes that house the nickel-based catalyst. The heat required for the reaction is transferred from the furnace to the tubes via radiation [28]. The primary reformer partially converts around 60% of feed methane to carbon monoxide and hydrogen using the following reaction:

$$CH_4 + H_2O \rightarrow CO + 3H_2 \qquad \Delta H^\circ = 206 \text{ kJ/mol}$$

During the secondary reforming process, the nitrogen required for the reaction is introduced as air and the feed gas conversion is completed. The process air is compressed and heated to 500-600°C such that the methane content is very low.

The carbon monoxide shift conversion uses CO as a reducing agent for water to produce hydrogen and carbon dioxide. CO is a temporary poison to the iron catalyst, commonly used in the ammonia converter. Thus, the shift conversion eliminates CO according to the following exothermic reaction:

$$CO + H_2O \rightarrow CO_2 + H_2$$
 $\Delta H^\circ = -41.2 \text{ kJ/mol}$

Subsequently, carbon dioxide is removed for the produced stream, as it is also considered as a temporary poison for the iron catalyst. There exist several processes to remove CO_2 from the feed gas, most of them are chemical-based solutions through absorption reactions. Captured CO_2 is often used downstream to produce Urea in fertilizer plants.



The last treatment step is the catalytic methanation to remove CO and CO₂ traces and bring them down to the parts-per-million levels using a nickel-based catalyst. Although this reaction consumes hydrogen in the process, preventing iron catalyst poisoning is of utmost importance.

2.2. Conventional Haber-Bosch loop

The H_2/N_2 syngas is compressed to high pressure prior to entering the synthesis loop. State-of-the-art centrifugal compressors are used in modern plants to pressurize the syngas to a level ranging from 150 to 250 bar. These compressors are easily scalable, cheaper and limit the risk of syngas contamination from the lubricating oil comparatively to reciprocating compressors for instance.

After the syngas is compressed it enters the ammonia synthesis loop. The reaction for the formation of ammonia from nitrogen and hydrogen is exothermic, releasing approximately 2.7 gigajoules of heat per metric ton of ammonia produced. This is equivalent to about 8% of the energy input for the entire process. Because of the thermodynamic and kinetic equilibrium limitations of this reaction, the syngas is only partially converted into ammonia; typical single pass conversions are between 20% and 35%¹.

As depicted in Figure 2, the state-of-the-art ammonia synthesis loop typically contains the following units:

- A reactor or series of reactors containing the catalysts, temperature control and heat recovery equipment.
- Cooling units for condensing the product ammonia and recovering heat.
- Separation units for ammonia recovery from the unreacted gas.
- Preheating units for the reactor feed integrated with other heat recovery units.
- Purge gas removal equipment to prevent the build-up of inert gases such as argon and methane in the synthesis loop.
- Recirculation compressors for moving the unreacted gas back to the synthesis reactor.



Figure 2. Layout of ammonia loop for typical 1500 TPD plant.

¹ Appl M. Ammonia: Principles and Industrial Practice. Wiley-VCH Verlag; 1999.



2.3. Technical requirements of the conventional Haber-Bosch loop

Following the conventional route, the typical gas composition at the inlet of the Haber-Bosch synthesis loop is typically as follow:

Component	Fraction %vol
H ₂	74.6
N ₂	24.4
CH ₄	0.30
CO	0.30
CO ₂	0.10
Ar	0.30

Conventional ammonia synthesis reactors are highly nonlinear and have numerous operating variables. The process has nearly one hundred years of industrial experience that led to an optimized and energy efficient production while lowering the related costs.

Parameter	Variation	Consequence
Prossuro	Increased value	Improved conversion rate through rate and equilibrium enhancement
r lessure	Decreased value	More flexible process and less equipment O&M constraints
Inlet temperature	Increased value	Faster reaction rate and lower equilibrium ammonia concentration
	Decreased value	Higher equilibrium concentration
Space velocity	Increased value	Higher ammonia production but lower ammonia concentration
Inert gases	Increased value	Decreased partial pressure of reactants leading to decreased ammonia yield
Catalyst particle size	Decreased value	Higher conversion rate due to lower diffusion limitations but increased pressure drop

Table 1. Impact of operating parameters on ammonia converter.

The manipulation of these process parameters dictates the rate of ammonia production and the reactor response to load variation as well as changes in inlet temperature, feed concentration, or reactor pressure.

Besides the highly pure composition of the syngas, other technical requirements are derived from the catalyst choice and the reactor design. The active catalyst for ammonia synthesis is originally derived from magnetite-based ore (Fe₃O₄) incorporated with several promoters, such as calcium oxide, alumina, potassium oxide, etc. Today, the composition of the catalyst used in industry is still more or less identical to that demonstrated in 1910. The iron-based catalysts used in the present Haber–Bosch process are effective for ammonia synthesis above 350 °C, so that the maximum ammonia yield per single-pass is low, despite excess pressurization accompanied by large energy consumption. The commonly used iron-based catalyst is of fragile nature with regards to frequent load variations, pressure/temperature cycling and



impurities such as oxygen and sulphur compounds. Other design choices in conventional plants are related to the continuous operation of the plant at nominal production. This allow for improved economics, lower maintenance and longer catalyst lifetime.

These are serious drawbacks for sustainable ammonia production from renewable energy. A lower temperature is favourable for ammonia synthesis with respect to yield and energy consumption and more efficient ammonia production is required to overcome the kinetic barrier at a lower temperature to achieve the equilibrium. However, conventional catalysts equally lose the catalytic activity for ammonia formation from N₂ and H₂ at 100–200 °C. Lower operating conditions should also allow for a more flexible control of the process.

Synthesis loop operating parameters	Technical requirements		
1- Catalyst heat rates	38°C/hr up to 400°C 19°C/hr from 400 to 510°C		
2- Catalyst replacement	10m ³ /yr for a 1500 TPD plant		
3- Catalyst life	10 years		
4- Syngas ratio	3:1 stochiometric ratio		
5- Compression	100 to 200 bar depending on reactor design		
6- Temperature	350 to 550°C depending on reactor design		
7- Single-pass conversion	20 to 35%		
8- Synthesis loop conversion	Up to 98%		
9- Recycle ratio of synthesis gas	4.5 to 5.5%		
10- Purged gas ratio	10 to 15%		
11- Turn-down ratio	40% for the ammonia converter 50% for the centrifugal compressors		
12- Product quality	99.5% ammonia, 0.5% water with oil traces Pure ammonia after downstream purification steps		
13- Heat of reaction	8% of the required input energy of the whole plant		
14- Plant availability	90% (330 days/yr) Average 5.7 showdowns per year Startup procedure takes several days		

Table 2. Technical requirements of conventional Haber-Bosch process.



3. POWER-TO-AMMONIA SYSTEMS

Sustainable ammonia can be produced from renewable electricity through using water electrolysis to produce hydrogen and air separation to produce nitrogen. Figure 3 depicts the elementary process units of such system as well as the expected inputs and outputs of each step.

The key obstacles to sustainable ammonia synthesis via the Haber-Bosch process lie at the interface between renewable water electrolysis and the Haber-Bosch process. Specifically, a sustainable Haber-Bosch plant must be compatible with an intermittent influent of hydrogen at relatively low pressure. As mentioned before, the nitrogen in air is separated from oxygen by the combustion of unreacted methane in the conventional process; should electrolytic H_2 be used instead, alternative means of producing pure N_2 at large scales are required. Additionally, current industrial Haber-Bosch plants are run at a steady state for long periods of time, catalysts and other Balance of Plant components of the process have not yet been tested under dynamic operating conditions.

The reduced pressure of hydrogen from water electrolysis also represents a significant challenge for conventional Haber-Bosch plants. Current state-of-the-art catalysts operate at temperatures of at least 400°C to achieve reasonable reaction rate, which in turn requires high operating pressures of more than 150 bar to maintain a reasonable conversion due to the negative entropy of the overall reaction. Hence, efficiently operating the Haber-Bosch process at the pressure set by water electrolysis requires the discovery of ammonia synthesis catalysts that are active at lower temperatures than those known today.



Figure 3. Input & output diagram of Power-to-ammonia plant.



The main hurdle of Power to ammonia concept is how to deal with intermittency issues created by the renewable energy source. An easy and direct solution would be to introduce hydrogen and nitrogen storage capacities to buffer the needs of the synthesis reactor. Although technically efficient, this solution is highly unlikely to be economical, particularly for small to medium size plant capacities addressing the decentralized energy storage market. An alternative solution would be to let the synthesis loop absorb the intermittency. This is a challenging task since load variation can have devastating consequences on the synthesis reactor. The main risks associated with dynamic operations are the damage of the NH₃ synthesis catalysts due to thermal cycling and the loss of containment due to H_2 embrittlement if the system is shut down and pressure is maintained.

3.1. High temperature electrolysis

3.1.1. Current state of the technology

The SOEC cell consists of two porous electrodes on either side of a thin oxygen-ion conducting electrolyte. The electrolyte is usually made of yttria-stabilized zirconia (YSZ) or gadolinia-stabilized ceria (GDC). The cathode and anode can be made of Nickel/YSZ and YSZ/LSM (Lanthanum Strontium Manganate) or LSC (Lanthanum Strontium Cobaltite). Other materials are likely to outperform the current state-of-the-art which a part of the development foreseen in ARENHA project to improve the overall performance. High temperature electrolysis is operated in temperatures between 700 and 900 °C. On the one hand, when compared to other low temperature technologies (alkaline and PEM electrolysis), a key advantage of SOECs is the capability of a high production rate at a high efficiency. On the other hand, a disadvantage of SOECs is the stability of materials at high temperature. The main reactions in a SOEC for water electrolysis are as follow:

Anode :	$2H_2O \rightarrow 2H_2 + 2O^2 + 4e^2$
Cathode :	$20^{-} + 4e^{-} \rightarrow O_2$
Overall reaction :	$2H_2O \rightarrow 2H_2 + O_2$

So far, while solid oxide electrolysers are getting closer than ever to commercialization, a great deal of discussion revolves around the choice of the most effective type of cells. On the one hand, anode supported cells allow to operate at lower temperature with reduced CAPEX and OPEX of the BOP, their degradation is still considered as too high, penalizing the efficiency on the long run. On the other hand, electrolyte supported cells maintain a high standard of performance due to lower degradation while operating at much higher temperature. Nonetheless, these state-of-the-art cells operating in electrolysis mode still suffer high polarization resistance due to materials and cells microstructure primarily designed for operation in SOFC mode.

At the system level, centralized hydrogen production by means of high temperature electrolysis is out of scale which can be reached in a mid-term time horizon. Semi-central and distributed hydrogen production however can be realized. The reasonable size of electrolyser unit is rather limited regarding to the low-end hydrogen production amount. However, too small unit sizes are considered as not economically feasible due to high costs of balance of plant components and complicated safety sub-systems required for every SOEC-based system. Amongst several designs considered by involved industrial developers, the modular plant design seems to appeal the most. Usually, components development for SOEC systems is inspired from existing SOFC systems which are at higher technology readiness level.



Figure 4. Modular system design envisaged for scale-up of SOEC systems².

In summary, current research and development efforts are focused around 4 main areas:

- Improve SOEC performance to achieve higher stack electrical efficiency resulting in significant reduction in cost of electricity usage for hydrogen production
- Develop SOEC system design configuration to achieve >75% overall thermal efficiency
- Enhance SOEC stack endurance by reducing cell and stack degradation rates
- Impart subsystem robustness for operation on load profiles compatible with intermittent renewable energy sources

3.1.2. Technical requirements

The SOEC system which can be designed following a modular approach, must be supported by auxiliary balance of plant components to ensure temperature and pressure control, provide steam at required quality, operate heat-up, shut down and emergency shutdown protocols, control hydrogen purification and compression sub-systems, manage load variation in the electrical source and allow a safe operation of the system while protecting the cells/stacks from undesired operating conditions. Thus, a successful SOEC system design should continuously monitor steam quality and remove contaminants, adequately manage thermal stresses of stacks to prevent degradation and conveniently prevent undesired operating conditions.

Amongst most important factors for high temperature stack operation there is steam utilization. Commonly considered system designs of SOEC consider partial recirculation of exhaust product gas from SOEC stack in the system. The overall gas utilization in system is therefore defined by direct utilization in stack and recirculation rate related to system layout. Typical values for steam utilization in stack are in the range of 65 to 75%³ resulting in steam content at the stack outlet below 32%. Other important operating parameters include current density, cell potential, steam quality, oxygen purity, cell pressure and

 ² Ghezel-Ayagh H., Modular SOEC System for Efficient H2 Production at High Current Density. Fuelcellenergy, 2017
 ³ R. Peters et al., Influence of operating parameters on overall system efficiencies using solid oxide electrolysis technology, International Journal of Hydrogen Energy. 2015



temperature. Table 3 highlights the impact of each one of these parameters on the overall system behavior and performance.

Table 3.	Impact of	operating	conditions	on the r	performance of	of SOEC	svstem ⁴ .
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Operating parameter	Possible range	Impact on system at higher end of range
		(+) Reduced stack cost (CAPEX)
	0 75 to 2 0	(+) Higher stack exit temperature for heat
Current density	A/cm ²	recovery
	70011	(-) Lower stack efficiency
		(-) Life impact (faster degradation)
		(+) Reduced stack cost (CAPEX)
Cell overall potential	1 1 to 1 32 \/	(+) Higher stack exit temperature for heat
	1.1 (0 1.52 V	recovery
		 (–) Life impact (faster degradation)
	50% to 90%	(+) Simpler hydrogen purification
Steam utilization		(-) Lower stack efficiency
		(–) Life impact (faster degradation)
Steam inlet	40% to 100%	(+) Higher stack efficiency
concentration		(+) Reduced system cost
concentration		(-) Life impact (potential faster degradation)
	1 to 10 bars	(+) Simpler hydrogen purification
Cell pressure		(+) Reduced downstream compression cost
Och pressure	1 10 10 0013	 (–) Life impact (potential faster degradation)
		 (-) More sophisticated stack design
		(+) Less sweep air flow to anode
Anode O_2 concentration	40% to 100%	(+) Simpler design for pressurized operation
	40701010070	(–) Higher voltage (less efficiency)
		(-) Life impact (potential faster degradation)
	650°C to	(+) Higher stack efficiency
Operating temperature	800°C	(-) Life impact (potential faster degradation)
	000 0	 (–) Higher materials cost

When the SOEC system is designed in a suitable fashion, intrinsic stack reliability and durability should be the only main factors affecting lifetime of the whole system. From a technoeconomic standpoint, the lifetime expectancy of an SOEC electrolysis system is forecasted to be 20 years in best case scenarios, meaning that at end of life conditions, the system should have surpassed a certain threshold of power consumption that is defined to be economically unfavorable.

3.2. Nitrogen production systems

In chemical industry, the three main methods of obtaining pure nitrogen gas are cryogenic distillation, polymer membrane separation, and pressure swing adsorption. Of these, cryogenic nitrogen purification

⁴ Jolly, S.. VII.C.4 Modular SOEC System for Efficient Hydrogen Production at High Current Density. 2018.



accounts for about 90% of all commercial production⁵. The chosen technology is often related to the capacity required for the specific application. Table 4 describes nitrogen plant capacity relatively to each separation technology and the purity of produced nitrogen. The final choice of the separation technology is often a trade-off between the technical requirements and the economic evaluation of each available solution.

	-			
Separation Method	Capacity (Nm ³ /h)	Purities	Load range (%)	Start-up time
Hollow fibre membranes	1 to 1,000	<99.50%	30 to 100	Minutes
Pressure Swing Adsorption	5 to 5,000	<99.99%	30 to 100	Minutes
Cryogenic distillation	200 to 400,000	ppb range	60 to 100	Hours

Table 4. Application range of nitrogen separation processes⁶.

3.2.1. Cryogenic air separation

First, cryogenic air separation exploits the difference of boiling points in the three main components of air: nitrogen, oxygen and argon (resp. 77.4K, 90.2K and 87.3K). Cryogenic nitrogen plants or air separation units (ASU) have been developed for decades by industrials, customized and optimized for commercial utilization. Although ASU units may differ in size and architecture, the general process involves several key steps including:

- Air compression and precooling to reduce humidity
- Dry air purification to remove contaminants (specifically carbon dioxide)
- Dry air-cooling using waste product from the distillation column
- Dry air cryogenic cooling to due point (approx. 97 K)
- Dry air distillation using a single distillation column

The first flexibility issue is the minimum turn-down ratio that can be achieved by the plant. If we consider a one train configuration. On the one hand, compressors are able to achieve a turn-down of 75% of their nominal capacity without recycling or venting. On the other hand, the cold box is able to achieve a turn down of 50% of its nominal capacity. Thus, in case of requirements to achieve a 50% turn-down, the compressors should be either duplicated (2x50%) or a multi-train configuration should be considered. Both scenarios should impact the total cost investment of the unit. The second flexibility issue is the load change velocity usually expressed as %/min. In the case of conventional ASU units, the typical load change velocity is approximately 0.5%/min⁷.

➔ A faster load variation increasing this load velocity would impact the purity of the produced nitrogen and oxygen. To allow for a more flexible system, more advanced process control and specific cold box equipment and design should be adopted.

⁵ Kerry FG, Industrial gas handbook : gas separation and purification, 1st ed. Boca Raton, 2007

⁶ Heinz-Wolfgang H. Industrial Gases Processing, 1st ed. Weinheim: Wiley-VCH, 2008

⁷ Dubettier R. et al., Air separation Unit: Flexibility & Energy Storage, 2nd Oxyfuel Combustion Conference, 2011







3.2.2. Pressure Swing Adsorption

In a Pressure Swing Adsorption process, the separation of nitrogen and oxygen from air takes place in an adsorber vessel filled with carbon molecular sieve (CMS). This is based on the fact of faster kinetic diffusion of oxygen molecules into the pore structure of the carbon molecular sieve than for nitrogen molecules. During generator operation, the CMS becomes saturated with oxygen. The CMS is systematically regenerated by desorbing the oxygen and moisture at a lower pressure.







As depicted in Figure 6, major equipment components include a feed air compression system; feed air pre-treatment equipment; vessels containing adsorbent; process piping and valves; a nitrogen compressor (when nitrogen is required at higher pressure); process control systems; and other auxiliary components such as coolers, separators, storage receivers, and instrument air systems.

→ Nitrogen product purity is affected by adjusting the operating pressure, temperature, bed switch frequency and flow velocity through the adsorber vessels.

3.2.3. Hollow fibre membranes

Membrane nitrogen generators are used to produce gaseous nitrogen at a specified purity, flow, and pressure from a compressed air source. At the core of these systems is the membrane module. This module typically consists of thousands of small diameter hollow fibres that are bound together on each end by tube sheaths, formed into bundles, and contained within a protective outer shell. The compressed air feed stream can be introduced either on the shell or bore side of the membrane fibres. Since oxygen permeates more rapidly than nitrogen through the membrane wall, the feed air is separated into two gas streams. The first, the nitrogen product, is produced at a pressure approximately 2 bars lower than the compressed air pressure. The second, the waste stream, is enriched in oxygen and is at a much lower pressure, generally close to atmospheric pressure.

Major equipment components include the feed air compression system; feed air pre-treatment equipment; membrane modules; process piping and valves; a nitrogen compressor (when the user's process requires higher pressures than the nitrogen membrane output pressure); process control systems and other auxiliary components such as separators, storage receivers, and instrument air systems. The compressed air is generally treated to remove any condensed liquids, entrained mists, solid particulates, and sometimes vapor-phase contaminants before introduction into the membrane separator. The degree of clean up required depends on the particular contaminants present, the effects those contaminants will have on the performance and lifetime of the membrane, and the final product purity requirements. Pre-treatment steps typically include filtration, and temperature and/or pressure control. After pre-treatment, the clean compressed air is fed to the membrane separator(s), which can be arranged alone or in multiple parallel or series banks.

➔ The nitrogen purity is affected by controlling the operating pressure, temperature, and flow through the membrane module.







Besides these conventional solutions already mature and widely used in industry for N_2 separation from Air, a promising alternative would be electrochemical N_2 production by extracting oxygen from Air using renewable electricity. The main advantages of such an electrochemical N_2 separation route are: 1) scale flexibility which can be designed to match the renewable electricity production capacity/small scale Haber-Bosch NH₃ synthesis unit. 2) More energy efficient and higher system efficiency can be achieved by heat integration with the high temperature SOEC unit. This type of N_2 production solution is investigated in the ARENHA project.

3.3. Advanced Haber-Bosch loop systems

The aim of these new advanced systems is to come up with technical solutions that will allow the Haber-Bosch process to operate at much larger turn-down ratio while limiting the impact on the sensitive components of the process. This would essentially allow the process to absorb more curtailed energy which would significantly impact the economic viability on the long run. In the following sections, we introduce some of the most promising solutions and concepts that are reported in scientific literature.

3.3.1. Proton Ventures solution

The use of renewable energy to produce green ammonia is currently going from academic research to pilot scale plants. The NFUEL units of Proton ventures which are small scale ammonia plants already have industrial references in Argentina, China and Switzerland⁸. It is based on decentralized production of Ammonia from surplus electricity from renewable sources, NFUEL is designed for three production capacities (3, 10 and 60 t/d) following three developments steps, the unit is made in skids, each fitting 12m standardized containers, without surpassing 12 meters of height. The process is fully automated and remotely operated.



Figure 8. Proton Ventures NFUEL® process

This technology is based on a large-scale electrolysis conventional Haber-Bosch with an energy consumption of nearly between 9-11 kWh/kg NH₃. Although it relies on conventional Haber-Bosch process because (operation at high pressure of 300-460 bar and high temperature 400-550°C⁹), this solution has already reached a high technology readiness for this kind of application. The system is powered by renewable electricity derived from wind turbines or solar panels, and is designed to withstand rapid power

⁸ Nitrogen and syngas 354, *Sustainable ammonia for food and power*, 2018

⁹ Kevin H.R. et al., Islanded ammonia power systems: Technology review & conceptual process design, Elsevier Ltd, 2019



fluctuations, in term of lifecycle the reactor, compressor can operate efficiently for more than 30 years due to the specifically tailored design to resist fatigue through possible cycles of pressurization/depressurization¹⁰. Figure 9 depicts the main blocks of the process and Table 5 provides a cost estimate for a 3 TPD ammonia production capacity plant.



Figure 9. Proton ventures NFUEL® block diagram.

Table 5. Capital cost estimation of Proton Ventures for a 3 TPD unit

Block	Cost (k€)
Wind turbine	800.00
Air separation	200.00
Demi-H ₂ O	50.00
Water electrolysis	1310.00
Ammonia synthesis plant	1600.00
Ammonia storage	40.00

3.3.2. Thyssenkrupp conceptual design of small-scale systems

Thyssenkrupp, a key player in the carbon-free hydrogen production from renewable electricity and water, announced the launch of its technology for the production of green ammonia. The project will be started in collaboration with H2U (the Australian hydrogen infrastructure firm) in port Lincoln as a first demonstration plant.

The project in which the Thyssenkrupp technology will be used will include a 20 MW water electrolysis plant and an ammonia production facility with a capacity of 50 TPD, a small quantity of the ammonia produced will serve the local market whereas the other part will either be used as an energy storage or will be exported to other regions. Based on this demonstrator another facility will be designed for an ammonia production capacity of 300 TPD requiring 120 MW. The project will use 2 gas turbines, 16 MWe each, based on the combustion of hydrogen.

¹⁰ A. Patil, L. Laumans, H. Vrijenhoef, Solar to Ammonia – via Proton's NFuel units, Elsevier Ltd, 2013



The announced energy consumption will be similar to Proton Ventures technology, for both production capacities, the plant will consume 10 kWh/kg of ammonia and use 85 kg of cooling water per kg of ammonia for the 50 TPD plant and 125 kg of cooling water/kg of ammonia for 300 TPD.

The company aims at developing specific markets and applications such as sustainable fertilizers, renewable energy storage and chemical feedstock, moreover it evaluated five key viability drivers for the small scale production units starting by capital expenditure next to availability and cost of renewable power, in addition to possible restrictions on and cost of ammonia transportation and CO₂ emissions.



Figure 10. Thyssenkrupp and H2U Power-to-ammonia concept.

3.3.3. Minnesota University's pilot demonstrator

The University of Minnesota has developed a pilot for ammonia synthesis using water and air, powered by locally produced wind energy. The pilot was constructed in 2012 and uses the electricity produced from a wind turbine. Hydrogen is produced through water electrolysis and nitrogen is captured from the air through adsorption or membrane solutions. The pilot capacity is 80 kg of ammonia per day stored in a 11 m³ tank.

In order to increase the energy efficiency, the plant is equipped with a series of shell and tube heat exchangers (4 Hex), in which the compressed mixture of feed gases and recycled H_2 and N_2 will be preheated by the reactor product before feeding heater, the product has high temperature because of the exothermicity of the reaction, it will be condensed via a series of chiller in order to be separated to have the final product containing ammonia at -26°C and under 17 bar¹¹. The operating condition inside the reactor are mentioned in the process diagram presented in Figure 11. The targeted applications for such system range from sustainable fertilizer production, renewable energy storage to alternative fuel production.

¹¹] G. Soloveichik, A. McCormick, *wind energy to ammonia synthesis*, University of Minnesota, 2017



Figure 11. University of Minnesota ammonia synthesis process

The technology is operated under 150 bar which is lower than the conventional operating conditions, however in order to make it more feasible for small scale plant, the developers are looking to make the process more efficient and operating at lower conditions, 400°C and only 20 bar, by absorbing ammonia at modest pressures as soon as it is formed. Basically the partially converted product is sent into a separator after cooling where the ammonia is captured and removed by selective absorption, which allows the synthesis to operate at lower pressure without changing other units of the process (see Figure 12).



Figure 12. University of Minnesota's low-pressure ammonia synthesis process.

3.3.4. SIEMENS and STFC's pilot demonstrator

SIEMENS has developed a pilot plant for ammonia synthesis in Oxford shire-UK with a daily production capacity of 30 kg and a storage capacity of 350 kg. The system relies on renewable energy from a wind turbine to provide electricity to the process. An air separation unit is used to produce nitrogen and a water electrolyser to produce hydrogen. The syngas is then sent into the synthesis reactor to produce green ammonia used as a mean to store renewable energy. SIEMENS targets application for renewable energy storage and ammonia use as an alternative fuel. The following block diagram shows the major steps of this pilot. Since 2020, the demonstrator belongs to the STFC (Science and Technology Facilities Council) located at the Rutherford Appleton Laboratory.



Figure 13. Block diagram of STFC/SIEMENS pilot.



Figure 14. STFC/SIEMENS installed ammonia pilot demonstrator.

4. AMMONIA DECOMPOSITION SYSTEMS

The dissociation of ammonia is endothermic and requires the input of heat to drive the reaction to completion. The total energy input required to achieve and maintain ammonia decomposition depends on recovery rate and reaction temperature. The dissociation rate depends on temperature, pressure and catalyst. The theoretical limitation for lowest working temperature is given by the chemical equilibrium (i.e. thermodynamic limitation). For an almost complete decomposition of ammonia (i.e. high conversion rate > 99.5%) this temperature is approximately 430°C at atmospheric pressure. However, with regard to reaction heat, the endothermic NH₃ decomposition occurs at high temperatures to attain high conversion with fast kinetics, indicating strong heat transfer limitations.

4.1. Conventional systems

In commercial systems, liquid ammonia stored in bottles or ammonia tanks is pumped and pre-heated in a heat exchanger and a vaporizer then cracked in the main furnace unit. In these available systems, the furnace is often electrically heated by special Light Gauge Over-bend (LGO) heating elements.

Typically, small to medium scale dissociation of ammonia gas takes place at temperatures as high as 950° C in the presence of a special nickel catalyst within the electrically heated furnace. The cracked gas then goes through the heat exchanger for pre-heating the incoming gas. Depending on end-use, the generated gas (75% H₂, 25% N₂) may or may not undergo a final purification step.





Figure 15. Process layout of conventional ammonia cracking unit.

So far, ammonia cracking is operated on heterogeneous nickel catalyst requiring at least 900°C to ensure complete NH_3 conversion. In a use case where ammonia would be seen as an energy carrier, the development of efficient ammonia crackers able to generate pure hydrogen is required. As for the needs of large-scale green ammonia decomposition units, there are no available or known processes to decompose ammonia at industrial scale. Most commercially available solutions offer an electric-based furnace solution at a production capacity ranging from 1 to 2 TPD. These commercial units usually produce a forming gas (H_2 and N_2 mixture) for various industrial applications and rarely include any additional steps to produce hydrogen at high purities required for utilization in fuel cells.

Manufacturer	LINDBERG	CI HAYES	KOYO THERMO	BOREL
H2 output (kg/day)	273	136 to 228	96	11
Temperature	-	- 920		950
	75% H ₂ ,	75% H ₂ ,	75% H ₂ , 25% N ₂	75% H ₂ ,
Product purity	25% N ₂	25% N ₂	NH₃ (-65°C)	$25\% N_2$
Electricity consumption (kW)	140	64 to 140	59.5	7.5
Efficiency (%)	63	55 to 66	55	51
Weight (kg)	5800	2180	-	Ι
Footprint cm×cm	290×240	180×170	280×100	85×56
Cost (\$)	155,000	70,000	_	_

Table 6.Technical requirements for commercial ammonia crackers¹².

¹² V. Hacker and K. Kordesch, Ammonia Crackers, Handbook of Fuel Cells – Fundamentals, Technology and Applications, 2003



4.2. Advanced systems

Although ammonia cracking is a common operation in the industry to produce a forming gas with typical composition of 75% hydrogen, 25% nitrogen and approximately 1000 ppm ammonia, scarce technical solutions are available to recover pure hydrogen from ammonia for energy storage application. In order to reach the purity level required at the inlet of a PEM fuel cell (0.1 ppm NH₃ content), it is necessary to develop a selective ammonia cracking unit able to separate effectively both nitrogen and ammonia. Ammonia cracking being an endothermal reaction, it is favored at high temperature and low pressure. Using low cost nickel catalyst with a conventional fired cracker appears to be the easiest way especially if ammonia content in the outlet flux is not a limitation. If higher conversion rates are reached at higher temperature, this is achieved at the expense of the overall energy efficiency; ammonia being used to heat the gas mixture within the cracker. Conventional ammonia crackers are thus more adapted for large-scale decomposition where effect of scale can be enormous on capital expenditures. When it comes to decentralized ammonia cracking for pure hydrogen generation and fuel cell application; it is of high interest to operate the ammonia decomposition at lower temperature for better energy efficiency, particularly if post-cracking operation is performed. Therefore, most of the research and development efforts in this field are directed towards finding suitable catalysts for ammonia decomposition at mild conditions. Table 7 lists some of the most promising catalysts for this application.

Metal/Catalyst	Comments
Ru-based	 Under development since 2004 Widely known state of the art catalyst Ru dispersion is improved by addition of carbon nanofibers, graphite and so on Best performing Ru catalyst is 12%wt Ru doped with potassium supported on MgO and CNT With KOH modified Ru, complete conversion is obtained at 500°C Main disadvantage rely in abundance of Ru precious metal Shorter catalytic lifetime than Fe catalysts
Fe, Co, Ni	 High catalytic activity for ammonia decomposition at high temperature (> 600°C) and low pressure Transition metal nanoparticles dispersed on alumina matrix, CNT and carbon nanofiber shows significant improvement at 550°C
Alkali Amide	- Examples include lithium amide and sodium amide
MnN-Li ₂ NH	 Composite of metal nitride and alkali amide/imides showing comparable activity compared to state-of-the-art Ru/CNT

	Table 7. Catalyst for	he low temperature	decomposition of	ammonia ¹³
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¹³ S. Mukherjee et al., *Low-temperature ammonia decomposition catalysts for hydrogen generation*, Applied Catalysis B: Environmental, 2018.



	 Can be much more stable than lithium amide/imide alone if optimized
Fe2N-Li2NH	 Promising for low temperature ammonia decomposition provided that the reaction conditions and molar ratios of the component are optimized

Aside from catalyst development, reactor technology development is also a key aspect for an efficient and cost-effective ammonia cracking process. Several new technologies have emerged in the last decade focusing on producing high purity hydrogen from ammonia. For instance, RenCat is commercializing a patented fuel processing technology to process ammonia to Fuel Cell grade hydrogen. Their technology uses a non-noble metal catalyst (FeNi alloys) for hydrogen generation and an abundant inexpensive metal oxide to purify the hydrogen of trace ammonia. Instead of using Pd based membrane to eliminate ammonia, RenCat uses a selective oxidizer that allows a high hydrogen purity generation, the process is called selective ammonia oxidation (SAO). This technology is protected with two patents regarding catalyst material for selective oxidation of ammonia. RenCat is currently providing 2 main products with capacities of 1 and 10 l/min of hydrogen. They are also scaling up their generators to 5 kW. Few information is available on the performance of this technology and its advantages compared to other solutions. However, this technology still requires a downstream purification step to produce high purity hydrogen.



Figure 16. RenCat technology for ammonia cracking and hydrogen purification.

Membrane technology seems to offer a promising potential for ammonia cracking. In a membrane reactor, the chemical reaction and the selective separation of a product of the reaction occur simultaneously inside the reactor. In chemical reactions limited by the thermodynamic equilibrium like in ammonia decomposition, the selective separation of one of the hydrogen products will shift the thermodynamic equilibrium according to Le Chatelier's principle and will allow the system to go beyond this thermodynamic limitation. This will bring several advantages over conventional systems:

- Pure H₂ is recovered through the membranes, which can be fed directly in fuel cells, and avoids any costly downstream separation unit.
- The thermodynamic equilibrium limitation is circumvented, and it is possible to obtain full fuel conversion, reducing the downstream cleaning of unconverted species.
- The separation of H₂ allows to get higher efficiencies at lower operating temperatures, which has a corresponding benefit from an energetic point of view.
- Since the whole process occurs in a single unit, the footprint of the technology is significantly reduced.

For these reasons, membrane technology has gained more interest in the last years for the ammonia decomposition application. For instance, CSIRO has developed a vanadium-based membrane dedicated



to hydrogen purification, the main application being to recovery a pure hydrogen flux with purity meeting ISO14687 for PEM fuel cell of a forming gas coming from a prior ammonia cracker.

CSIRO has developed a complete system integrating a catalytic cracker operating between 450°C and 500°C. The catalyst used is provided by Johnson Matthey and consists in a 1.5 mm granular catalyst composed of a 0.5% wt% Ru Layer on Al_2O_3 support. Ammonia conversion in condition ruling within the cracker (450°C, 5 bar pressure) is high and very close to the equilibrium conversion rate (97.9%).



Figure 17. Ammonia cracking system developed by CSIRO for the recovery of pure hydrogen from ammonia¹⁴.

Experiments have been made in a complete system in Brisbane (Australia). In the system developed by CSIRO, the ammonia stored within a tank is directly feeding the cracker. This demonstration was part of a two-year project to demonstrate CSIRO H_2 -production system aiming to deliver at least 5 kg/day of H_2 , directly from NH₃.

Observed results have shown that the actual ammonia cracking rate was much lower than anticipated mainly as a consequence of failed heating elements and poor heat transfer. Indeed, it has been observed that the unseasonably cold Brisbane winter limited the NH₃ supply pressure. This translated in poor recovery through membranes. Vanadium membranes have poor catalytic activity for hydrogen and are easily oxidized; therefore, a thin Pd coating is typically deposited on both sides of the membrane to prevent oxidation and promote hydrogen dissociation/association forming a sandwich like membrane. However, when the membrane is operated at more than 400°C, an interdiffusion between Pd and V takes place eventually leading to degradation. Thus, hydrogen separation should be carried out at lower temperatures (300- 350 °C as shown in Figure 17).

¹⁴ M. Dolan, « Delivering clean hydrogen fuel from ammonia using metal membranes », presented at ENERGY, 2017.



5. CONVENTIONAL AMMONIA STORAGE SOLUTIONS

The physical properties of ammonia demand high pressure or high refrigeration to store it in a liquid phase. If ammonia is stored in high pressure vessel, it would require nearly 17 bars of pressure to maintain liquid phase and meet minimum safety requirements for storage and transportation. However, full pressure, shop fabricated storage vessels are practical to the size of 300 tons¹⁵.

The other method to store ammonia is using refrigerated vessels. They have much higher storage capacity (as high as 66.2 million liters), and do not require high pressure. The temperature requirement for low pressure storage is -33°C at 1 atm. Depending on the energy requirements to meet these demands and expected storage capacity and costs associated with the system, the decision of whether to use pressurized vessel or refrigerated vessel is determined.

Assuming an energy density of 11.5 MJ/liter, a single storage tank of 40,000m³ of ammonia can store enough energy to meet the annual electricity demand of 30,000 households. Although the storage tanks do not require any special materials, ammonia has a high coefficient of thermal expansion. The volumetric density at saturation pressure and 0°C is 636 kg/m³ whereas at 20°C it reduces to 609 kg/m³. Therefore, tank walls need to be insulated to avoid material stress and potential bursting.

Туре	Pressure (bar)	Temperature (°C)	Tons of ammonia per ton of steel	Capacity (tNH₃)	Refrigeration compressor	
Pressure storage	16 to 18	ambient	2.8	<270	N/A	
Semi- refrigerated storage	3 to 5	0	10	450 to 2,700	Single stage	
Low temperature storage	1.1 to 1.2	-33.6	41 to 45	4,500 to 45,000	Two stage	

Table 8. Conventional ammonia storage solutions¹⁶.

Besides these conventional solutions already mature and widely used in industry, a promising alternative would be to store ammonia in a solid state. The main advantage of such solid-state storage via ammonia ab/desorption in an absorbent is to reduce the capital investment for the storage systems at increased safety level when compared to liquefied ammonia. This eliminates the need of large cooling, large insulation, pumping to high pressure and high-pressure tank designs. This type of storage solution is investigated in the ARENHA project.

 ¹⁵ J. R. Bartels, *A feasibility study of implementing an ammonia economy*, Iowa State University, 2008.
 ¹⁶ Appl M. Ammonia: *Principles and Industrial Practice*. 1st ed. New York: Wiley-VCH, 1999.



6. Summary of technical requirements of industrial processes

The table below summarizes the technical requirements of the conventional processes as per available data on literature.

	Process	Pressure (bar)	Temperature (°C)	Capacity	Product or reactant purity	Load range	Start- up time	Other characteristics
SOE	C electrolysis	ambient	650 to 850	_	>99%	_	Minutes	0.75- 2.0 A/cm ² 1.10- 1.32 V/cell SU 50% to 90%
Nitrogen production	Hollow fiber membranes	5 to 16 ¹⁷	ambient	1 to 1,000 Nm ³ /h	<99.50%	30% to 100%	Minutes	0.43 to 0.65 kWh/m ³ of N _{2 @ 8} _{bar}
	Pressure Swing Adsorption	5 to 12	ambient	5 to 5,000 Nm ³ /h	<99.99%	30% to 100%	Minutes	0.26 to 0.65 kWh/m ³ of N _{2 @ 8} _{bar}
	Cryogenic distillation	1.3 to 6	-190	200 to 400,000 Nm³/h	ppb range	60% to 100%	Hours	0.15 to 0.25 kWh/m ³ of N _{2 @ 8} _{bar}
Hab	er-Bosch loop	100 to 200	350 to 550	100 to 3,300 TPD in single train	74.6% H ₂ 24.4% N ₂ 0.30% CH ₄ 0.30% CO 0.10% CO ₂ 0.30% Ar	40% to 100%	Days	_
de	Ammonia composition	1 to 3	800 to 950	10 to 2,000 kgH₂/day	75% H ₂ , 25% N ₂ NH3 ppm	_	Hours	50 to 65% electric efficiency
Ammonia storage	Pressure storage	16 to 18	ambient	<270 tNH₃	-	_	_	-
	Semi- refrigerated storage	3 to 5	0	450 to 2,700 tNH₃	-	Ι	Ι	Single stage compressor
	Low temperature storage	1.1 to 1.2	-33.6	4,500 to 45,000 tNH₃	_	_	_	Two stage compressor

¹⁷ Rautenbach, R.; Struck, A.; Roks, M.F.M. A variation in fiber properties affects the performance of defect-free hollow fiber membrane modules for air separation. J. Membr. Sci. 1998, 150, 31–41



7. Conclusion

In the ARENHA project, the goal is to demonstrate the feasibility of ammonia as an effective zero emission energy carrier. To unlock this new potential, both technical and economic barriers must be lifted first. A first step is to start with existing state-of-the-art technology to establish what can be improved to deal with renewable energy application, more specifically to cope with intermittency.

Currently, ammonia is produced using fossil fuel feedstock: steam methane reforming if natural gas is used as feedstock or partial oxidation if fossil fuel or coal feedstocks are used. Regardless of the chosen route, the synthesis process emits a substantial amount of CO₂ mainly attributed to the syngas production. Sustainable ammonia can be produced from renewable electricity through using water electrolysis to produce hydrogen and air separation technologies to produce nitrogen. Furthermore, by improving the ammonia converter flexibility, the need for storing feed reactants can be substantially reduced. This is essentially a direct way of reducing investment and O&M costs for such application. Improving the flexibility of the synthesis loop can be achieved by means of reducing the operating conditions (i.e. pressure and temperature) which corelated to finding new materials for catalysts and ammonia recovery units.

The transport of renewable energy in the form of green ammonia is a viable way to scale up the production of these renewable sources, through the already existing distribution network of this chemical compound, thus effectively solving the storage issue which is intrinsic to renewable sourcing. Nonetheless, once the renewable energy is stored and distributed, technical solutions must be developed to efficiently recover this energy back. With this regard, several options could be considered, including ammonia decomposition to recover pure hydrogen.

Although ammonia cracking is a common operation in the industry to produce a gas mixture with typical composition of 75% hydrogen, 25% nitrogen and approximately 1000 ppm ammonia, scarce technical solutions are available to recover pure hydrogen from ammonia for energy storage application. In order to reach the purity level required at the inlet of a PEM fuel cell (0.1 ppm NH₃ content), it is necessary to develop a selective ammonia cracking unit able to separate effectively both nitrogen and ammonia. In this framework, the ARENHA project aims to investigate and demonstrate a novel membrane-based process for efficient ammonia decomposition and hydrogen separation.